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Research Paper

Enhancing the Synthesis of Biolubricant from Used Chicken Fat: Optimization of Operating Parameters Using Magnesium Oxide Nanoparticles as a Catalyst and Response Surface Methodology

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Abstract

In light of the growing concerns regarding the environmental impact and sustainability of mineral oil-based lubricants, the use of biolubricants has been advocated as a renewable alternative. The double transesterification of used chicken fat oil involves two steps of converting the triglycerides into methyl esters (UCFME) using methanol and Magnesium oxide Nano Particles (MgO NPs) as catalyst, and utilizing Trimethylolpropane (TMP) and MgO NPs to produce the final biolubricant (UCFBL). This research aimed to optimize the reaction parameters for the transesterification process involving used chicken fat methyl ester (UCFME) and TMP using response surface methodology. A series of 20 individual experiments were conducted, focusing on the variables of reaction temperature, time, and UCFME-to-TMP molar ratio. Through statistical modeling, it was predicted that the transesterification process would yield a maximum conversion rate of 97.5% under the optimized conditions of a reaction temperature 114 °C, a reaction time 227 minutes, and a UCFME-to-TMP molar ratio 10.5:1. Experimental results, obtained from three independent replicates conducted under these optimal conditions, demonstrated an average yield of 98.3 % for the production of UCFBL, which aligned closely with the model's predicted range of 98.35%. The resultant biolubricant has remarkable lubrication qualities, such as a pour point of -5 °C, flash point of 289°C, viscosity index of 213, and kinematic viscosities (KV) of 38.5 and 9.2 cSt at 40 and 100 °C, respectively. These qualities revealed that the biolubricant generated fulfilled the ISO VG-32 criteria, making it an acceptable replacement for petroleum-based lubricants in industrial machine applications.

1. Introduction

In response to the evolving environmental regulations, the scientific community is developing lubricants that exhibit improved biodegradability and reduced toxicity. As part of this effort, traditional mineral oils derived from petroleum are being substituted with lubricants produced from bio-based materials, commonly known as biolubricants. (Onuh et al., 2017). Biolubricants are generated from renewable sources, with characteristics such as biodegradability, increased lubricity, suitable viscosity, oxidative stability, low toxicity, and thermal stability. Thus, biolubricants are environmentally friendly because of their capacity to naturally degrade, decrease friction and energy consumption, offer adequate lubrication under varying circumstances, resist oxidation, maintain worker safety, and operate well at high temperatures (Sanchez et al., 2013). As a result, these biolubricants can be applied across a diverse range of industrial uses,

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such as serving as emulsifiers, lubricants, plasticizers, surfactants, plastics, solvents, and resins (Cecilia et al., 2020). Since vegetable oils are typically utilized in the food chain, it is crucial to choose the beginning oil for creating a biolubricant. The usage of these oils for industrial purposes would lead to more speculation, which would raise prices and exacerbate socioeconomic inequalities (Sharma, & Sachan, 2019). Based on these findings, using vegetable oils, which don't affect the food chain, is the most sustainable solution.

Magnesium oxide nanoparticles (MgO NPs) are emerged as a catalyst for their remarkable properties. Their simple synthesizing method, large surface area and adjustable properties make MgO NPs useful (Ahmad & Biswas, 2023). MgO NPs have excellent catalytic properties in oxidation, reduction, acid reaction, organic synthesis, environmental remediation, energy storage, and heterogeneous catalysis applications (Amina et al., 2020; Rotti et al., 2023). These nanoparticles have a high catalytic activity due to factors such as large surface areas, large active sites, basic properties that facilitate acid-based reactions, oxidation and reduction reactions, stability that ensures durability and reusability (Vanitha et al., 2022). Due to the presence of bioactive compounds, leaves of *Azadirachta Indica* (neem) have been studied for the production of MgO NPs. Neem leaves contain limonoids, flavonoids and terpenoids as reducing and stabilizing agents (Sanjeev, 2023). Green synthesis methods use neem leaf extracts to convert magnesium precursors into MgO NPs. Bioactive compounds in Neem leaves effectively reduce precursors and stabilize nanoparticles to prevent agglomeration (Noorjahan et al., 2015). Green synthesis offers sustainable and natural alternatives to nanoparticle production due to its environmental efficiency, cost-effectiveness and lack of toxic chemicals. The chemistry of the leaves of *Azadirachta Indica*, together with their bioactive compounds, is an efficient and sustainable catalyst for reducing and stabilizing the green synthesis of MgO NPs for various applications (Shafiee et al., 2018).

The majority of the oils and fats used as feedstock come from plants and animals (Keneni & Marchetti, 2017). For use in the transesterification process, these feedstock sources are mostly biodegradable and non-toxic compounds. A first and fundamental need in the development of biolubricants is choosing quality raw materials (Kumar et al., 2017). Oil from plants is primarily divided into edible and non-edible (Amdebrhan et al., 2015). The production and cultivation of feedstock are largely influenced by the local geography. Since the manufacture of biolubricants was mostly obtained from edible oils like safflower, palm oil, sunflower oil, and coconut oil, etc. (Bashiri et al., 2021; Alang et al., 2018). It is extensively researched whether animal and plant waste products, organic waste materials, starch, cellulosic, and lignocellulosic may be used to produce biogas and bioethanol (Agrawal et al., 2017; Khan et al., 2022). However, it presented major problems with food security and rising costs from increased consumption, which reduced its widespread use. A viable and affordable feedstock for the manufacturing of biolubricants can potentially include animal fats and used cooking oil in addition to plants (Silviana et al., 2017). As high fatty acid concentration results in soap generation, these waste animal fat oils with free fatty acids are finally decreased by applying pre-treatment procedures and the transesterification method (Shehu et al., 2019).

Aside from MgO, several other nanoparticles have been investigated for their potential roles in biolubricant synthesis but have met constraints. Titanium dioxide (TiO_2) nanoparticles have photocatalytic activity, inducing lubricant deterioration when exposed to UV light, but they agglomerate, decreasing their dispersion and efficacy (Sankar et al., 2015). While silver (Ag) nanoparticles have antibacterial characteristics, their expensive cost and probable toxicity prevent them from being widely used. Because of their low friction coefficients, carbon-based nanoparticles such as carbon nanotubes (CNTs) and graphene have excellent lubricating characteristics; nevertheless, dispersion issues and environmental concerns limit their widespread application (Amina et al., 2020; Waqas et al., 2021). Overcoming photocatalytic activity, agglomeration, high cost, toxicity, dispersion, and environmental impact concerns are critical for the effective integration of previous nanoparticles into biolubricant manufacturing.

In general, liquid lubricants are the most widely used kind of lubricant; they are made up of 70–99 % base

stock and 1-30 % performance-enhancing additives (Reeves et al., 2017). However, the base stock, which may be prepared from waste animal fat oils, determines the lubricant's final performance (Matiliunaite & Paulauskiene, 2019).

2. Materials and Methods

2.1. Materials used

The major laboratory grade chemicals utilized for the biolubricant production were waste chicken fat, Analytical grade (AG) methanol (99.5%), magnesium nitrate from HiMedia, Sodium hydroxide from RCI labscan, trimethylolpropane (TMP), potassium hydroxide from RCI labscan, sulphuric acid from Pharma RCI labscan, n-hexane from pure chemicals co., phenolphthalein indicator from Qualigens, Nice, and Merck, sodium thiosulphate from RCI labscan, and starch indicator.

2.2. Sample preparation

The source material for this study was chicken fat obtained from a poultry processing plant in Deber Zeyit, Ethiopia. The fat was first washed and cleaned with deionized water to remove blood traces and other impurities. The purified fat was ground using a mill crusher, and the sample was then ready for the next stage (oil extraction).

