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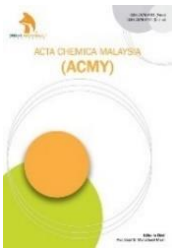

RESEARCH ARTICLE

DEVELOPMENT AND EVALUATION OF BIOGREASE FROM BIOBASED OIL USING PALM BUNCH LYE, AND *MORINGA OLEIFERA* LEAF

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ABSTRACT

In this study, the chemical modification of glycerin; from cottonseed oil to grease, a lubricant of economic significance, and the utilization of lye, a bio alkali from a waste product of plant origin has been explored. Glycerin was obtained through transesterification of cottonseed oil, characterized and designed using Fourier transform infrared spectrophotometry FTIR and Response Surface Methodology (RSM). Samples of bio-alkali and *Moringa oleifera* extract were prepared and characterized using FTIR. The produced grease samples were characterized using FTIR. The performance of the grease samples were analyzed based on their dropping point, BOD, water and corrosion. The functional groups of glycerin include polar atoms, single and double bond structures, which show that the glycerin is good for lubricating grease. The *Moringa oleifera* leaf was found to be rich in alkaloids, flavonoids, and a high number of saponins. Functional groups of lye and *Moringa oleifera* show suitable thickening and corrosion inhibitory properties respectively. Experimental glycerin yield was obtained as 49.01%. Dropping points of 167.77°C and 166.32°C were obtained for grease samples produced using lye and NaOH soaps respectively. meanwhile 14.94 ppm, and 17.67 ppm were the corresponding respective BOD values. Comparative analysis of the grease parameters showed that grease with a thickener of palm residue competes favorably with that of NaOH as a thickener. The quadratic model effectively describes the glycerin yield. The point at which it drops is able to handle temperatures produced with gearboxes, and the bio-alkali grease's consistency is within ASTM standards, making it appropriate for a variety of applications.

KEYWORDS

FTIR, Grease, Lye, Moringa Oleifera Extract, Bio-Lubricant.

1. INTRODUCTION

The current global trend in the utilization of more eco-friendly lubricating grease is spurred by the need to maintain a sustainable environment. Also, heavy emphasis is being placed on its affordability, efficiency, in addition to toxicity, and biodegradability (Kimura et al., 2003). Greases have been regularly applied to moving hardware for a very long time, for reasons including: heat expulsion, contaminant evacuation, friction reduction, corrosion prevention, and contaminant elimination. The primary purpose of greases is to significantly reduce wear between moving components. According to the consensus organization ASTM International, grease is characterized by its thickener composition, base fluid type, and additional material content. It is characterized as a semi-fluid to solid product of a thickening agent dispersed in a liquid lubricant base. Mineral oil, often referred to as synthetic oil, and bio-based oil are the three main categories into which lubricating grease base fluids fall (Sander et al., 2013). Petroleum-based oil is harmful to the environment and a major source of pollution; the use of oil-based lubricants has sparked debate about the viability and impact of oil-based sources on the economy and environment.

Greases made from mineral oil base fluid and synthetic soap thickeners can potentially endanger the environment because of the high potential emission of such pollutants as heavy metals, particulate matter, and chemical additives during use. One litre of mineral lubricating oil can pollute a million litres of water. These harmful emissions pose a threat to both humans and their environment, owing to their high level of toxicity and persistence in nature. Their release has led to such ripple effects as: an elevation in the concentrations of the earth's greenhouse gases, global warming, economic meltdown, disease pandemics, mutations, and ecological extinction, or loss of biodiversity, amongst others. The use of alternative metal soap thickeners in the grease industry, as well as the replacement of lubricants of mineral origin, is on the rise, due to the depletion of the main fossil fuels and the polluting effects of these and their derivatives on the environment (Manuel et al., 2015). Also worthy of consideration are the increasingly strict regulations imposed by regulatory bodies on the use of certain chemicals and substances due to their negative environmental and health impacts.

Past literature reviews on the synthesis and use of bio-lubricants reported them as superior alternatives to their conventional counterparts. Among the many strong attributes of bio-based lubricants are their renewability, environmental friendliness, ease of accessibility, availability, and their degradable nature. Researchers have reported employing fumed silica (FS), spent bleaching earth (SBE), and waste cooking oil (WCO) to create biodegradable greases from palm oil industry wastes (Abdulbari et al., 2018). Thermal stability, decomposition temperature, penetration, corrosivity on a copper strip, dropping point, friction coefficient, and other properties of the various grease formulations were assessed using standard techniques, and the grease formulations with and without the

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additive were compared. The findings indicate that, although the addition of fumed silica decreased the quantity of SBE and increased the amount of base oil, the grease without fumed silica needed a high percentage of SBE (up to 80% w/w). Fumed silica eliminated the dropping point, raised the decomposition temperature, made the grease mildly corrosive, and increased the grease's penetration number by one standard (NLGI standard). Without fumed silica, the formulated grease's average friction coefficient was 0.095, while, with fumed silica, it was 0.11

The overall results show that SBE and WCO can be used to formulate grease and that fumed silica can increase the performance of the formulated grease. A studied bio-grease made from sugar cane filter cake vegetable oil, sodium soap, and molybdenum disulphide as an additive (Pedro et al., 2017). A group researcher investigated the production of bio-grease from black-date *(Canarium schweinfurthii) (*Awoyale et al., 2011). The production conditions, measurement standards, and outcomes for their experiment were: water temperature (60–70°C) for extracting black-date flesh from the seed; 100°C, 170°C, and 210°C for grease production. Cone penetration of grease (consistency) results met the ASTM D217-IP50 standard, with the maximum generated grease dropping point being near that of Oando grease (control). The BOD test results were within the permitted range of 4.0–8.0.

The purpose of this study is to investigate the synthesis and characterization of bio-grease made from palm bunch lye solution as a thickener, cottonseed oil as a lubricant base, and Moringa oleifera extract as a corrosion inhibitor. The shift towards bio-based lubricants and soaps in the production of grease reflects a broader transition towards more sustainable and environmentally friendly practices in the industrial sector. The use of these agricultural feedstocks will not pose the environmental concerns that arise from the utilization of mineral oil and synthetic soaps for grease production. Due to the low cost, ease of accessibility, renewability, and affordability of the raw materials, the success of this work has added to the list of raw materials that can be used in the production of a eco-friendlier product of high economic value.

2. Materials and Methods

2.1 Oil, Moringa oleifera leaf, waste palm bunch source

The cottonseed oil used for this study was procured commercially in the market. Waste palm bunch and Moringa oleifera leaf were obtained from the Agro farm, Awka, Anambra State.

2.1.1 Chemicals

Methanol, diethylene glycol, polyethylene oxide, monoethylene glycol, ammonium hydroxide, acetic acid, ethanol, H_2SO4, $AlCl_3$, $BaCl_2$, $FeCl_3$, NH_4OH, NaOH, sodium chloride, hydrochloric acid, and distilled water.

