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PRODUCTION OF BIODIESEL FROM VERNONIA GALAMENSIS OIL USING ETHANOL WITH ALKALI CATALYST

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Abstract - This work was done with the aim of producing biodiesel from vernonia galamensis oil by using ethanol with alkali catalyst, sodium hydroxide. Additionally it was investigated the effects of catalyst amount from 0.25 %(w/w) to 2 %(w/w) of weight of oil, molar ratio of ethanol to oil from 6:1 to 12:1 and reaction temperature from 35°C to 75°C on biodiesel yield. Vernonia galamensis oil was extracted using solvent extraction and mechanical pressing. The extracted oil was purified through degumming, neutralization, washing and drying sequentially. Acid value, amount of free fatty acid, saponification value and flash point of the extracted oil were determined. Biodiesel was produced from vernonia galamensis oil using anhydrous ethanol 99.5% (w/w) and sodium hydroxide catalyst 97% (w/w). The experimental design was done by using the Design Expert 7.0.0 software three levels; three factors Central Composite Design with full type in the optimization study, requiring 20 experiments. To determine the effect of temperature, amount of catalyst and molar ratio of alcohol to oil experiments were done in the ranges of 35°C to 75°C, 0.25% to 2.0% (w/w) and 6:1 to 12:1 subsequently. The maximum biodiesel yield was 87 % (w/w) at 55°C, 9:1 molar ratio of alcohol to oil and 1.125% (w/w) sodium hydroxide catalyst amount. In contrast, the minimum biodiesel yield was 52% (w/w) at 75 ° C, 12:1 molar ratio and 2% (w/w) catalyst amount. The viscosity, density, flash point, acid value, saponification value, moisture content and ash content of the produced biodiesel were determined. These properties were matched with ASTM specifications. Based on the preliminary economic analysis evaluation, the suggested project is feasible.

Keywords: Ethanol, saponification value, Vernonia galamensis, transesterification, Triglycerides

1.Introduction

Biodiesel is the name for a variety of ester based fuels (fatty ester) generally defined as monoalkyl ester made from renewable biological resources such as vegetable oils (both edible and non edible), recycled waste vegetable oil and animal fats. This renewable source is as efficient as petroleum diesel in powering unmodified diesel engine. Today's diesel engines require a clean burning, stable fuel operating under a variety of conditions. Using biodiesel not only helps maintaining our environment, it also helps in keeping the people around us healthy. Biodiesel is miscible with petro-diesel in all ratios. In many countries, this has led to the use of blends of biodiesel with petro-diesel instead of neat biodiesel [1].

There are different types of feed stocks that are used for the production of biodiesel. These includes linseed oil, palm seed oil, waste cooked vegetable oil, sunflower seed oil, cotton seed oil, jatropha seed oil, vernonia galamensis seed oil and animal fats. Oilseed plants are used for the production of biodiesel through the process called transesterification reaction which is a process by which alcohol reacts with vegetable oil in the presence of catalyst. Triglycerides are major components of vegetable oils and animal fats. Chemically, triglycerides are esters of fatty acids with glycerol. Fatty acid ethyl esters are mostly involved because ethanol is the cheapest alcohol, but other alcohols, namely methanol, may be employed as well. In this way, highly viscous triglycerides are converted in long chain monoesters presenting much lower viscosity and better combustion properties to enhance the burning. Homogeneous or heterogeneous catalysis are used to enhance the reaction rate. Since different fatty acid has different physical and chemical properties, the fatty acid content is the most important parameter influencing the corresponding properties of a vegetable oil or animal fat [4].

Vernonia galamensis is a new potential industrial oilseed crop with origin in East Africa. The seed oil has unique chemical and physical properties that will permit its use in the formulation of reactive diluents, products to serve as solvents that become part of the dry paint surface and do not evaporate to pollute the air. Vernonia galamensis is one of the feed stocks which are

rich in a useful epoxy fatty acid called vernolic acid. It has the largest oil content, up to 42%; and the extracted oil can contain vernolic acid (78-80%), linoleic acid (12-14%), oleic acid (2-3%) palmic acid (2-3%) and a trace amount of arachidic acid (2-3%), and used in paint formulations, coatings, plasticizers, and as a reagent for many industrial chemicals [2].