2.3. Soxhlet extraction of oil from UCF

The tallow was extracted from used chicken fat using a Soxhlet extractor with hexane, as described by Gore (2018). Hexane and the used chicken fat (UCF) were charged into the flask at a ratio of 5:1 (v/w). Specifically, during the extraction, 100 ml of hexane were used for every 20 g of UCF. The extraction procedure was performed at a constant temperature of 70 °C for a duration of 3 h, with a stirring speed of 250 rpm. Following the extraction, the hexane solvent was recovered from the oil through a vacuum rotary evaporator at 70 °C. The recovered solvent (hexane) was subsequently condensed using a condenser for further reprocessing (Azad et al., 2016).

Following the degumming procedure, the oil was transferred to a beaker and 0.05 N NaOH was added at 70 °C. Following that, the mixture was agitated at a speed of 200 rpm and kept at a temperature of 70 °C for 1 h (Iloh et al., 2015). To remove any remaining sodium hydroxide and to avoid soap development, the mixture was thoroughly washed with distilled water. According to Bankovic-Ilic et al. (2012), the mixture was then exposed to a hot air drier working at 105 °C for 6 h to eliminate any leftover moisture. The oil yield of UCF was calculated using Equation 1.

$$\text{Oil yield (\%)} = \frac{\text{mass of oil extracted}}{\text{total mass of beef fat sample}} x100 \ldots (1)$$

2.4. Characterization of extracted oil

The physiochemical properties of UCF oil were assessed through the analysis of various process parameters, including Acid value, Free Fatty Acid (FFA) content, Saponification value, Kinematic viscosity, Specific gravity, and Iodine value. These parameters were determined as part of the characterization process for the UCF oil (Abdullah et al., 2016).

2.5. Green synthesis of MgO NPs by using *Azadirachta indica* leaves for biolubricant production

Azadirachta indica leaf extract, derived from the neem tree, was employed as a crucial component in the sustainable synthesis process to generate MgO NPs. The extract was critical in the reduction of magnesium ions (Mg^{2+}) and their conversion into MgO NPs. The reduction process was aided by exploiting the features of the extract, such as its hydroxyl and carbonyl groups, enabling for the transformation of the magnesium ions into the required nanoparticles of magnesium oxide. This environmentally friendly technique of manufacturing MgO NPs not only utilized the natural qualities of *Azadirachta indica* leaf extract but also created an eco-friendly way of extracting them (Waqas et al., 2021). The *Azadirachta indica* extract, which is high in hydroxyl and carbonyl groups, performed an important dual role in the creation of MgO NPs as a stabilizing and reducing agent. Its presence maintained the stability of the reaction mixture and enabled magnesium ion reduction, resulting in the successful production of the nanoparticles. A solution of 10 mL 0.1 M magnesium nitrate ($Mg(NO_3)_2$) was combined with 40 mL of *Azadirachta indica* extract solution, followed by continuous stirring for 60 min. The reaction rate was

regulated by adding 6 mL of 0.2 M sodium hydroxide (NaOH) drop by drop, resulting in the creation of MgO NPs and the development of a visible precipitate as proof of effective reduction and synthesis (Zahid et al., 2023). Subsequently, the solution was left undisturbed at a temperature of 25 °C. The formed precipitates were rinsed using distilled water, and the resulting wet precipitate was then subjected to a drying process. Following this, the dried precipitate underwent calcination at a temperature of 600 °C for a duration of 4 h, resulting in the production of a refined and pure powder.

2.6. Biolubricant synthesis by double transesterification of UCFO

The biolubricant derived from UCF oil was synthesized using a double transesterification technique. In the first step of the double transesterification process, the UCF oil underwent transesterification with methanol and MgO NPs as catalyst. This reaction converts the triglycerides present in the oil into methyl esters, resulting in the production of used chicken fat methyl esters (UCFME).

The UCFME obtained from the first transesterification was further reacted with TMP in the presence of MgO NPs as catalyst. This second transesterification reaction lead to the formation of the final biolubricant known as used chicken fat biolubricant (UCFBL) (Nwokocha & Aremu, 2017). To carry out the two procedures, the following actions were taken:

2.6.1 Synthesis of methyl ester using MgO NPs

A heating pan was used to transfer 100 mL of purified Used Chicken Fat Oil (UCFO), which was then heated to 500^0C. 25 mL of methanol was added to a separate beaker, followed by 0.920 g of MgO NPs. The mixture was agitated with a magnetic stirrer until the MgO NPs were completely dissolved. The warmed UCFO was then transferred to a 500 mL round bottom distillation flask, which served as the reaction vessel, and the catalyst (1% mass UCFO) was added. The contents of the reaction vessel were vigorously swirled for a duration of 15 min. Subsequently, the reaction mixture was heated under reflux in a water bath at 70^0C for 2 h, allowing the transesterification reaction to occur. After 20 min, the contents of the reaction vessel were transferred into a separatory funnel, and a visible separation of the supernatant liquid, containing methyl esters, was observed. Overnight, the reaction mixture were separated into two distinct layers: an upper layer consisting of amber-colored methyl esters and a lower layer containing impure glycerol. Each fraction was collected separately by allowing them to flow into labeled measuring cylinders. To eliminate water-soluble contaminants and residual catalysts, a series of washing processes were performed. The methyl ester was gently shook or mixed with a small amount of distilled water and then the layers were allowed to separate, to gently remove the aqueous layer. This cleaning technique was repeated multiple times until the water layer became clear (Alang et al., 2018). The yield of methyl ester was determined using Equation 2.

$$\text{Methyl ester yield } (\%) = \frac{\text{product volume}}{\text{sample (UCFO)volume}} x100 \ \ldots (2)$$

2.6.2 TMP-triester (biolubricant) synthesis using MgO NPs

In a 500-mL reaction vessel, 100 mL of UCFME synthesized in the above procedure was heated using a water bath to a temperature of 70°C. Subsequently, a catalyst solution containing 0.9 g of MgO NPs was added to the reaction vessel. After a 10 min interval, 20 g of TMP crystals were introduced to the reaction vessel, and the reaction was allowed to proceed for 4 h under reflux at a temperature of 100°C. Once the reaction time elapsed, the mixture was allowed to cool down to room temperature. The resulting mixture was then transferred into a separatory funnel, where the bottom viscous layer, consisting of the TMP triester (biolubricant), was collected (Chen et al., 2019). The overall reaction mechanism was illustrated in Figure 1 and the biolubricant yield was determined using equation 3.

$$\text{Biolubricant yield}(\%) = \frac{\text{Volume of product}}{\text{Volume of sample UCFME}} x100\%..(3)$$

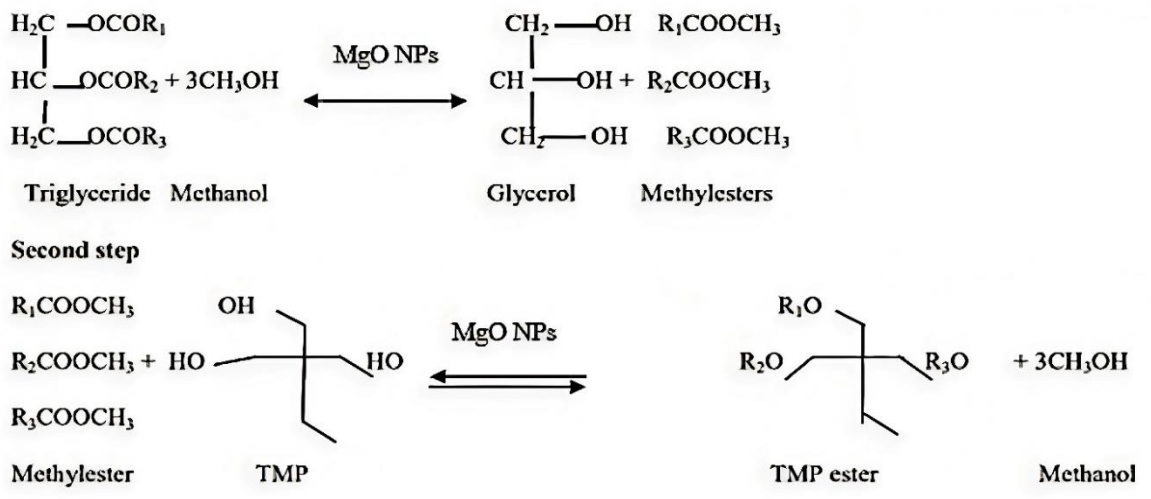

Figure 1: Double transesterification of used chicken fat oils to obtain biolubricants

2.7. Characterization of UCFO based biolubricant

To evaluate the biolubricant, standard analytical methods were employed to determine its physical and chemical properties. The chemical composition of the biolubricant was investigated using Fourier-transform infrared spectroscopy (FT-IR) (Samidin et al., 2021).