2.1.2 Equipment

UV-spectrometer, separating funnel, beakers, stopwatch, weighing balance, water bath with thermostat, viscometer, glass measuring cylinder, electronic weighing balance, conical flask and cork, Erlenmeyer flask, filter paper, spatula, retort stand, oven, volumetric flasks, Soxhlet extractor, and condenser.

2.2 Methods

2.2.1 Extraction of lye from palm bunch

The standard method used was employed to obtain lye from empty palm fruit bunches (Undiandeye et al., 2015). Five grams of the ash sample was dissolved in 100 ml of distilled water in a beaker. The resulting solution was continuously stirred for one hour with a magnetic stirrer, and proper absorption was allowed for 12 hours. The solution was then filtered using filter paper, and a clear extract was obtained. Drops of methyl orange indicator were added to the fluid until an orange coloration was observed. The molarity of the pure alkali extract was found to be 0.23 M. The extract was made up to 1000 ml using distilled water in a 1000 ml volumetric flask. Two hundred millilitres of the extract was mixed with 5 ml of 12% $BaCl_2$ and titrated against 0.5 M HCl acid via phenolphthalein (as an indicator) until the endpoint was attained.

2.2.2 Extraction of Moringa oleifera leaf extract

The approach by was used to obtain the Moringa oleifera plant extract (Omotioma and Onukwuli., 2019). Thirty-five grams of pulverized Moringa oleifera leaf in 1000 ml of ethanol was steeped for 48 hours before filtering. Evaporating the ethanol from the mixture produced a concentrated filtrate. A test tube was filled with 1 g of solvent-free leaf extract to identify alkaloids. A few drops of diluted hydrochloric acid were added, mixed, and filtered. Alkaloid reagents Dragendorff's reagent (orange precipitate) and Meyer's reagent (cream precipitate) were used to thoroughly evaluate the filtrate. To identify cardiac glycosides, a vessel was filled with 1 ml of the sample, 5 ml of water, and 2 ml of glacial acetic acid. One drop of $FeCl_3$ was added, followed by 1 ml of concentrated H_2SO4, resulting in a brown ring. To determine flavonoids, 1 g of the sample was mixed with 25 ml of water and heated for 15 minutes at approximately 100°C in an oven.

Two millilitres of NH_4OH were added to 2 ml of the sample, followed by 1 ml of concentrated H_2SO4. Flavonoids were present due to the emergence of a yellow color. Adding a few drops of a 1% (w/v) ferric chloride solution and then 1% (w/v) gelatin in sodium chloride at the same concentration allowed researchers to detect the presence of phenols. Phenols were present because a precipitate formed. To determine the amount of saponins present, 1 g of the sample was cooked in 4 ml of water before being filtered. After shaking 10 ml of the filtrate vigorously, froth started to develop. Three drops of oil were added, and the mixture was shaken again. To determine tannin content, 1 g of the sample was mixed with 25 ml of water. After 15 minutes at 100°C, 1 ml of the sample was added to 10 ml of water, and the mixture was brought to a boil. A few drops of 0.1% FeCl3 were added. Tannins were present because a green tint developed.

2.2.3 Glycerin production process

A transesterification process was utilized for glycerin production. One hundred grams of cottonseed oil was placed in a measuring vessel, pre-warmed with an electric heater to approximately 55°C and transferred to another conical flask. One and a half grams of NaOH (catalyst) was measured and completely dissolved in the required amount of methanol using a hot plate and magnetic stirrer to form a sodium methoxide solution. The prepared mixture of NaOH and methanol from the conical flask was carefully poured into the oil. The combination of oil, NaOH, and methanol was covered, placed on a magnetic stirrer, and turned on. Agitation in the magnetic stirrer was maintained for 55 minutes at 250 rpm. The reaction mixture was poured from the conical flask into a separating funnel. This was allowed to stand overnight (12 hours) while phase separation occurred by gravity settling. The biodiesel was separated from the glycerin. The yield of glycerin indicates the amount of glycerin produced relative to the volume of oil used.

2.2.4 Preparation of bio-thickener

Twenty-five weight percent of palm bunch lye was added to distilled water to make the thickener and heated. A magnetic stirrer was used to continuously swirl the palm bunch lye as it dissolved in the solution. The mixture was heated to 110°C for two hours without stirring to remove the water from the thickener. After that, it was cooled to room temperature. The same procedure was followed to prepare the sodium hydroxide thickener.

2.2.5 Production of grease.

Standard method used was used in the production of grease (Huei et al., 2020). A sample of grease with the concentration of the thickener (palm bunch lye solution) at 12% (w/v) of the total volume of glycerin was prepared. Initially, 50% of the total volume of glycerin was charged in the reaction vessel and heated up to 90°C. Then 50% of the palm bunch lye solution was added and stirred vigorously for intimate mixing. The temperature was gradually increased to 160°C to let the palm bunch lye solution be fully mixed in the cottonseed oil. At this temperature; 160°C, another 50% glycerin and 50% thickener (palm bunch lye solution) were added along with rigorous stirring for 3 hours. The temperature was lowered gradually until it reached room temperature whereas the viscous glycerin turn into semisolid form grease". In the experiments of grease production, 1g of *Moringa oleifera* extract was added to the grease formulation. Also, for the production of NaOH soap grease, the exact method above was followed with equally the addition of 1g of Moringa Oleifera extract.

2.3 Phytochemical, physicochemical, and FTIR analysis

For characterization of the cottonseed oil, standard methods were used to determine the density of the oil, saponification value (SV), free fatty acid (FFA), iodine value, acid value, and peroxide value, and their results were tabulated. Standard methods used by previous authors were adopted for the phytochemical analysis of the Moringa oleifera leaf extract. Glycerin, Moringa oleifera, lye, and grease samples were characterized using FTIR (Mada et al., 2012; Omotioma et al., 2019).

2.4 RSM experimental design matrix

For response surface methodology (RSM), the central composite design of Design-Expert software was employed to design the experiment. Multiple regression was used to fit the coefficients of the response's polynomial model to associate the response variable with the independent factors. Analysis of variance (ANOVA) and a test of significance were used to assess the model's fit quality. The fitted quadratic response model is described as follows:

$$Y = b_0 + \sum_{i=1}^{k} b_i x_i + \sum_{i=1}^{k} b_{ii} x_i^2 + \sum_{i<j}^{k} b_{ij} x_i x_j + e \quad (1)$$

Where;

Y is the response variable (sesame biodiesel yield), bo is the intercept value, bi (i = 1, 2,..., k) is the first-order model coefficient, bij is the interaction effect, and bii represents the quadratic coefficients of Xi, and e is the random error.

2.5 Performance Test

2.5.1 Dropping points of the grease.

The lowest temperature at which oil will flow and the phase of grease transitions from semi-solid to liquid is known as the dropping point, and it serves as a gauge of the grease's heat resilience. The ASTM D566 standard procedure was used to calculate this temperature. It was determined that the temperature at which the initial decrease is noticeable is the dropping point.