2.Materials and Methods

2.1Materials

Materials and Chemicals used for biodiesel production were vernonia galamensis seeds oil, ethanol alcohol, sodium hydroxide catalyst, Potassium hydroxide, ethanol alcohol, phenophtaline, hydrochloric acid, Phosphoric acid, distilled water. Equipments used in the production were centrifuge, conical flask, glass jacketed reactor, beaker, stirrer, crushing mill, vacuum filtration, rotary evaporator, condenser, cotton cloth, screw presser, conical flask, dryer, water thermostat bath, vibro-viscometer, oven, graduated cylinder, hydrometer, furnace, stove and burning cup.

2.2 .Methods

2.2.1.Vernonia galamensis seeds sample preparation

Vernonia galamensis seeds were cleaned and prepared. Seeds were crushed by motor mill with 1.0 mm to 2.0 mm sieve size in size reduction laboratory; then the sample was ready for oil extraction.

2.2.2. Vernonia galamensis oil extraction

The oil was extracted by solvent extraction and mechanical pressing extraction methods. Procedures of oil extraction using hexane solvent [22].

2.2.3.Oil extraction using solvent

Seeds were crushed in a crushing mill with a particle size of 1.0 mm to 2.0 mm. Then, the crushed seeds and hexane solvent were placed in the extraction unit at a solvent to solid ratio of 5:1(v/w). The solvent and crushed mixtures were mixed at 200 rpm speed and heated at constant temperature of 65^{o}C for 15 hours to extract the oil. After extraction the solids and the solvent oil mixtures were separated by a settling followed by a vacuum filtration. The solvent and oil were separated in a rotary evaporator at a temperature of 70^{o}C.

2.2.4.Oil extraction using mechanical pressing

Seeds were crushed using a crushing mill with a particle size of 1.0 mm to 2.0 mm. Next, the crushed seeds were tied by a cotton cloth and fed at the top of a presser. At each batch 1kg of the crushed seed was fed into the mechanical presser at atmospheric temperature. The presser was rotated manually until the screw tight strongly where the oil extraction was taking place. Since the crushed kernel was tied by the cotton cloth, there was no need for a cake filtration. The oil was collected at the bottom of the mechanical press.

2.2.5. Vernonia galamensis oil refining

For refining the oil settling, degumming and neutralization methods were applied.

2.2.5.1.Settling: It is crude oil separating from impurities by using a centrifuge at a speed of 800 rpm for 20 minutes.

2.2.5.2.Degumming: It is used to remove phosphorus compounds of crude oil using a phosphoric acid and a hot water. Distilled water 3% (v/v) of oil at 70^{o}C and 1.5% phosphoric acid (v/v) of oil were mixed with the oil which was heated at 70^{o}C. The mixtures were stirred at speed of 200 rpm for 1 hour at a temperature of 70^{o}C.

2.2.5.3.Neutralization: After determining the free fatty acid (FFA) of oil, the free fatty acid was neutralized by 0.05N of NaOH. Neutralization was done by heating the oil at 70^{O}C. The impurities were separated using a centrifuge at a speed of 800 rpm for

20 minutes. The mixture of oil and NaOH solution were stirred at 200 rpm at a temperature of 70°C for 1 hour. The mixture was washed with a distilled water to remove a trace NaOH and produced soap. Finally, trace water was removed in an oven drying at a temperature of 105°C for 6 hours.

3. Experimental work design

Process variables revised are reaction temperature, molar ratio of ethanol to oil and weight percentage of catalyst. To get maximum conversion; reaction period and rotation speed was set at 2 hours and 500 rpm respectively and at constant atmospheric pressure. The operating limits of the biodiesel production process conditions are reasons to choose levels of the variables.

Three level three factors Central composite Design(CCD) was made use of in the optimization study, needing 20 experiments to be done. Catalyst concentration, ethanol to oil molar ratio and reaction temperature were the independent variables selected to optimize the conditions for biodiesel production by using sodium hydroxide as main catalyst for performing trans esterification reaction.

Twenty experiments were done and the data was statistically analyzed by the Design Expert 7.0.0 software and to get suitable model for the percentage of fatty acid methyl ester as a function of the independent variables.