2.8. Experimental design for the synthesis of biolubricant

In the synthesis of used chicken fat oil biolubricant, Stat-Ease Design-Expert-13 software was carried out using Response Surface Methodology (RSM) with a central composite design (CCD). This method enabled the study of the connection between the response variable and a specified set of experimental conditions (Shehu et al., 2019). As shown in Table 1, the study took three experimental conditions into account: reaction temperature, reaction duration, and the molar ratio of UCFME to TMP. Using the formula (2k + 2k + N), the combination of these three elements determined the number of experimental runs, resulting in a total of 20 experiments. Following that, the generated UCFO biolubricant was characterized using American Society for Testing and Materials (ASTM) standard techniques. The overall process mechanism for double transesterification of used chicken fat oils to obtain biolubricants was illustrated using Figure 2.

Table 1: Independent variables and levels used for CCD for biolubricant

S/n	Independent variables	unit	Levels		
1	Reaction temperature	^{0}C	110	120	130
2	Reaction time	min	150	190	230
3	UCFME-to-TMP molar	ml/g	8:1	11:1	14:1

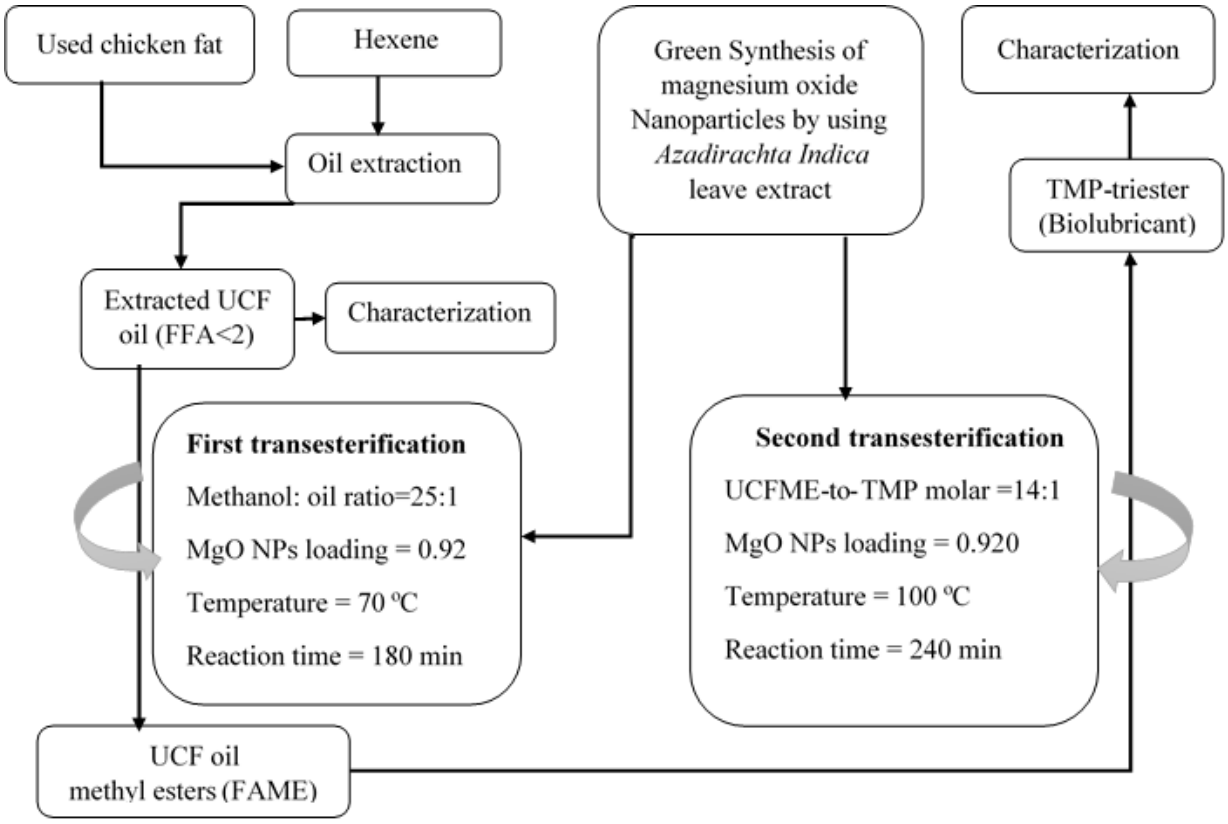

Figure 2: Simplified block flow diagram for the synthesis of UCF based biolubricant

3. Results and Discussion

3.1. Physicochemical properties of UCFO

From the biolubricant's standard physicochemical characteristics, the acid value of 3.9 mgKOH/g shows that the oil has a low quantity of free fatty acids (Table 2). The percentage of free fatty acids is 1.95 %, indicating a modest concentration of free fatty acids. The density at 15 °C is 879 kg/m^3, reflecting the mass per unit volume of the sample at that temperature. The saponification value of 185.5 mgKOH/g oil offers information on the fatty acids' average molecular weight. The iodine value of 20.3 gI_2/100g indicates that the oil is highly unsaturated. The specific gravity is 0.87, which represents the relative density of water. At 40°C, the viscosity is 46.25 mm^2/s, reflecting the oil's reluctance to flow. The oil yield is 67.8%, reflecting how much oil was recovered from the UCFO.

Table 2: Physiochemical properties of used chicken fat oil

Physiochemical properties	**Unit**	**Value**
Acid value	mgKOH/g	3.9
Free fatty acid content	%	1.95
Density@15 ^{0}C	kg/m^3	879
Saponification value	mgKOH/g oil	185.5
iodine value	gI2/100g	20.3
Specific gravity	-	0.87
Viscosity (40 ^{0}C)	mm^2/s	46.25
Oil yield	%	67.8

Figure 3 and Table 3 show that a series of 20 tests were carried out. These studies were carried out while three variables were varied: reaction temperature (110 to 130°C), reaction duration (150 to 230 minutes), and the UCFME to TMP molar ratio (between 8 and 14 ml/g). The primary goal of these studies was to create a biolubricant, and the biolubricant produced by each experiment was measured and documented with its expected value. This method allowed for the evaluation and study of the impacts of various component combinations on the end product, making it easier to identify ideal conditions for biolubricant manufacturing.