2.5.2 Test for Biodegradability of the grease.

The quantity of oxygen that microorganisms consume to break down the organic matter in a sample over time and at a certain temperature is known as the biochemical oxygen demand, or BOD (Awoyale et al., 2011). The usual time frame and temperature needed for this test were five days and 20°C, respectively. Twenty millilitres of the generated grease, designated 1, 2, and 3, were combined with pond water containing microorganisms in incubation bottles, each of which was sealed to prevent light from passing through. Twenty millilitres of mineral grease (Controls) were also added to two additional incubation bottles; one litre of distilled water was combined with one millilitre of each of the following: calcium chloride, magnesium sulphate, phosphate buffer, and iron (ii) chloride.

An oxygen meter was used to measure the amount of dissolved oxygen after the prepared diluted water was introduced to each incubation container and left at room temperature for about two hours. Following five (5) days of maintaining the incubation bottles at the same temperature of 20 °C, the dissolved oxygen content was measureable. We computed and documented the variations in the amount of oxygen dissolved.

The biodegradability of the grease produced was calculated using Equation 2 and 3:

$$BOD = \frac{DOi-DOf}{P} * \frac{300}{1000} \quad (2)$$

$$P = \frac{v}{1000} \quad (3)$$

where P is the sample percentage, v is the sample volume (cm3), DOi is the initial dissolve oxygen, DOf is the final dissolve oxygen after five days, and BOD (ppm) is the biochemical oxygen demand.

2.5.3 Water resistance of the grease.

The ASTM D1264 standard procedure was used to determine that water could effectively cleanse greases. This standard test procedure is used to ascertain if grease lubricants on bearings are stable against water washing. Three duplicates of this exam were conducted.

2.5.4 Determination of corrosion resistance of the grease using thermometric technique.

With minor adjustments, the approach taken was implemented (Onukwuli et al., 2016). A water bath with a thermostat set to 30 °C was used for the measurements. Until a constant temperature value was achieved, the temperatures of the system containing the metal and the corresponding test solution were routinely recorded. The reaction number (RN) was evaluated using Equation:

$$RN = \frac{T_m-T_i}{t} \quad (4)$$

Where;

T_m and T_i are the maximum and initial temperatures (in ^{0}C) respectively, and t is the time in minutes elapsed to reach T_m.

The inhibitor efficiency was determined using Equation:

$$IE\% = (1 - \frac{RN_{add}}{RN_{free}}) * 100 \quad (5)$$

Where;

RN_{free} and RN_{add} are the reaction numbers for the free and inhibited corrosive media respectively.

3. Results and Discussion

3.1 Physicochemical Analysis of Cotton Seed Oil.

Physiochemical properties of cotton seed oil is shown in Table 1. Based on ASTM D 2270, The viscosity index (VI) of 211, significantly higher than that of a comparable mineral oil (VI = 95), indicates superior thermal stability, making the oil less susceptible to viscosity changes with temperature variations. With stable viscosity, good oxidative stability, and favorable rheological properties, cotton seed oil is suitable for bio-lubricating functions (Spikes et al., 2014).

Table 1: Physicochemical Characteristics of Cotton Seed Oil.

Characteristics	Values
Density (g/cm^3)	0.8979
Specific Gravity	0.965
Kinematic viscosity @40^{o}C(cSt)	31.87
Kinematic viscosity @100^{o}C(cSt)	6.97
Saponification Value (mg/g)	195.4
Acid Value (mg/g)	5.34
Free Fatty Acid (% oleic acid)	2.67
Iodine Value (g/100g)	10.2
Peroxide value (mg/g)	3.25

3.2 Phytochemical analysis of *Moringa oleifera* leaves extract.

Table 2 displays the findings of the phytochemical examination, both qualitative and quantitative, of Moringa oleifera leaves. According to the findings, the extract of Moringa leaves has an undetectable level of tannins along with saponins, alkaloids, and trace amounts of cardiac glycosides, flavonoids, phenol, and phytotates. This outcome is comparable to the experimental findings published by (Kasolo et al., 2010). Previous findings have shown that active substances such flavonoids, tannins, saponins, and alkaloids are responsible for the inhibitory qualities of the majority of green plants (Ekpe et al., 1994; Ebenso, et al., 1996; Odusote, et al., 2013).

Table 2: Phytochemical (Qualitative and Quantitative) Analysis of *Moringa oleifera.*

Phytochemicals	Qualitative analysis	Quantitative analysis
Alkaloids (mg/100g)	++	109.4
Cardiac glycosides (mg/100g)	+	19.5
Flavonoids (mg/100g)	++	214.3
Phenolics (GAE/g)	+	57.2
Phytates (mg/100g)	+	28.5
Saponins (mg/100g)	+++	325.1
Tannins (mg/100g)	-	8.3

+++ (highly concentrated), ++ (concentrated), + (in traces), and – (absent or too little to be observed qualitatively).

3.2.1 Results of the FTIR Analysis of the Moringa Oleifera leaf

Figure 1 shows the results of the FTIR analysis of the *Moringa Oleifera* leaf. The identified functional groups include O-H stretch, N-H symmetric, C-H stretch, C=O. Presence of heteroatoms (N and O) indicates that *Moringa oleifera* leaf extract is a suitable Corrosion inhibitive additive for bio-hydraulic fluid synthesis.

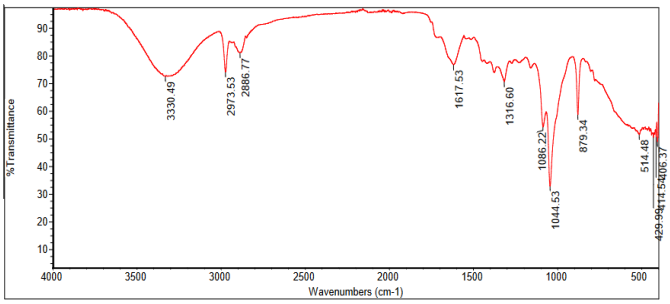

Figure 1: FTIR of Moringa leaves extract.

3.3 Results of the FTIR analysis of lye from palm bunch.

Figure 2 shows the results of the FTIR analysis of lye from palm bunch ash. Every spectrum shows the link between transmittance and wave number with various peaks representing the functional groups. The identified

functional groups include =C-H stretch, C-O, and C-I fingerprint of the alkyl halides. The lye is suitable for thickening agent in grease production.

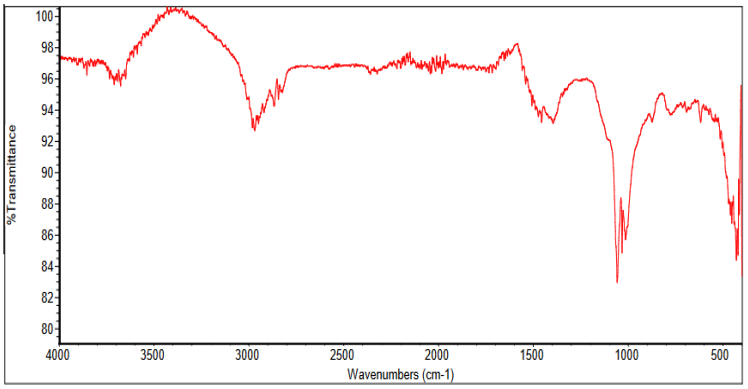

Figure 2: FTIR of lye from palm bunch ash.