Table3.1: Complete experimental design matrix of CCD

Variables	Factor Coding	Unit	Levels		
			-1	0	+1
Reaction Temperature	x_1	°C	35	55	75
ethanol to Oil ratio	x_2	-	6	9	12
Amount of Catalyst	x_3	g	0.22	0.99	1.76

Table3.2: Experimental design matrix

Run	Coded Factors			Actual Factors			Actual Biodiesel yield %(w/w)
	X_1	X_2	X_3	Temperature (°C)	Ethanol to oil	Catalyst (g)	
1	-1	+1	+1	35	12	1.76	
2	+1	+1	-1	75	12	0.22	
3	0	0	0	55	9	0.99	
4	-1	-1	-1	35	6	0.22	
5	0	0	0	55	9	0.99	
6	+1	-1	+1	75	6	1.76	
7	0	0	0	55	9	0.99	
8	-1	-1	+1	35	6	1.76	
9	+1	+1	+1	75	12	1.76	
10	0	0	0	55	9	0.99	
11	-1	+1	-1	35	12	0.22	
12	+1	-1	-1	75	6	0.22	
13	0	0	0	55	9	0.99	
14	0	+1	0	55	12	0.99	
15	0	0	-1	55	9	0.22	
16	-1	0	0	35	9	0.99	
17	0	0	+1	55	9	1.76	
18	0	-1	0	55	6	0.99	
19	+1	0	0	75	9	0.99	
20	0	0	0	55	9	0.99	

This table indicates the complete experimental design matrix of CCD for the factorial design. Order in which the runs were made was randomized to avoid errors which are caused by systematic.

4. Results and Discussions

4.1.Extraction of Oil

The oil was extracted using mechanical pressing and hexane solvent. The required amount of oil was extracted using mechanical pressing to reduce the cost; even though the extraction efficiency of mechanical pressing was poor as it results some residue and the oil is get rid of together with the cake. From 100 g of purified seed, 35 g oil was found, that shows 35 %(w/w) of oil was extracted in the seed by using hexane. And by using mechanical pressing, from 100 g of purified seed, 20 g oil was found, that shows 20 %(w/w) of oil was extracted in the seed.

Table4.1: Physicochemical properties of the oil

Physicochemical properties	Units	Values
Density @ 20°C	kg/m^3	880
Kinematics viscosity @ 40°C	mm^2/s	12.84
Acid value	mgKOH/g	4.48
Free fatty acid	mg/g	2.24
Saponification value	mgKOH/g	153.52
Flash point	°C	205
Moisture content	% (w/w)	0.375
Ash content	% (w/w)	0.04

4.2. Biodiesel Production and Yield Analysis for One Factor Experimental Design

4.2.1. Effect of reaction temperature on biodiesel production

Setting reaction time and mixing rate were 2 hours and 500 rpm, respectively for all the runs. The effect of temperature at 35°C, 55°C and 75°C on biodiesel yield for 1.125% (w/w) catalyst, 100 ml oil feed and 9:1 ratio of alcohol to oil is indicated in Table4.

Table 4.2.1: Effect of temperature on biodiesel yield

Run number	Temperature(°C)	Biodiesel Yield%(w/w)
1	35	70
2	55	87
3	75	53

Biodiesel yield was directly proportional to temperature from 35°C to 55°C and inversely proportional from 55°C to 75°C. The yield obtained at 75°C was as small as the formation emulsion was facilitated. Thus, the maximum and minimum yield was obtained at a temperature of 55°C and 75°C, correspondingly.

4.2.2.Effect of catalyst amount on biodiesel production

Setting reaction time and mixing rate were 2 hours and 500 rpm, respectively for all runs, the effect of catalyst at 0.25% (w/w), 1.125% (w/w) and 2.0 % (w/w) sodium hydroxide on biodiesel yield at 55°C temperature, 100ml oil feed and 9:1 ratio of alcohol was indicated in Table 5.