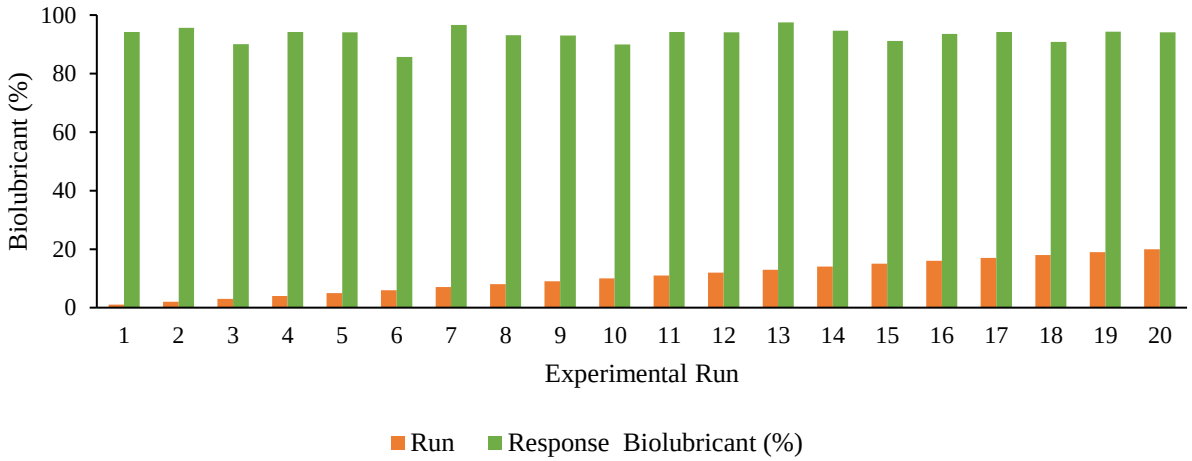

Figure 3: Number of runs vs biolubricant yield in percentage

Table 3: Experimental result fot the synthesis of biolubricant yield

Std	Run	Factor 1	Factor 2	Factor 3	Response	
		A: Reaction Temperature (^{0}C)	B: Reaction Time (min)	C: UCFME-to-TMP molar ratio (ml/g)	Biolubricant (%)	Predicted Value (%)
19	1	120	190	11	94.16	94.16
8	2	130	230	14	95.60	95.65
2	3	130	150	8	90.10	90.31
20	4	120	190	11	94.18	94.16
17	5	120	190	11	94.15	94.16
13	6	120	190	5.95	85.70	85.55
12	7	120	257.27	11	96.60	96.64
10	8	136.82	190	11	93.15	93.03
14	9	120	190	16.05	93.00	93.19
4	10	130	230	8	89.90	89.93
15	11	120	190	11	94.17	94.16
16	12	120	190	11	94.15	94.16
7	13	110	230	14	97.50	97.27
9	14	103.18	190	11	94.60	94.75
3	15	110	230	8	91.20	91.28
6	16	130	150	14	93.50	93.40
18	17	120	190	11	94.17	94.16
1	18	110	150	8	90.80	90.73
11	19	120	122.73	11	94.30	94.29
5	20	110	150	14	94.15	94.10

3.2. Model fitting and ANOVA analysis

To achieve the maximum biolubricant content, a three-level, three-factorial central composite design (CCD) was chosen to optimize the transesterification process variables. Analysis of variance (ANOVA) was employed as a multivariate technique to identify the optimal reaction conditions. All the 20 experimental runs as per the design were conducted, and the results were analyzed using multiple regression analysis. Based on the experimental data, a quadratic polynomial equation was derived to predict the biolubricant content. The equation, expressed in terms of coded variables, is presented as equation (4).

According to the model equation, the molar ratio coefficient exhibits the highest positive value, indicating its significant influence on the conversion process. A positive coefficient for a model term suggests that an increase in the corresponding variable leads to an increase in the yield.

3.3. ANOVA for used chicken fat oil based TMP ester yield

Tables 4 presents the results of the model summary test and lack of fit test for the yield of used chicken fat-based TMP ester. The selection of the highest order polynomial was based on the significance of additional terms and the absence of aliasing in the model. The P-values summary indicated that a quadratic model provided a good fit in the ANOVA, and therefore, it was chosen as the suggested model. The quadratic model was preferred over the cubic model due to limitations in the number of runs available in the Central Composite Design (CCD), which were insufficient to support a full cubic model. A significance level of 95% was employed, and thus, all terms with P-values less than 0.05 were considered statistically significant.

P-values less than 0.0500 indicate model terms are significant. In this case A, B, C, AB, BC, A^2, B^2, C^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

At the Std. Dev. of 0.1485, mean of 93.25 and C.V (%) of 0.1592, there is reasonably close agreement between the Predicted R^2 (0.9867) and the Adjusted R^2 (0.9968), with a difference of less than 0.2, indicating a good fit of the model. The Adequate Precision, which measures the signal-to-noise ratio, is a desirable value when it exceeds 4. In this case, the ratio of 111.668 indicates a strong signal. These results suggest that the model is reliable and can be utilized to explore and navigate the design space effectively.

Figure 4 displays the normal plot of residuals and the predicted vs actual plot. Upon observing the plots, it is evident that the points are closely distributed along the straight line, indicating a strong relationship between the experimental values and the predicted values of the response. Although some minor scatter, resembling an 'S' shape, is expected, these plots confirm the adequacy of the selected model in accurately predicting the response variable based on the experimental values.

$$\text{Biolubricant} = 94.16 - 0.5117.A + 0.6969.B + 2.27.C - 0.2312.AB - 0.0687.AC + 0.6562.BC - 0.0958.A^2 + 0.4611.B^2 - 1.70.C^2 \quad (4)$$

Table 4: Fit Summary of P-values for UCF based TMP ester

Source	Sequential p-value	Lack of Fit p-value	Adjusted R^2	Predicted R^2	Remark
Linear	0.0014	< 0.0001	0.5383	0.3194	Not suggested
2FI	0.7847	< 0.0001	0.4751	0.3078	Not suggested
Quadratic	**< 0.0001**	**< 0.0001**	**0.9968**	**0.9867**	**Suggested**
Cubic	< 0.0001	0.0076	0.9999	0.9954	**Aliased**