3.4 RSM result of the glycerin yield

The RSM result of the glycerin yield is displayed in table 3. It showed how the yield (glycerin) relied on the independent variables (temperature, time, catalyst concentration, and oil/methanol ratio). At a ratio of 3:1, a catalyst concentration of 1.5 weight percent, duration of 80 minutes, and a temperature of 55 degrees Celsius, the highest glycerin yield was achieved as 49.01.

Table 3: Experimental Results for glycerin yield from transesterification of cotton seed oil.

Std	Run	Factor 1 A: Methanol /oil ratio	Factor 2 B: Catalyst dosage (wt%)	Factor 3 C: Temperature (^{0}C)	Factor 4 D: Time (minutes)	Response Glycerin yield %
24	1	3	1.5	55	80	49.01
11	2	1	2.5	45	80	36.72
7	3	1	2.5	65	40	35.94
6	4	5	0.5	65	40	38.58
29	5	3	1.5	55	60	48.75
21	6	3	1.5	45	60	45.98
22	7	3	1.5	65	60	47.99
8	8	5	2.5	65	40	42.64
28	9	3	1.5	55	60	48.75
12	10	5	2.5	45	80	46.53
18	11	5	1.5	55	60	47.51
17	12	1	1.5	55	60	42.87
14	13	5	0.5	65	80	41.13
15	14	1	2.5	65	80	38.63
4	15	5	2.5	45	40	41.33
3	16	1	2.5	45	40	35.44
26	17	3	1.5	55	60	48.75
16	18	5	2.5	65	80	48.01
20	19	3	2.5	55	60	47.63
23	20	3	1.5	55	40	43.94
9	21	1	0.5	45	80	33.99
25	22	3	1.5	55	60	48.75
1	23	1	0.5	45	40	33.42
27	24	3	1.5	55	60	48.75
13	25	1	0.5	65	80	35.19
30	26	3	1.5	55	60	48.75
19	27	3	0.5	55	60	41.14
2	28	5	0.5	45	40	35.00
10	29	5	0.5	45	80	39.25
5	30	1	0.5	65	40	34.61

3.4.1 ANOVA (analysis of variance) for quadratic model of glycerin yield

Table 4 displays the corresponding ANOVA for the glycerin yield quadratic model. According to Table 4 The model F-value of 121.41 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. P-values less than 0.0500 indicate model terms are significant. In this case A, B, C, D, AB, AD, BD, A^2, B^2, C^2, D^2 are significant model terms. The predicted R^2 of 0.9556 is in reasonable agreement with the Adjusted R^2 of 0.9831; the difference is less than 0.2. Adequate precision measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 29.810 indicates an adequate signal. This model can be used to navigate the design space (Omotioma et al., 2021).

Table 4: ANOVA for quadratic model of glycerol yield from cotton seed oil.						
Source	**Sum of Squares**	**df**	**Mean Square**	**F-value**	**p-value**	
Model	**910.31**	**14**	**65.02**	**121.41**	**< 0.0001**	**significant**
A-Methanol/oil ratio	**157.06**	**1**	**157.06**	**293.27**	**< 0.0001**	
B-Catalyst dosage	**91.40**	**1**	**91.40**	**170.66**	**< 0.0001**	
C-Temperature	**12.60**	**1**	**12.60**	**23.53**	**0.0002**	
D-Time	**42.20**	**1**	**42.20**	**78.79**	**< 0.0001**	
AB	**14.12**	**1**	**14.12**	**26.36**	**0.0001**	
AC	**0.7439**	**1**	**0.7439**	**1.39**	**0.2569**	
AD	**9.38**	**1**	**9.38**	**17.51**	**0.0008**	
BC	**0.4389**	**1**	**0.4389**	**0.8196**	**0.3796**	
BD	**2.71**	**1**	**2.71**	**5.07**	**0.0398**	
CD	**0.0008**	**1**	**0.0008**	**0.0014**	**0.9705**	
A^2	**24.44**	**1**	**24.44**	**45.64**	**< 0.0001**	
B^2	**38.93**	**1**	**38.93**	**72.70**	**< 0.0001**	
C^2	**4.22**	**1**	**4.22**	**7.88**	**0.0133**	
D^2	**8.27**	**1**	**8.27**	**15.44**	**0.0013**	
Residual	**8.03**	**15**	**0.5355**			
Lack of Fit	**8.03**	**10**	**0.8033**			
Pure Error	**0.0000**	**5**	**0.0000**			
Cor Total	**918.35**	**29**				
Std. Dev.	**0.7318**		**R^2**		**0.9913**	
Mean	**42.50**		**Adjusted R^2**		**0.9831**	
C.V. %	**1.72**		**Predicted R^2**		**0.9556**	
			Adeq Precision		**29.8098**	

The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients. Final equation in terms of coded factors is given as:

$$\text{Glycerol yield} = +48.51 + 2.95A + 2.25B + 0.8367C + 1.53D + 0.9394AB + 0.2156AC + 0.7656AD - 0.1656BC + 0.4119BD - 0.0069CD - 3.07A^2 - 3.88B^2 - 1.28C^2 - 1.79D^2 \quad (6)$$

3.4.2 Validation and confirmation of results of glycerin

The 3D plots, Figures 4 – 10 presented the RSM graphical results. Each one shows a parabolic curve, typical of a quadratic model. The diagnostic report revealed straight-line (linear) graphs in Figures 3. The points on the graph clustered along the line of best fit. Thus, the experimental data were effectively predicted by the generated RSM model (Omotioma et al., 2021; Patel et al., 2016). A confirmatory analysis based on the response surface optimization of glycerol yield from cotton seed oil was carried out to access the deviation of the experimental yield from the predicted optimum value at the specified process conditions. The result of this validation is presented in Table 5. At a methanol/oil ratio of 3, catalyst dosage of 1.5, temperature of 55°C and reaction time of 60 minutes, a glycerol yield of 48.51% was predicted for the process using the response surface method of optimization. The result of the validation test carried out gave a percentage deviation of 1.24%.