Table 4.2.2: Effect catalyst amount on biodiesel yield

Run number	Catalyst %(w/w)	Biodiesel Yield%(w/w)
1	0.25	54
2	1.125	87
3	2.0	53.5

Biodiesel yield was directly proportional to catalyst amount from 0.25% (w/w) to 1.125% (w/w) and inversely proportional from 1.125%w/w to 2.0% (w/w). As catalyst amount increases further above the optimum amount the yield reduced as a result of formation of the soap and emulsion. Thus, biodiesel yield was achieved maximum for a catalyst amount of 1.125% (w/w) while the minimum yield was obtained at 2.0% (w/w) catalyst amount.

4.2.3. Effect of molar ratio of alcohol to oil on biodiesel production

Setting reaction time and mixing rate were 2 hours and 500 rpm, respectively for all runs, the effect of molar ratio of alcohol to oil of 6:1, 9:1and 12:1 for 1.125% (w/w) catalyst, 100ml oil feed and at a temperature of 55^{o}C on biodiesel production is shown in Table6.

Table4.2.3: Effect Molar ratio of alcohol to oil on biodiesel yield

Run number	Molar ratio of alcohol to oil	Biodiesel Yield%(w/w)
1	6:1	74
2	9:1	87
3	12:1	78

4.3.Statistical Analysis on Factors Affecting Biodiesel Yield

Experimental design was selected for the statistical analysis of the study by selecting Central Composite Design (CCD) and the response measured is the yield of biodiesel or fatty acid methyl esters (FAME). The three trans esterification process factors chosen to be studied were reaction temperature, ratio of ethanol to oil and weight of catalyst. Regression analysis and analysis of variance (ANOVA) was done by using Design Expert 7.0.0 program. The software program was used to generate surface plots, using the fitted equation obtained from the regression analysis, keeping one of the independent variables constant. Response of the transesterification process was used to develop a mathematical model that correlates the yield of biodiesel to the trans esterification process variables studied. Design Expert software version 7.0.0 was used for the regression analysis of the experimental data and also for evaluation of the statistical significance of the equation developed. The central composite design results and responses, and the statistical analysis of the ANOVA are given in Tables4.3.1 and 4.3.2, respectively. The actual yield of biodiesel produced at different process parameters. The model was tested for adequacy by analysis of variance.

Biodiesel yield %(w/w)= Mass of biodiesel Produced/Mass of oil feed

Table4.3.1: Experimental values of biodiesel yield

Run	Temp. (^{0}C)	Ethanol to oil	Catalyst (g)	Biodiesel (ml)	Density of biodiesel(kg/m^{3})	Actual biodiesel yield %(w/w)	Predicted biodiesel yield %(w/w)	Residuals %(w/w)
1	35	12	1.76	65	842	62.2	60.4	1.8
2	75	12	0.22	64	841	59.2	55.84	3.36
3	55	9	0.99	88	840	84	84.51	-0.51
4	35	6	0.22	57	839	54	57.26	-3.26
5	55	9	0.99	89	840	84.9	84.51	.39
6	75	6	1.76	61	843	58.4	54.99	3.41

7	55	9	0.99	90	840	85.9	84.51	1.39
8	35	6	1.76	59	838	56.2	59.41	-3.21
9	75	12	1.76	50	841	52.0	54.99	-2.99
10	55	9	0.99	91	840	87.0	84.51	2.49
11	35	12	0.22	63	842	58.8	57.26	1.54
12	75	6	0.22	60	839	57.2	55.84	1.36
13	55	9	0.99	90	842	85.9	84.51	1.39
14	55	12	0.99	83.7	841	79.8	80.51	-0.71
15	55	9	0.22	68	843	65.2	66.70	-1.50
16	35	9	0.99	76	842	72.7	70.06	2.64
17	55	9	1.76	56	838	53.3	51.8	1.50
18	55	6	0.99	78	842	74.6	73.51	1.09
19	75	9	0.99	89	840	54.5	56.8	-2.30
20	55	9	0.99	91	840	87.0	85.51	1,49

From Table4.3.1, the maximum yield was 87.0 % (w/w), at experiment number 20 and10, while the minimum yield was 52 % (w/w), at experiment number 9. Also from the table experiment numbers 3, 5, 7, 13, 14 and 19 were maximum amount of yield gained. Therefore it was concluded that the maximum amount of biodiesel yield was gained at 1.125% (w/w) of catalyst, 9:1molar ratio of ethanol to oil and 55^{0}C. The minimum yield was obtained at a temperature of 75^{0}C, 2% (w/w) catalyst and 12:1 molar ratio of alcohol to oil, at experiment number 9.