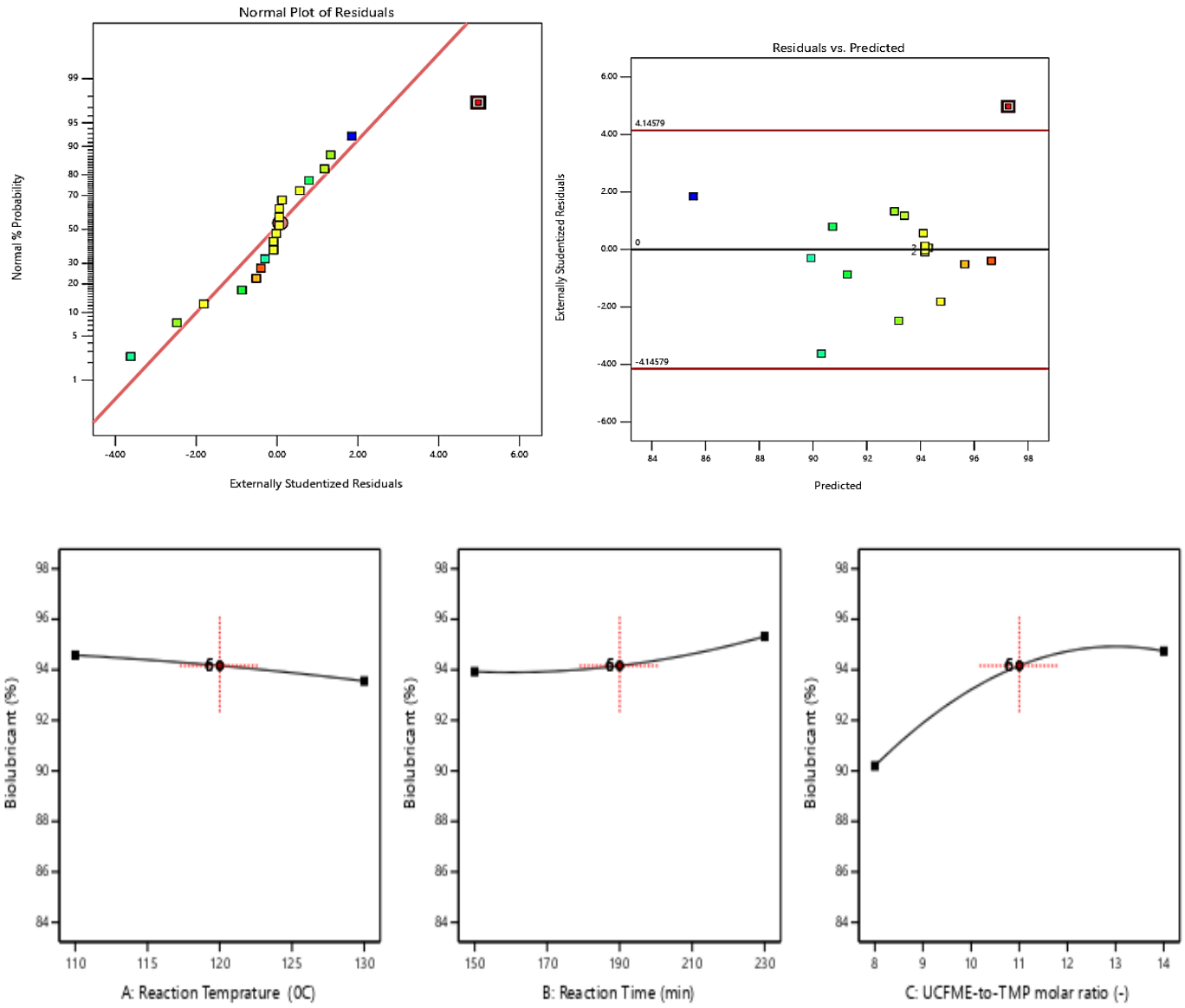

Figure 4: The impact of process parameters on UCFO-based biolubricant

3.4. Effect of reaction temperature and UCFME-to-TMP molar ratio

Figure 5 illustrates the combined effects of temperature and the UCFME-to-TMP molar ratio on the synthesis of UCF-based biolubricant, while keeping the reaction time constant. The temperature ranged from 110 to 130 °C, and the UCFME-to-TMP molar ratio varied from 8:1 to 14:1, with a constant reaction time of 190 min. It was observed that the conversion of UCFME-based biolubricant remained constant as the temperature increased, while it increased with an increase in the UCFME-to-TMP molar ratio. However, when a low temperature was combined with a high molar ratio, a yield of 85.5 wt% was obtained. A high temperature in combination with a low molar ratio, on the other hand, resulted in an improved yield of up to 94.17 wt%. This implies that the molar ratio of UCFME to TMP has a considerable influence on yield. Furthermore, the combination of a high temperature and a low molar ratio resulted in a larger yield than the reverse scenario, with an increase of roughly 11 wt%. As a result, it can be deduced that the UCFME-to-TMP molar ratio has a greater influence than temperature.

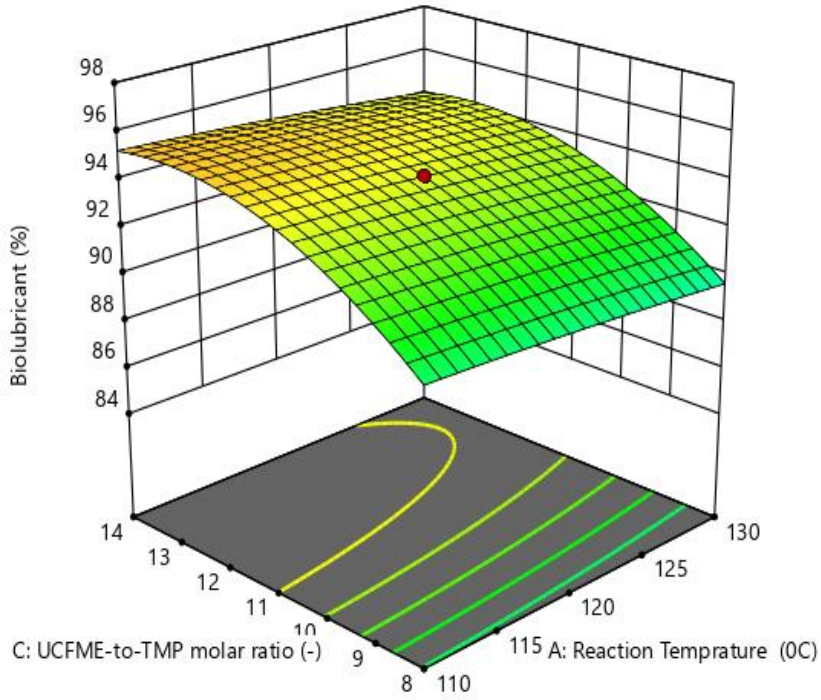

Figure 5: 3D surface response plot between UCFME-to-TMP molar ratio and temperature against UCF based biolubricant yield

3.5. Effect of reaction time and UCFME-to-TMP molar ratio

Figure 6 shows the influence of reaction time and the UCFME-to-TMP molar ratio on the production of a UCF-based biolubricant while keeping the temperature constant at 120°C. The molar ratio of UCFME to TMP was altered from 8:1 to 14:1, and the reaction time was varied from 150 to 230 min. The curve depicts the interaction between the molar ratio of UCFME to TMP and reaction time, which has a major influence on the synthesis of UCF-based biolubricant. The yield of UCF-based biolubricant increased with greater UCFME-to-TMP molar ratios and longer reaction durations. Increasing the UCFME-to-TMP molar ratio clearly leads in a higher production of UCF-based biolubricant. Furthermore, a greater concentration of UCFME results in a more effective conversion of UCF into biolubricant in a shorter period of time. The production of UCF-based biolubricant grew from 87.5 to 93.50 wt% in this investigation, with the greatest biolubricant seen after 230 min of reaction time.

3.6. Effect of reaction temperature and reaction time

Figure 7 displays the impact of reaction time and reaction temperature on the yield of UCF-based biolubricant, while maintaining a constant UCFME-to-TMP molar ratio of 11:1. The reaction time was varied from 150 to 230 min, while the process temperature ranged from 110 to 130 °C. The plot reveals that the quantity of UCF-based biolubricant yield increased when the reaction temperature remained constant. This indicates that higher reaction temperatures enhance the yield. On the other hand, an increase in reaction time improves the catalytic activity, particularly at lower temperatures.

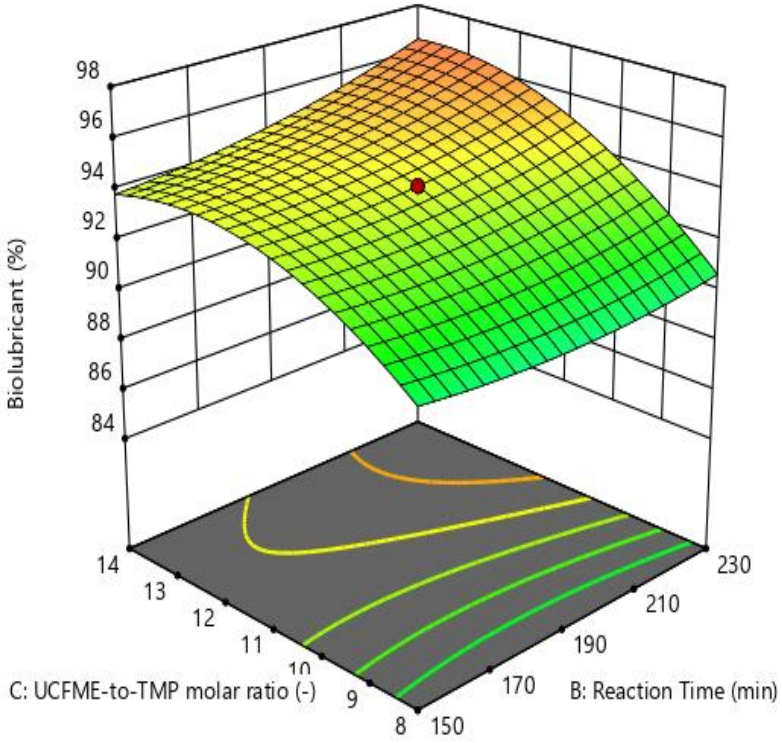

Figure 6: 3D surface response plot between UCFME-to-TMP molar ratio and reaction time compared to UCF based biolubricant yield