Table 5: Validation of optimum result of the glycerin yield from cotton seed oil						
Methanol /oil ratio	**Catalyst dosage (wt%)**	**Temperature (°C)**	**Time (minutes)**	**Predicted Glycerol yield %**	**Experimental Glycerol yield %**	**Percentage deviation %**
3	1.5	55	60	48.51	49.12	1.24

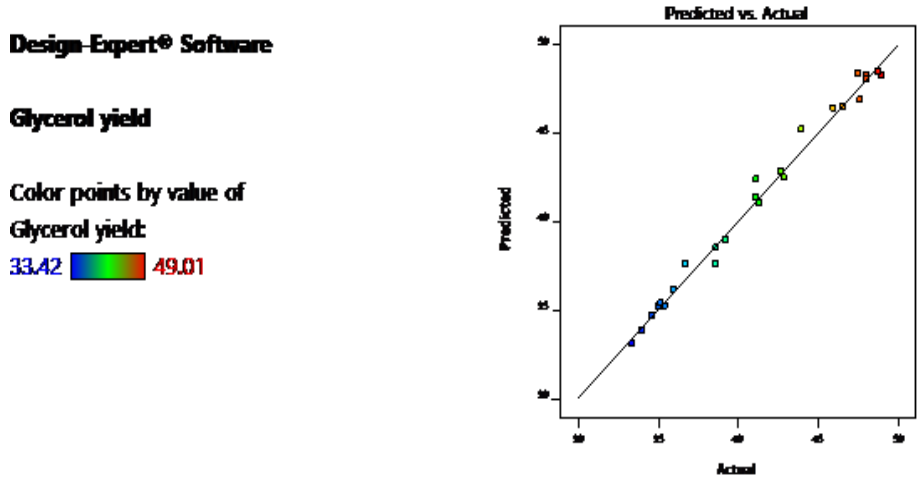

Figure 3: Parity plot of predicted versus actual for glycerol yield from cotton seed oil

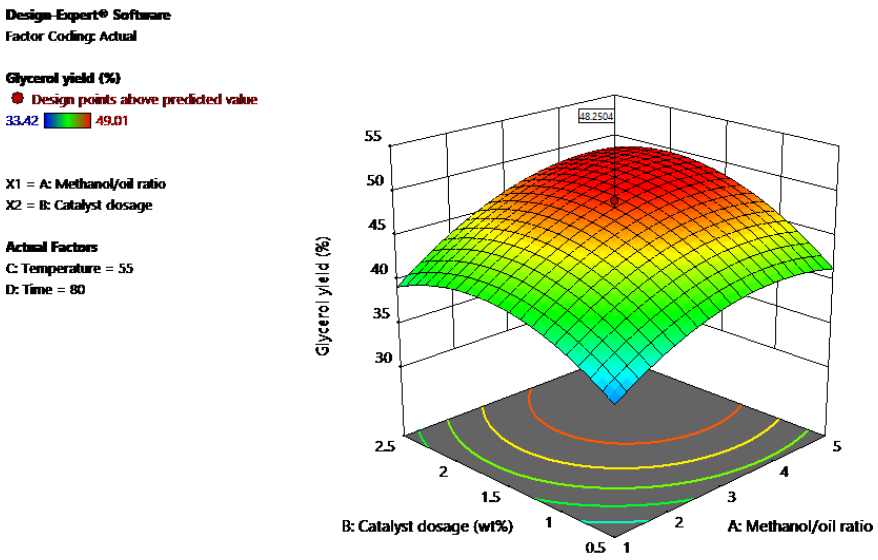

Figure 4: Response surface plot of catalyst dosage and methanol/oil ratio for glycerol yield.

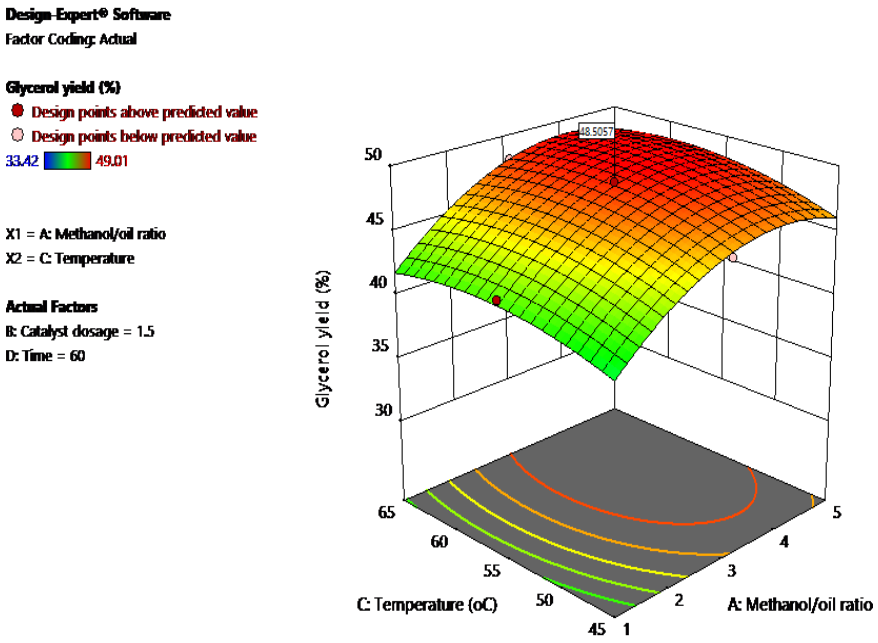

Figure 5: Response surface plot of temperature and methanol/oil ratio for glycerol yield.

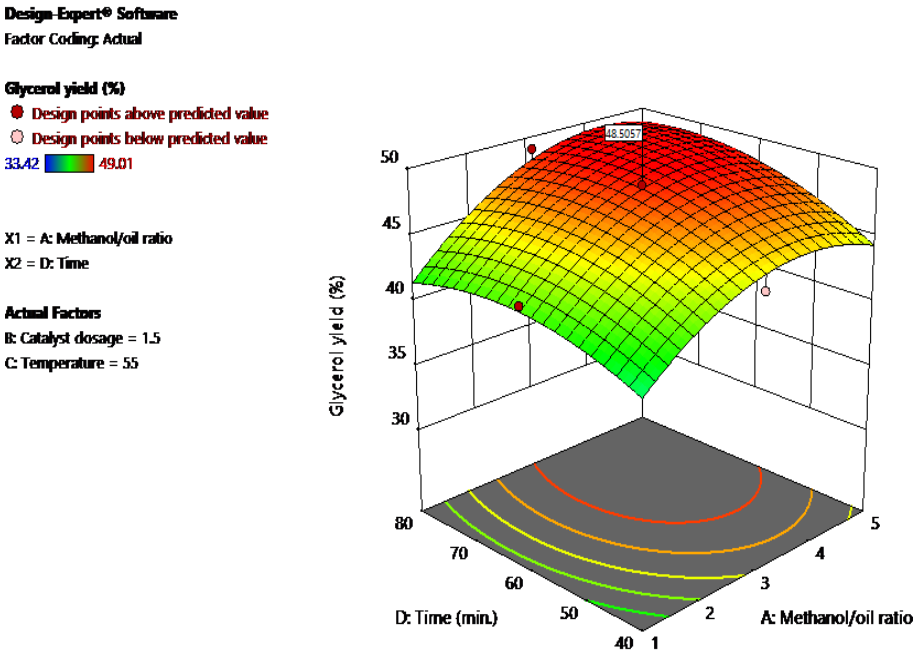

Figure 6: Response surface plot of time and methanol/oil ratio for glycerol yield.