4.3.1.Effect of Interaction between Process Variables

The process variables were found to have significant interaction effects. Figure 4.3.1, 4.3.3 and4.3.5 shows the interaction between catalyst weight and reaction temperature, Weight of catalyst and ethanol to oil molar ratio and reaction temperature to ethanol to oil molar ratio, respectively, on the yield of biodiesel yield. Generally, an increase in reaction temperature is found to increase the yield of biodiesel up to some optimal value in all three cases. Additionally it was observed that at lower range of reaction temperature, higher weight of catalyst and higher molar ratio of ethanol to oil, always resulted in higher yield than when using lower weight of catalyst and lower ratio of ethanol to oil.Reactions which were carried out using lower ratio of ethanol to oil and lower weight of catalyst is found to have higher yield as compared to reactions using lower reaction temperature, higher molar ratio of ethanol to oil and higher weight of catalyst. However, at higher range of reaction temperature, the observations showed that using a combination of both, higher reaction temperature and higher molar ratio of ethanol to oil or higher weight of catalyst used is not beneficial in increasing the yield of biodiesel. This is probably because at these conditions, the higher reaction temperature is already sufficient to push the reaction forward. This phenomenon is further supported by the fact that reaction temperature is the most significant process variable that affects the yield of the biodiesel as indicated by graphs.

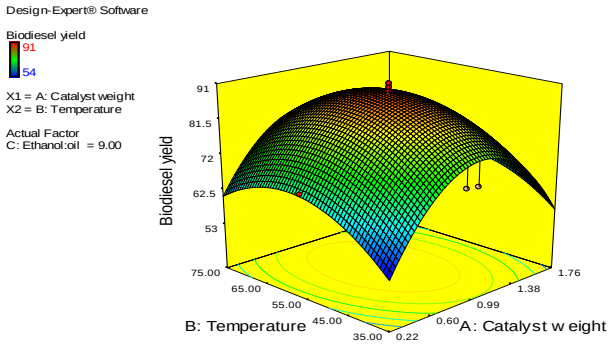
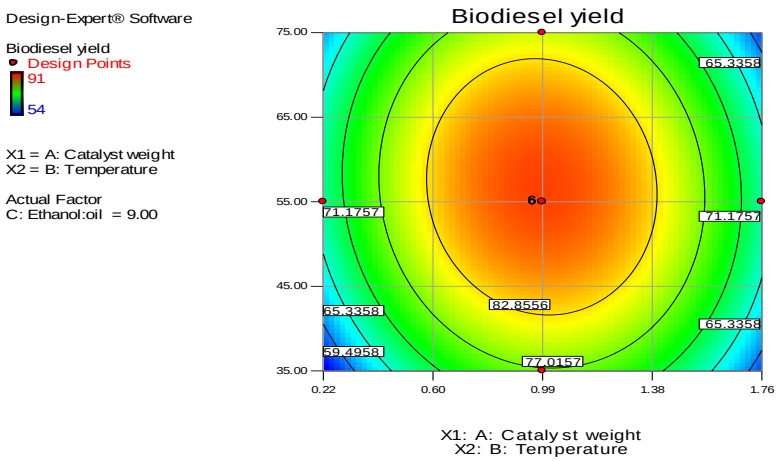


Figure4.3.1: Surface plot of the interaction effect of temperature and catalyst weight versus biodiesel yield



Figur4.3.2: Contour plot of the interaction effect of temperature and catalyst weight versus yield