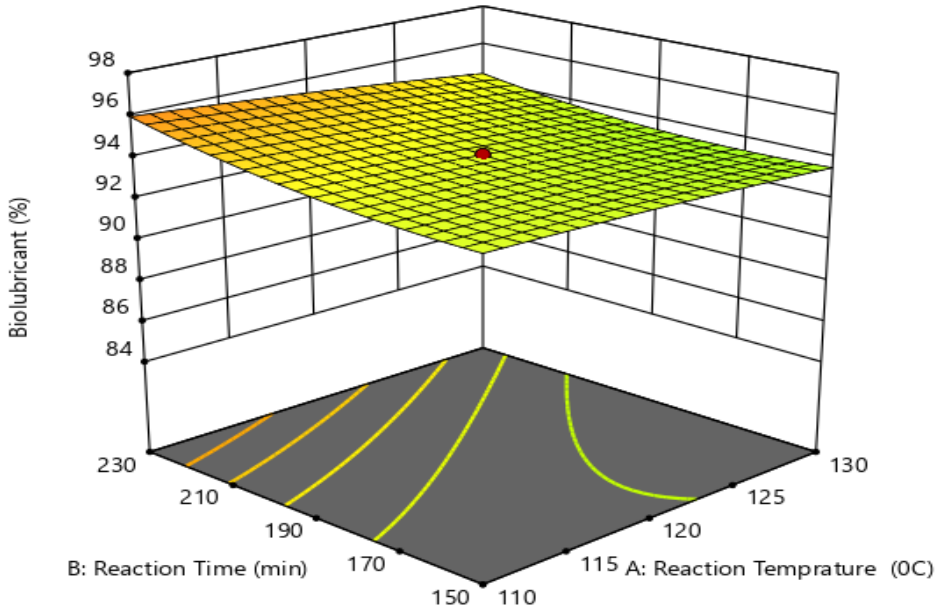

Figure 7: 3D surface response plot between reaction time and reaction temperature compared to UCF based biolubricant yield

Table 5 compares the characteristics of the UCFBL and the ISO-VG-32 standard for lubricants. UCFBL has advantages that meet or exceed ISO-VG-32 requirements. First, the pour point of -5 °C indicates that the UCFBL remains liquid even at low temperatures, exceeding the standard requirement of less than -10 °C. The flash point of 289 °C exceeds the minimum requirement of 204°C, indicating high ignition resistance of the UCFBL. The viscosity index of the UCFBL 213 is high, surpassing the standard minimum

of 90 at high temperatures. At 40 °C, the kinetic viscosity is 38.5 cSt, higher than the standard minimum of 28.80 cSt, and at 100°C, the kinetic viscosity is 9.2 cSt, higher than the standard minimum of 4.10cSt. These results show that the UCFBL has desirable flow and lubrication properties. Furthermore, the free fatty acid content of UCFBL is 1.95 %, which is lower than the standard level of 2 %, indicating a good quality with low free fatty acid concentration. Overall, UCFBL meets ISO-VG-32 standards and can be considered as an appropriate replacement for petroleum-based lubricants.

3.7. Process variable optimization

A numerical optimization technique utilizing the desirability function was employed to determine the optimal conditions for maximizing the production of UCF-based biolubricant. The goal was to establish ideal conditions for biolubricant synthesis by setting ranges for the reaction temperature, UCFME-to-TMP molar ratio, and reaction time, based on the specific requirements of the biolubricant (Ocholi et al., 2021). Using these criteria, the software performed an optimization process and identified the potential optimum conditions: a reaction temperature of 114 °C, a UCFME-to-TMP molar ratio of 10.5:1, and a reaction time of 227 min (Figure 8). Under these optimized conditions, an average yield of UCF-based biolubricant of 98.3 % was achieved in three independent replicates, which closely aligned with the predicted range (98.35 %) from the model. The desirability value for these conditions was 1.00, indicating a high level of desirability and suitability for producing the desired biolubricant.

3.8. FT-IR spectroscopy of TMP-triester (biolubricant)

During the second transesterification reaction, the FTIR spectra (FTIR-65, Perkin-Elmer) was utilized to track the progress of the reaction quantitatively. In these spectra, the ester carbonyl peak was the most prominent, extending to 1734 cm^{-1}. However, this peak cannot be utilized for reaction monitoring since the "C=O" stretch of the ester is equally strong in both the initial triglyceride and the fatty acid ethyl ester. To monitor the production of biolubricant through transesterification of oil, the intensity of the C-O-ester peak at 1177 cm^{-1} was employed. This peak served as an indicator of the formation of the desired biolubricant. Figure 9 depicts the results of the FTIR spectrum analysis of the biolubricant derived from used chicken fat.

Table 5: physiochemical properties of used chicken fat based biolubricant

Properties	**UCFBL**	**ISO-VG-32 standard**
Pour point (°C)	-5	< -10
Flash point (°C)	289	> 204
Viscosity index	213	> 90
Kinematic viscosity@ 40°C (cSt)	38.5	> 28.80
Kinematic viscosity@ 100°C (cSt)	9.2	> 4.10
Free fatty acid (%)	1.95	< 2.00