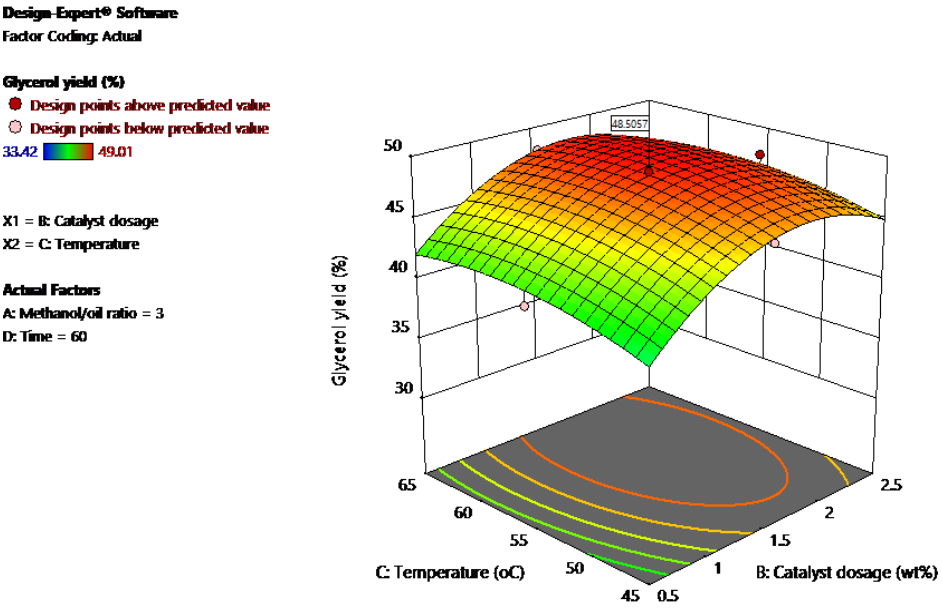

Figure 7: Response surface plot of temperature and catalyst dosage for glycerol yield.

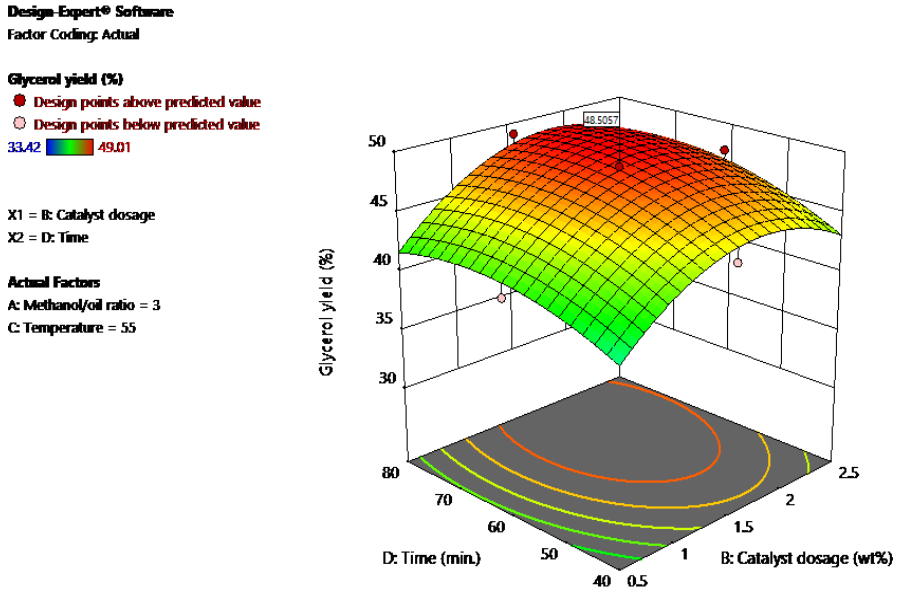

Figure 8: Response surface plot of time and catalyst dosage for glycerol yield.

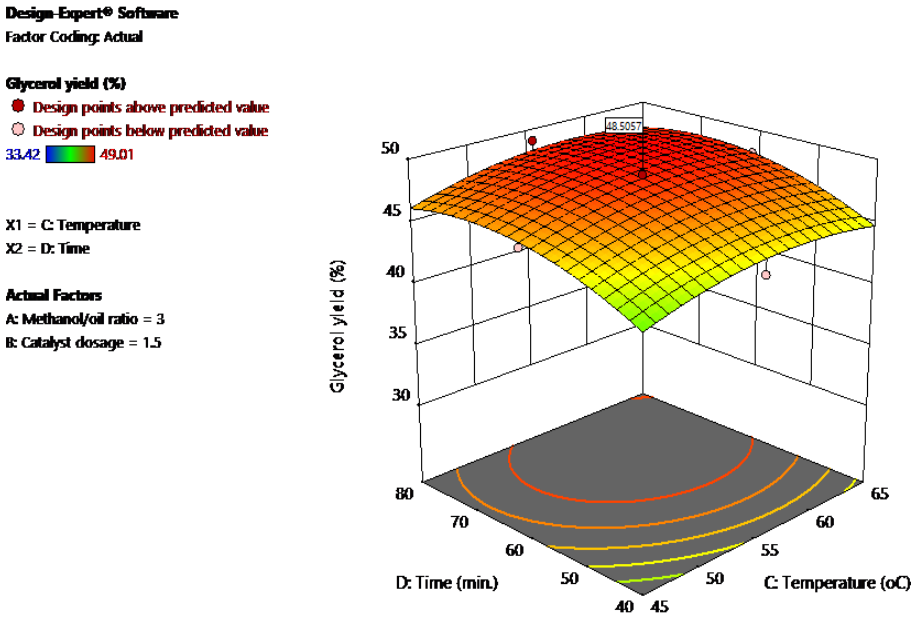

Figure 9: Response surface plot of time and temperature for glycerol yield.

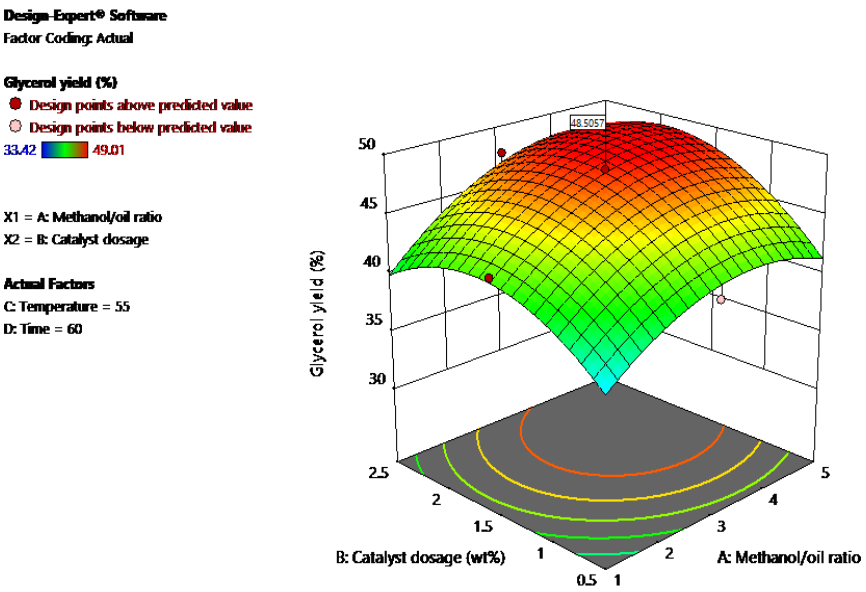

Figure 10: Response surface plot of catalyst dosage and methanol/oil ratio for glycerol yield.