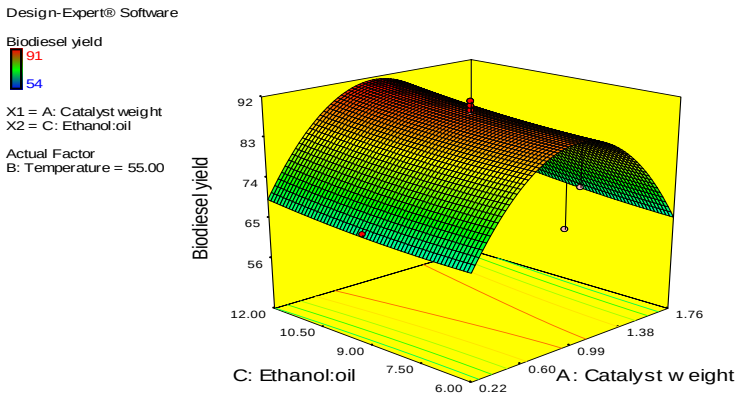


Figure4.3.1: Surface plot of the interaction effect of catalyst weight and ethanol to oil molar ratio versus biodiesel yield

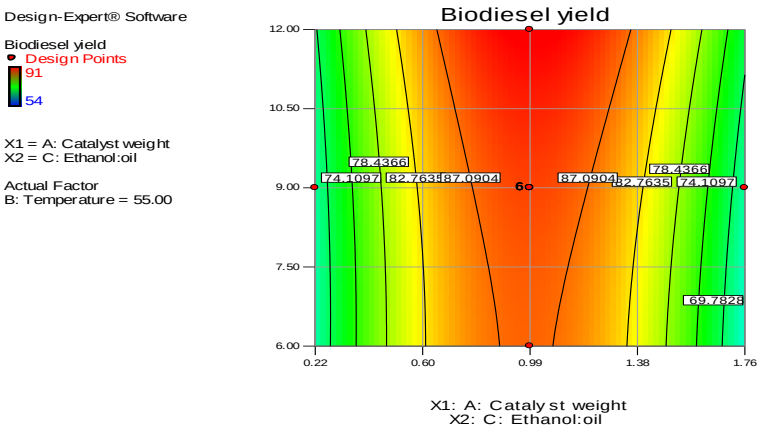


Figure4.3.4: Contour plot of the interaction effect of catalyst weight and ethanol to oil molar ratio versus biodiesel yield.

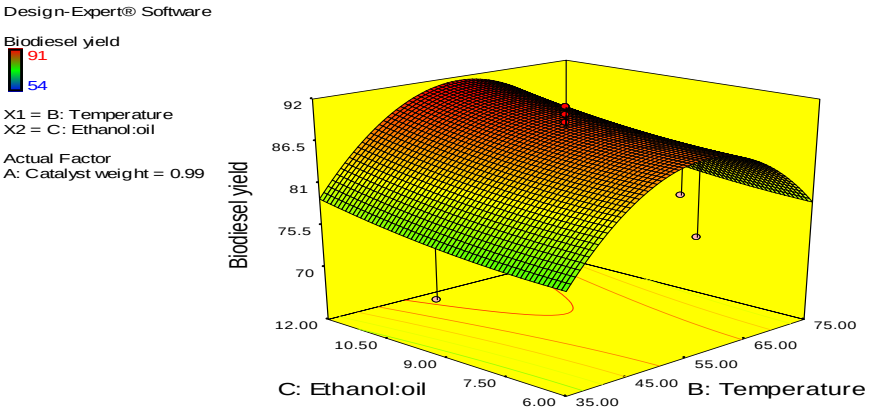


Figure4.3.2: Surface plot of the interaction effect of temperature and ethanol to oil molar ratio versus on biodiesel yield.

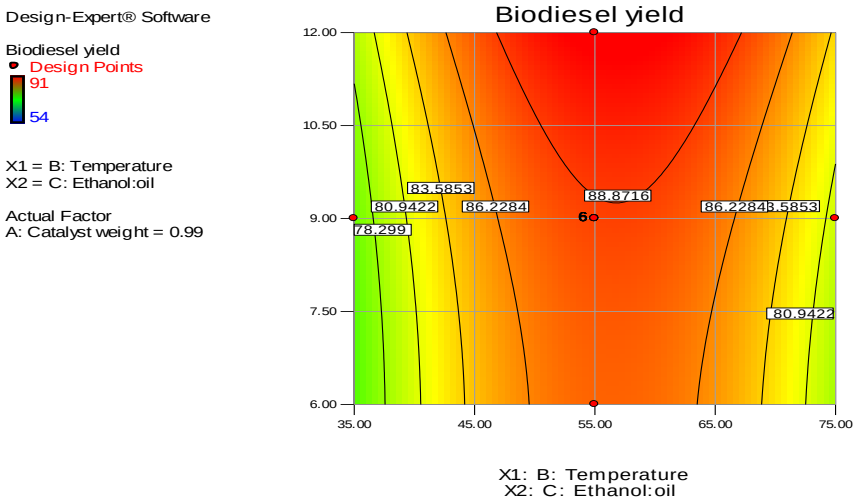


Figure4.5.3: Contour plot of the interaction effect of temperature and ethanol to oil molar ratio versus on biodiesel yield