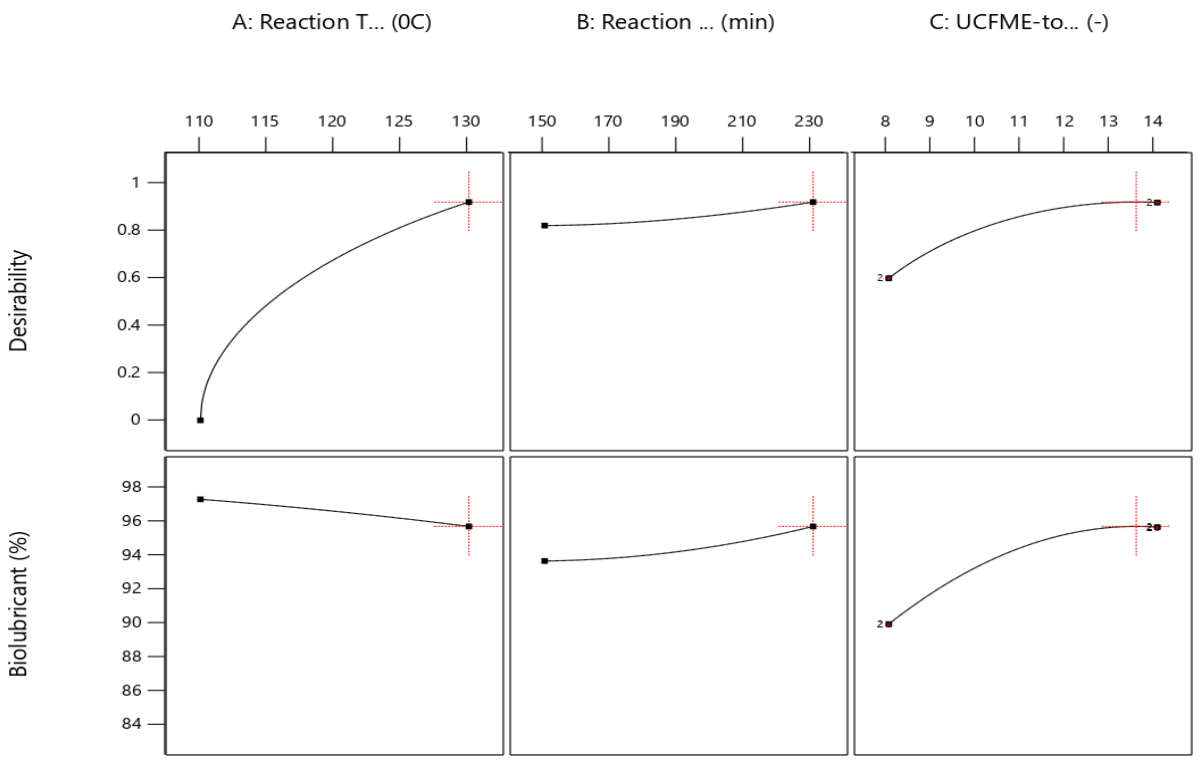

Figure 8: Desirability value for optimization of biolubricant

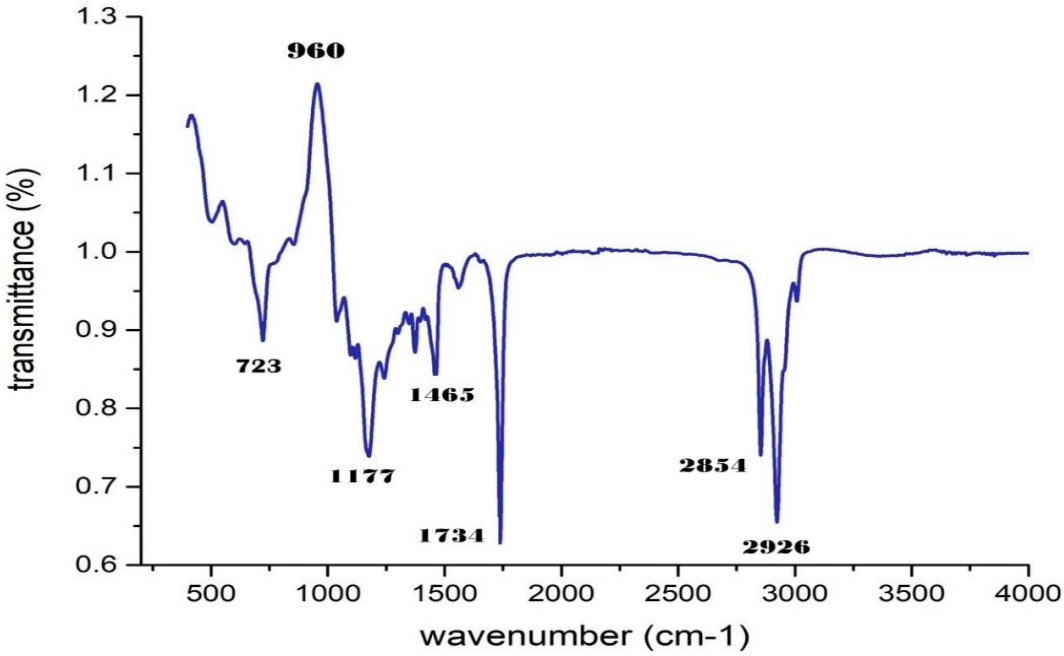

Figure 9: FT-IR spectroscopy of used chicken fat biolubricant

4. Conclusions and Recommendations

This research demonstrated that used chicken fat can be effectively utilized for the production of biodegradable lubricant through reactive extraction. The Central Composite Design (CCD) model in Response Surface Methodology (RSM) was successfully employed to optimize the reaction parameters for the synthesis of UCFBL. The study examined and optimized the effects of the UCFME-to-TMP molar ratio, reaction time, and reaction temperature on the synthesis of UCFBL. The resulting UCFBL exhibited significant lubricating properties, including a pour point

of -5°C, flash point of 289 °C, viscosity index of 213, and kinematic viscosities (KV) of 38.5 cSt at 40 °C and 9.2 cSt at 100 °C. It was observed that the produced biolubricant meets the ISO VG-32 standards, making it a suitable substitute for petroleum-based lubricants in industrial machine applications. Therefore, the synthesized biolubricant can effectively serve as an environmentally friendly alternative to traditional lubricants. As a byproduct, the poultry business creates significant volumes of chicken fat, which was historically considered trash. With an increasing emphasis on resource efficiency and circular economy concepts, excess chicken fat has found new applications in the production of biolubricants.

More studies may be undertaken on a bigger scale to investigate the scalability and cost-effectiveness of the UCFBL production process. This would include determining the viability of adopting the reactive extraction process in an industrial environment utilizing used chicken fat. Furthermore, the performance of UCFBL can be improved by exploring the impacts of additional process factors or additives that may improve its lubricating characteristics. Furthermore, testing the UCFBL for long-term stability and endurance under various operating circumstances would give vital insights into its performance and usefulness in real-world industrial applications. Finally, investigating the biodegradable lubricant's environmental impact throughout its lifespan, including disposal and deterioration, would add to a thorough knowledge of its sustainability and eco-friendliness.

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Abbreviations

ASTM	American Society for Testing & Materials
ANOVA	Analysis of Variance
AG	Analytical grade
CCD	Central Composite Design
FTIR	Fourier-transform infrared spectroscopy
FFA	Free Fatty Acid
h	hours
ISO-VG	International Standards Organization Viscosity Grade
KV	Kinematic Viscosities
MgO NPs	Magnesium oxide Nano Particles
min	minute
RSM	Response Surface Methodology
TMP	Trimethylolpropane
UCF	Used Chicken Fat
UCFBL	Used Chicken Fat Biolubricant
UCFME	Used Chicken Fat Methyl Ester
UCFO	Used Chicken Fat Oil

Reference

Abdullah, B. M., Zubairi, S. I., Huri, H. Z., Hairunisa, N., Yousif, E., & Basu, R. C. (2016). Polyesters based on linoleic acid for biolubricant basestocks: Low-temperature, tribological and rheological properties. *PLoS One*, 11(3), e0151603. https://doi.org/10.1371/journal.pone.0151603

Agrawal, A. J., Karadbhajne, V. Y., Agrawal, P. S., Arekar, P. S., & Chakole, N. P. (2017). Synthesis of biolubricants from non edible oils. Int. Res. J. Eng. Technol., 4(07), 1753-1757.

Ahmad, W., & Biswas, P. (2023). Study of the single step green synthesis of MgO Nanoparticles using peanut Shell Extract for the evaluation of in Vitro Antibacterial and Photocatalytic Properties. *J. Ultrafine Grained and Nanostructured Materials*, 56(1), 75-83. https://doi.org/10.22059/JUFGNSM.2023.01.08

lang, M. B., Ndikontar, M. K., Sani, Y. M., & Ndifon, P. T. (2018). Synthesis and characterisation of a biolubricant from cameroon palm kernel seed oil using a locally produced base catalyst from plantain peelings. *Green sustain. chem.*, 8(3), 275-287. https://doi.org/10.4236/gsc.2018.83018

Aloh, G. S., Obeagu, E. I., Emeka, O. C., Kingsley, O., Ezechukwu, O. C. & Uzoma, O. G. (2015). Extraction and Partial Characterization of Mustard Seed (Brassica Spp.) Oil. World Journal of Pharmaceutical Research, 4(3), 177-195

Amdebrhan, B. T., Damtew, L., Tesfay, D., Endris, H., & Tekeste, G. (2015). Production of biolubricant from castor (Ricinus) oil. Int. J. Eng. Innov. Res., 4(5), 737-741.