3.5 FTIR Analysis of grease from lye and sodium hydroxide.

Figure 11 and 12 shows the results of the FTIR analysis of of grease produced using lye from palm bunch and Sodium hydroxide as a thickener. Every spectrum shows the link between transmittance and wave number with various peaks representing the functional groups. The identified functional groups include methyl stretch, methyl bends C-C Vibration of methyne, OH stretch, C=O, CN stretch and C-O (Jiabao et al., 2015). Each of them have double bond structures and heteroatoms, hence the grease is suitable for lubricating and allied functions.

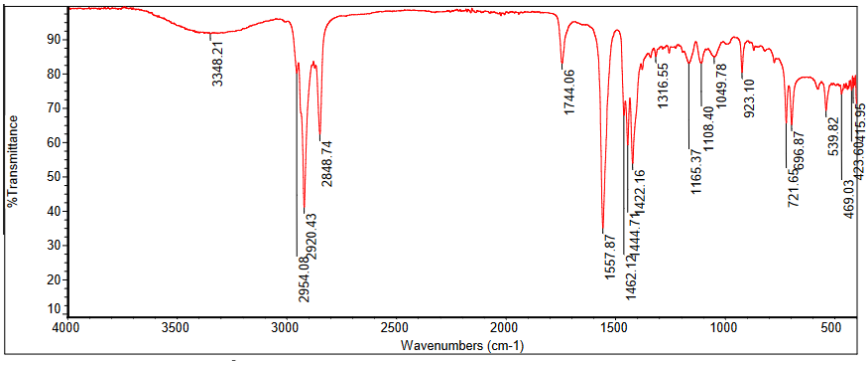

Figure 11: FTIR of grease produced using lye from palm bunch as a thickener.

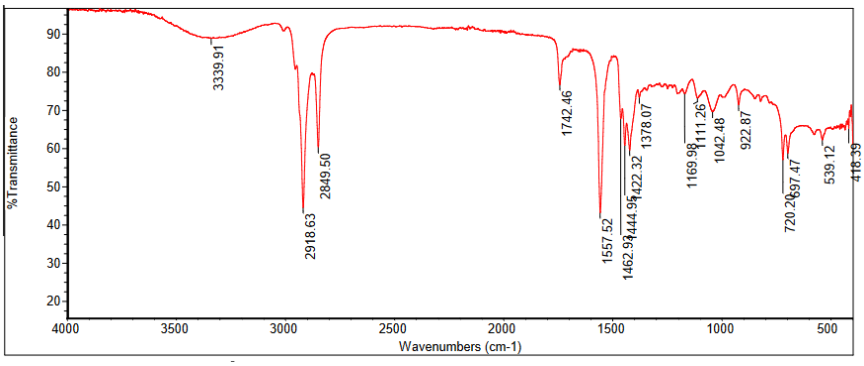

Figure 12: FTIR of grease produced using NaOH as a thickener.

3.6 RSM result of the produced grease

Table 6 and 7 present RSM results of the interactive effects of the variables on the dropping point and BOD of the produced grease. They show the result of the experimental data findings of the dropping point and BOD of the produced grease samples using lye and Sodium hydroxide as thickeners. Each table revealed how the process factors interacted to affect dropping point and BOD of the produced grease. At catalyst dosage of 9, temperature of 160 and time of 180 minutes the highest Dropping point and BOD was achieved as 169.27 and 14.23 for grease using lye as thickener. At catalyst dosage of 9, temperature of 160 and time of 180 minutes the highest Dropping point and BOD was achieved as 166.55 and 17.64 for grease using sodium hydroxide as thickener.

Table 6: RSM results for grease production from cotton seed oil using Lye as thickener.

Std	Run	Factor 1 A: Lye dosage wt%	Factor 2 B: Temperature (⁰C)	Factor 3 C: Time (hr)	Response 1 Dropping point (⁰C)	Response 2 BOD ppm
4	1	15	180	1	140.01	17.56
6	2	15	140	5	157.12	15.06

Table 6 (cont): RSM results for grease production from cotton seed oil using Lye as thickener.						
8	3	15	180	5	144.05	16.18
17	4	9	160	3	169.27	14.23
2	5	15	140	1	144.92	16.10
14	6	9	160	5	169.31	15.64
5	7	3	140	5	143.58	18.00
13	8	9	160	1	151.46	16.34
7	9	3	180	5	134.32	20.05
18	10	9	160	3	169.27	14.23
11	11	9	140	3	163.07	14.25
20	12	9	160	3	169.27	14.23
9	13	3	160	3	143.55	16.22
10	14	15	160	3	161.59	14.80
12	15	9	180	3	163.56	15.39
19	16	9	160	3	169.27	14.23
3	17	3	180	1	128.92	20.25
16	18	9	160	3	169.27	14.23
15	19	9	160	3	169.27	14.23
1	20	3	140	1	123.44	18.17

Table 7: RSM results of the produced grease from cotton seed oil using NaOH as thickener.

Std	**Run**	**Factor 1 A: NaOH dosage wt%**	**Factor 2 B: Temperature (°C)**	**Factor 3 C: Time (hr)**	**Response 1 Dropping point (°C)**	**Response 2 BOD ppm**
4	1	15	180	1	137.42	20.97
6	2	15	140	5	154.51	18.49
8	3	15	180	5	141.44	19.61
17	4	9	160	3	166.55	17.64
2	5	15	140	1	142.31	19.53
14	6	9	160	5	166.71	19.07
5	7	3	140	5	140.97	21.43
13	8	9	160	1	148.85	19.78
7	9	3	180	5	131.71	23.48
18	10	9	160	3	166.55	17.64
11	11	9	140	3	160.46	17.68
20	12	9	160	3	166.55	17.64
9	13	3	160	3	140.94	19.65
10	14	15	160	3	158.98	18.23
12	15	9	180	3	160.97	18.82
19	16	9	160	3	166.55	17.64
3	17	3	180	1	126.31	23.68
16	18	9	160	3	166.55	17.64
15	19	9	160	3	166.55	17.64
1	20	3	140	1	120.79	21.60

3.7 BOD of the grease Samples.

The grease samples biochemical oxygen demand (BOD) test results were recorded for five days at 20°C in a chilled incubator. As shown in table 6 below, the results of these tests were assessed and documented in parts per million (ppm) for grease made from lye and NaOH as thickeners, respectively. The amount of oxygen utilised during aerobic processes of organic material degradation, which are brought on by microorganisms, is represented by the BOD. As a result, the BOD gives information on the percentage of an organic samples content that is physiologically convertible. This prompts the evaluation of these materials in terms of their vulnerability to oxygen-induced oxidation.