Figure 4.3.5 show the interaction between ethanol to oil molar ratio and weight of catalyst used, respectively on the yield of biodiesel. From Figure 4.3.3, the percentage of biodiesel amount increased with increasing catalyst concentration at a low ethanol to oil molar ratio. From Figure1, the percentage of biodiesel amount increased with the increasing ethanol to oil molar ratio for a low reaction temperature.

4.3.2.Comparison of Biodiesel Physicochemical Properties against Standards

Comparisons of biodiesel physicochemical properties against standard specifications are given in Table8.

Table4.3.2: Comparison of biodiesel physicochemical properties against standards

Biodiesel properties	Measured values	ASTM Standard
Density @ 20°C (kg/m^3)	839-843	875-900
Kinematic viscosity @40°C (mm^2/s)	2.90 -4.50	1.9-6.0
Flash point (°C)	132 – 150	$\geq$130

Acid value (mgKOH/g)	0.68 -0.89	≤0.8
Saponification value (mgKOH/g)	115.5-124.5	-
Moisture content % (w/w)	0.011 -0.019	< 0.03
Ash content% (w/w)	0.019 -0.029	< 0.02
Iodine value (I_2g/100g)	-	≤120
Cetane number	-	≥47

Properties of biodiesel were under standard specification of ASTM. Iodine value and cetane number were not done due to lack of reagent chemicals and equipment. The lower density fuel burns quickly and consumed immediately while higher density fuel burns for longer time. The lower flash point fuel is more favor for spontaneous ignition while it is transported or stored for longer time. However higher flash point fuel resists such problems [23].

5.Conclusions

Biodiesel was produced using sodium hydroxide and ethanol alcohol catalyst at constant reaction time of 2 hours, mixing rate of 500 rpm and at atmospheric pressure. Production of biodiesel was performed by batch process system. The effects of amount of sodium hydroxide catalyst, reaction temperature and molar ratio of alcohol to oil on biodiesel yield were determined. By using Design Expert 7.0.0 soft ware three levels; three factor Central Composite Design with full type, when reaction temperature, catalyst amount and molar ratio of alcohol to oil were increased, the biodiesel yield increased until the optimal amount. However, further addition of these working variables during trans esterification reaction results in reduction of biodiesel yield due to formation of emulsion which made difficulty in the separation of biodiesel from glycerol.

Additionally, it was observed that the biodiesel yield has a quadratic response with temperature and catalyst weight and linear response with alcohol to oil ratio. From these three parameters and their interaction effects, the highest effect on biodiesel yield was observed due to molar ratio of alcohol to oil with both catalyst weight and temperature while the effect of temperature and catalyst weight was small relatively. The interaction effect of the three operating parameters was significantly detected on biodiesel yield. The maximum biodiesel yield was attained at a temperature of 55^{o}C, 1.125% (w/w)NaOH catalyst amount and for 9:1 molar ratio of alcohol to oil. In contrast, the average minimum biodiesel yield was at 75^{o}C, 12:1 molar ratio and 2% (w/w) catalyst amount.

The physicochemical properties of the biodiesel were determined, within ASTM standard values. Hence, the produced biodiesel can be used as an engine fuel. Based on an existing production process and using current best values for reagent, equipment, and supply costs an economic analysis it is suggested that the production cost of vernonia galamensis biodiesel would be approximately Birr 10.00birr/l. Thus, the preliminary economic analysis evaluation suggested that the project is feasible. Therefore, vernonia galamensis can be used as a less expensive supplementary feedstock for biodiesel production by curing the environment and increasing the agricultural earning.

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