Amina, M., Al Musayeib, N. M., Alarfaj, N. A., El-Tohamy, M. F., Oraby, H. F., Al Hamoud, G. A., Bukhari, S. I. & Moubayed, N. M. (2020). Biogenic green synthesis of MgO nanoparticles using Saussurea costus biomasses for a comprehensive

detection of their antimicrobial, cytotoxicity against MCF-7 breast cancer cells and photocatalysis potentials. *PLoS One*, 15(8), e0237567. https://doi.org/10.1371/journal.pone.023756

Azad, A. K., Rasul, M. G., Khan, M. M. K., Sharma, S. C., Mofijur, M., & Bhuiya, M. M. K. (2016). Prospects, feedstocks and challenges of biodiesel production from beauty leaf oil and castor oil: A nonedible oil sources in Australia. *Renew. Sust. Energ. Rev.* , 61, 302-318. https://doi.org/10.1016/j.rser.2016.04.013

Bankovic-Ilic, B. I., Stamenkovic, O. S., & Veljkovic, V. B. (2012). Biodiesel production from non-edible plant oils. Renew. Sust. Energ. Rev., 16(6), 3621-3647. https://doi.org/10.1016/j.rser.2012.03.002

Bashiri, S., Ghobadian, B., Soufi, M. D., & Gorjian, S. (2021). Chemical modification of sunflower waste cooking oil for biolubricant production through epoxidation reaction. *Mater Sci Energy Technol*, 4, 119-127. https://doi.org/10.1016/j.mset.2021.03.001

Cecilia, J. A., Plata, D. B., Saboya, R. M. A., de Luna, F. M. T., Cavalcante Jr, C. L., & Rodríguez-Castellón, E. (2020). An overview of the biolubricant production process: Challenges and future perspectives. *Processes*, 8(3), 257. https://doi.org/10.3390/pr8030257

Chen, J., Bian, X., Rapp, G., Lang, J., Montoya, A., Trethowan, R., Bouyssiere, B, Portha, J-F., Jaubert, J-N., Pratt, P. & Coniglio, L. (2019). From ethyl biodiesel to biolubricants: Options for an Indian mustard integrated biorefinery toward a green and circular economy. *Ind Crops Prod.*, 137, 597-614. https://doi.org/10.1016/j.indcrop.2019.04.041

Gore, M. M. (2018). Extraction and physicochemical characterization of oil from Maringa Stenopetala Seeds. *IOSR j. appl. chem.*, 11(6), 1-7. https://doi.org/10.9790/5736-1106010107

Khan, M. U., Usuman, M., Ashraf, M. A., Dutta, N., Luo, G., & Zhang, S. (2022). A review of recent advancements in pretreatment techniques of lignocellulosic materials for biogas production: Opportunities and Limitations. Chem. Eng. J. Adv., 10, 100263. https://doi.org/10.1016/j.ceja.2022.100263

Kumar, V., Azamuddin, Syed, A. & Thyagraj, L. (2017). Use of Vegetable Oils as Bio-Lubricants : Review. *Int. Adv. Res. J. Sci. Eng. Technol.*, *4*(7), 2393–2395. https://doi.org/10.17148/IARJSET

Matiliunaite, M., & Paulauskiene, T. (2019). From concept to practice: manufacturing of bio-lubricants from renewable resources. *Biomass Conv. Bioref.* 353–361. https://doi.org/10.1007/s13399-018-0356-0

Mohammad Shafiee, M. R., Kargar, M., & Ghashang, M. (2018). Characterization and low-cost, green synthesis of Zn^{2+} doped MgO nanoparticles. Green Process Synth, 7(3), 248-254. https://doi.org/10.1515/gps-2016-0219

Noorjahan, C. M., Shahina, S. K. J., Deepika, T., & Rafiq, S. (2015). Green synthesis and characterization of zinc oxide nanoparticles from Neem (Azadirachta indicia). Int. J. Sci. Eng. Technol. Res, 4(30), 5751-5753.

Nwokocha, L. M., & Aremu, T. B. (2017). Studies on the biolubricant properties of moringa oleífera seed oil: correlating viscosity and fatty acid composition. *Malaysian J. Sci.*, 36(2), 116-131. https://doi.org/10.22452/mjs.vol36no2.6

Ocholi, O., Menkiti, M. C., & Aniagor, C. O. (2021). Optimization of biodegradable lubricant basestock synthesis from Jatropha curcas seed oil using response surface methodology. *J. Eng. Appl. Sci.* 19 (1). Available at: http://www.facultyofengineeringnau.org/archive/2928266135102273355.pdf

Onuh, C. Y., Dosunmu, A., Anawe, P. A. L., Efeovbokhan, V., & Adebisi, A. (2017). Transesterification of Non-Edible Vegetable Oil for Lubricant Applications in Water-Based Mud : A Review. International Journal of Applied Engineering Research, 12(18), 7397–7401.

Reeves, C. J., Siddaiah, A., & Menezes, P. L. (2017). A review on the science and technology of natural and synthetic biolubricants. J Bio Tribo Corros 3(11), (2017). https://doi.org/10.1007/s40735-016-0069-5

Rotti, R. B., Sunitha, D. V., Manjunath, R., Roy, A., Mayegowda, S. B., Gnanaprakash, A. P., Alghamdi, S., Almehmadi, M., Abdulaziz, O., Allahyani, M., Aljuaid, A., Alsaiari, A. A., Ashgar, S. S., Babalghith, A. O., Abd El-Lateef, A. E. & Khidir, E. B. (2023). Green synthesis of MgO nanoparticles and its antibacterial properties. Front. Chem. 11:1143614. https://doi.org/10.3389/fchem.2023.1143614

Samidin, S., Salih, N., & Salimon, J. (2021). Synthesis and characterization of trimethylolpropane based esters as green biolubricant basestock. *Biointerface Res. Appl. Chem*, 11(5), 13638-13651. https://doi.org/10.33263/BRIAC115.1363813651

Sanchez, M., Aracil, J., & Martínez, M. (2013). Current status and prospects of biodiesel production from Brassica Species. *Int. Rev. Chem. Eng.(I.RE.CH.E.)*, 5(5), 342-350. Retrieved from https://www.praiseworthyprize.org/jsm/index.php?

Sanjeev, N. O., Vallabha, M. S., & Valsan, A. E. (2023). Adsorptive removal of pharmaceutically active compounds from multicomponent system using Azadirachta indica induced zinc oxide nanoparticles: analysis of competitive and cooperative adsorption. *Water Sci Technol.* 87(1):284-303. https://doi.org/10.2166/wst.2022.428.

Sankar, R., Rizwana, K., Shivashangari, K. S., & Ravikumar, V. (2015). Ultra-rapid photocatalytic activity of Azadirachta indica engineered colloidal titanium dioxide nanoparticles. *Appl. Nanosci.*, 5, 731-736. https://doi.org/10.1007/s13204-014-0369-3

Sharma, U. C., & Sachan, S. (2019). Friction and wear behavior of karanja oil derived biolubricant base oil. SN Applied Sciences, 1, 668. https://doi.org/10.1007/s42452-019-0706-y

Shehu, M. S., Lamido, S. I., & Alhassan, A. U. (2019). Optimization of double transesterification for biolubricant synthesis from Jatropha oil. *Int. Adv. Res. J. Sci. Eng. Technol.*, 6(5), 23-30. https://doi.org/10.17148/IARJSET.2019.6505

Silviana, S., Anggoro, D. D., & Kumoro, A. C. (2017). Waste cooking oil utilisation as bio-plasticiser through epoxidation using inorganic acids as homogeneous catalysts. *Chem. Eng. Trans.*, 56, 1861-1866. https://doi.org/10.3303/CET1756311

Vanitha, G., Manikandan, R., Sathiyamoorthi, K., & Dhinakaran, B. (2022). Review on green synthesis of nanoparticles using various strong electrolytic metal solutions mediated by various plant parts. *J. Nanosci. Tech.*, 8(2), 960-966. https://doi.org/10.30799/jnst.334.22080201

Waqas, M., Zahid, R., Bhutta, M. U., Khan, Z. A., & Saeed, A. (2021). A review of friction performance of lubricants with nano additives. *Materials (Basel*), 14(21), 6310. https://doi.org/10.3390/ma14216310.

Yadessa Gonfa Keneni, & Marchetti, J. M. (2017). Oil extraction from plant seeds for biodiesel production. AIMS Energy, 5(2), 316–340. https://doi.org/10.3934/energy.2017.2.316

Zahid, A., Mukhtar, Z., Qamar, M. A., Shahid, S., Ali, S. K., Shariq, M., Alathlawi, H. J., Hasan, M. A., Khan, M. S., Islam, S., Patil, R. B., Al Ansari, M. S., Nawaz, N. & Sher, M. (2023). *Synthesis of Mn-Doped ZnO Nanoparticles and Their Application in the Transesterification of Castor Oil*. *Catalysts*, 13(1):1-13. https://10.3390/catal13010105