The results are presented in the table 8 show that the values for BOD of the grease samples varied both with the process conditions and the nature of the thickener used. The values of BOD are between 14.23ppm and 20.25ppm for grease produced using lye as thickener, 17.64ppm, and

23.68ppm for grease produced using NaOH as a thickener. A group

researcher reported a BOD of 21.00ppm which is high above the optimal results of 14.23ppm and 17.64ppm gotten for lye and NaOH thickened grease using cottonseed oil as a base lubricant (Awoyale, et al., 2011). This shows that the grease produced in this work using cottonseed oil as a base lubricant, lye, and NaOH as thickeners are less susceptible to oxidative damage than that reported by (Awoyale, et al., 2011). These are, however, still far below the performance of control Oando and Texaco grease with very low BOD of 7.5ppm and 6.0ppm each. These differences could be a

result of the absence of other additives in the grease samples produced in this work, different from the commercial control grease samples (Awoyale, et al., 2011).

Table 8: Dropping point and BOD test results of the produced grease samples.			
S/N	**Sample**	**Grease from lye**	**Grease from NaOH**
1	**Dropping point (^{0}C)**	**169.27**	**166.55**
2	**BOD (ppm)**	**14.23**	**17.64**
3	**Status of water resistance**	**Excellent water resistance (resisted wash out)**	**Excellent water resistance (resisted wash out)**

3.8 Thermometric Results of the Inhibition Efficiencies of the grease samples

To determine the corrosion inhibition effect of the grease samples, the method used by was adopted with slight modification (Onukwuli and Omotioma., 2016). The measurements were carried out in an acid environment using a water bath with a thermostat set at 30^{0}C. Effect of inhibitor concentration on the reaction numbers and inhibition efficiencies of the grease produced with lye and NaOH as thickeners are presented in table 9 and 10 respectively. A plot of inhibition efficiency (%) against inhibitor concentration (g/L) is presented in figure 13. From the figure, it can be seen that the inhibition provided by the grease samples increased with increase in the concentration of the *Moringa olefeira* leaf extract used for the formulation of the grease. This can imply that the green inhibitor, *Moringa olefeira* leaf extract, has a corrosion inhibition role to play in the use of grease formulated from cottonseed oil. The highest inhibition efficiency was recorded to be 93.61% at 0.7g/L when lye from palm bunch was used as the thickener. For the inhibition efficiency when NaOH was used as the thickener, as high as 84.65% at 0.7g/L was recorded, this however was not comparable to the efficiency offered by the lye thickened lubricating grease. It can thus be said that the lye-thickened grease performed better than the NaOH-thickened grease and hence preferable for corrosion inhibition.

Table 9: Effect of inhibitor concentration on the reaction numbers and inhibition efficiencies of the grease produced with lye as a thickener.		
Inhibitor conc. (g/L)	**RN**	**IE (%)**
0.0	0.3259	
0.1	0.1321	59.48
0.3	0.0912	72.03
0.5	0.0483	85.17
0.7	0.0208	93.61
0.9	0.0371	88.61

Table 10: Effect of inhibitor concentration on the reaction numbers and inhibition efficiencies of the grease produced with NaOH as a thickener.		
Inhibitor conc. (g/L)	**RN**	**IE (%)**
0.0	0.3259	
0.1	0.1622	50.23
0.3	0.1235	62.09
0.5	0.0791	75.74
0.7	0.0500	84.65
0.9	0.0583	82.11

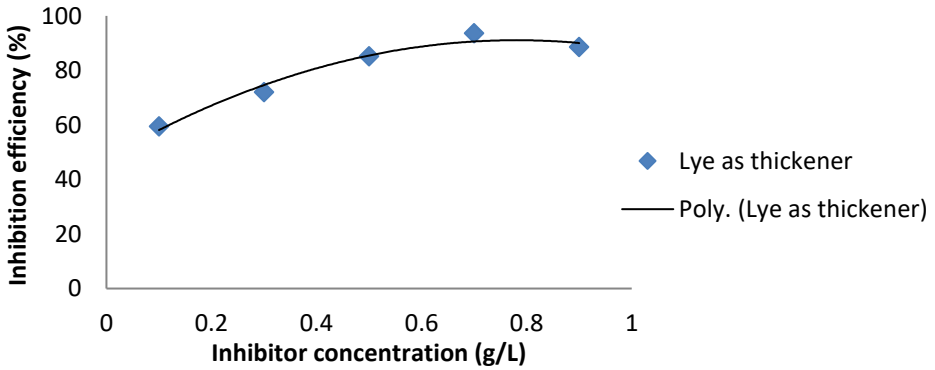

Figure 13: Effect of inhibitor concentration on the inhibition efficiencies of the grease samples

4. Conclusion

This work assessed the use of cottonseed oil as the base oil for grease formulation, lye from palm bunch as a thickener, and Moringa oleifera leaf extract as a rust-inhibiting ingredient. The use of NaOH as a thickener also yielded similar results. The dropping point, BOD levels, and water-washing resistance of both samples were used to evaluate their performance. Based on the study of the oil sample used for the formulation, cottonseed oil was determined to be an appropriate base oil for grease formulation. For cottonseed oil, the viscosity values were determined to be 31.87 cSt at 40°C and 6.97 cSt at 100°C. Thus, according to ASTM D2270, the viscosity index (VI) of cottonseed oil is 211, whereas the VI of a mineral oil with the same viscosity is 95. As a result, the vegetable oil's viscosity remains more stable across temperature variations, proving its suitability. The alkali from an empty palm bunch was found to make an excellent grease thickener and even outperformed the NaOH thickener. By utilising the large amounts of bio-waste that can be transformed into bio-alkali, companies will be able to reduce the cost of manufacturing compared to using NaOH and other synthetic thickeners.

ANOVA and the quadratic model provided a clear explanation of how the process variables interacted. The model was also examined and validated through three-dimensional plots of glycerin yield, showing a parabolic curve. The functional groups of glycerin from cottonseed oil include polar atoms and single and double bond structures, which show that the glycerin is suitable for lubricating grease. In comparison to the sodium-based control, the bio-alkali generated was excellent. When using grease made from cottonseed oil, the green inhibitor, Moringa oleifera leaf extract,

plays a role in preventing corrosion; the highest inhibition efficiency was recorded as 93.61% at 0.7 g/L when lye from palm bunch was used as the thickener. For the inhibition efficiency when NaOH was used as the thickener, a value as high as 84.65% at 0.7 g/L was recorded. Cottonseed oil is a viable alternative to mineral or petroleum oil as a base oil and is less expensive than synthetic oil. The biodegradable lubricating grease produced from cottonseed oil is suitable for various industrial applications. This study has expanded the range of feedstocks for bio-based lubricants for various industrial applications in both terrestrial and aquatic environments.

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Credit Authorship Contribution Statement.

- **Ifediorah Ezekiel Iyke**: Conceptualization, Methodology, Software, Data curation, Writing – original draft preparation, Reviewing, Visualization, Investigation, Software, and Validation.
- **A. K. Babayemi**: Supervision, Reviewing, and Editing.
- **O. L. Eluno**: Draft preparation, Writing, and Reviewing.
- **E. E. Eluno**: Computational modelling, Data analysis, Editing, and Reviewing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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