

Investigate the impact of blenders on Grease formulated from Waste Automobile Engine Lubricant as base oil

Hailemariam Nigus Hailu^{1*}, Neway Yilma Demuk²

¹ Department of Automotive Technology, Faculty of Mechanical Technology, FDRE Technical and Vocational Training Institute, Addis Ababa, Ethiopia.

² Department of Automotive Technology, Ethio-Italy Polytechnic College, Dire Dawa, Ethiopia.

*Corresponding author, E-mail: hailuqua@gmail.com

Abstract

In this study, the production process for lithium-based Grease using treated waste engine oil as the base oil was investigated. The waste engine oil was collected and purified by settling, filtration, thermal dewatering, acid treatment, neutralization, and post-treatment filtration to ensure suitability for grease production. The grease compositions were developed using Minitab's Extreme Vertex Mix Design software, varying the proportions of treated engine oil, lithium hydroxide (LiOH), Graphite, and calcium carbonate (CaCO₃). Fourteen formulations were prepared, and samples were tested according to ASTM standards for dropping point, oil separation, evaporation loss, and copper corrosion resistance. The best formulation was determined to be 81.66% treated base Oil, 11.67% LiOH, and 6.67% additives, which delivered superior thermal performance. The findings revealed that LiOH content was directly correlated with thermal stability, and that both LiOH and the additive contributed to reduced oil separation and evaporation loss. Mixing temperature also affected stability, with 160°C yielding better structural integrity than 200°C. All samples maintained low evaporation loss across temperature variations. The study demonstrates that recycled waste engine oil can serve as a reliable, cost-effective, and environmentally friendly raw material for grease production, contributing to circular economy goals and offering a viable solution for industrial-scale manufacturing.

Keywords: Lithium-based Grease, Mixture design, Oil separation, Evaporation loss, Sustainability.

1. Introduction

Recycling used engine oil to produce Grease promotes a sustainable environment. In Ethiopia, used engine oil constitutes the majority of waste. The depletion of natural resources, along with other environmental issues, has shifted the oil industry's focus toward resource opportunities. The industry is now focusing on reducing environmental harm caused by industrial waste [1].

Recycling leftover engine oil into Grease increases environmental sustainability. When waste oil is disposed of, it releases toxic chemicals and other contaminants into the soil [2]

[3]. As a result, these pollutants can severely contaminate groundwater [4]. Recycling used engine oil can significantly reduce the volume of waste oil that ends up in landfills. Furthermore, it helps reduce production costs by repurposing waste engine oil as a valuable resource[5-10].

Grease is a semisolid lubricant used in machinery to reduce friction, dissipate heat, prevent corrosion, and remove contaminants [11]. It consists of a lubricating oil thickened with a metal soap [12]. Typically, grease is made from petroleum oil thickened with metal soaps such as sodium, calcium, aluminum, and lithium [13].

Grease formulations consist of a base oil, which makes up 70% to 95% of the semisolid structure. The remaining portion includes thickeners, additives, and modifiers [14, 15]. Research indicates that the typical percentages of base oils, thickeners, and additives are approximately 70–75%, 25–30%, and 0–2%, respectively. Generally, the optimal weight percentages for different types of Grease that ensure good performance are above 8% for additives.

The choice and number of additives are crucial to a grease's ability to withstand high pressure and wear [16, 17]. Additives with a layered graphite structure reduce friction and wear rates, enhance load capacity and thermal stability, and provide dry-film lubrication under extreme-pressure and boundary conditions. Lithium grease with 2 wt% submicron graphite yields a 41% decrease in friction and a 15% smaller wear scar[18]. The inexpensive thickener CaCO_3 enhances grease stability and consistency while improving load distribution across surfaces. Researchers found that CaCO_3 nanoparticles substantially reduce friction and wear [19]. The combination of graphite and CaCO_3 offers a sustainable and cost-effective solution that delivers medium-duty performance and meets ASTM D4950 and NLGI guidelines.

Grease production relies on thickeners to create a semisolid structure that retains the base oil while controlling the grease's mechanical stability, load, and temperature performance [20]. Among various thickeners, lithium soap grease is produced by reacting lithium hydroxide (LiOH) with stearic acid or 12-hydroxystearic acid, yielding lithium stearate, the primary thickening agent. Standard industrial practice uses LiOH at 5-8 weight percent of the total grease composition, based on base oil viscosity, acid type, and desired NLGI consistency [21]. The Mixture Design experiment, using Minitab's Extreme Vertices Design, enabled researchers to establish an optimal LiOH concentration that met three critical conditions: complete saponification of the fatty acid, robust formation of a thickener network, and targeted NLGI Grade 2–3 consistency without excessive rigidity. The determined optimal LiOH level aligned with typical formulations found in the NLGI Lubricating Grease Guide and related studies on lithium greases derived from recycled or bio-based oils [22].

Using waste oils as the base for lubricating grease reduces water pollution by limiting contaminant levels. Water and mechanical impurities in waste oil samples varied with the conditions under which the samples were collected. Additionally, two types of non-soap

thickeners were used to produce Grease from waste transformer oil. The optimum formulation is a bentonite-based NLGI-grade 2 grease with a viscosity level in line with that of the great majority of commercially used Greases, and with bentonite and base oil contents of 20% to 80%. On the contrary, a lower base oil viscosity reduces storage and use stability in Grease[23, 24]

The Grease was prepared by mixing a sodium soap thickener with used engine oil for 2 hours at 150°C. To assess the prepared greases, tests were conducted for oil separation, oil bleeding, and consistency. Research indicates that waste oil can serve as a base material for synthesizing Grease, particularly when combined with red gypsum. The dropping point of 240°C indicates that this grease formulation can perform at high temperatures [25]. The grease's stability, oil bleeding, and consistency were evaluated. The Grease produced from synthetic and used oil tends to solidify and lose its lubricating properties at high temperatures, unlike motor oil grease [26]. Researchers have investigated the production of lubricating Grease using used engine sludge and cooking oil. This lubricating Grease is gentle and suitable for equipment that does not need to withstand high pressure[21, 27] .

A response trace plot analysis reveals each item's contribution to the outcomes, showing how each component influences the response. This is accomplished by drawing response traces along the component direction, representing a series of predictions from the fitted model [28, 29]. The trace curves illustrate how to adjust the right part along an imaginary line extending from the reference blend to the vertex [30]. Each component of the combination has a corresponding trace direction. The number of curves equals the number of components in the combination, and the positions along a component's trace direction are linked. When multiple components are present and a contour or surface plot cannot represent the entire response surface, response trace plots are useful. A response trace plot helps identify key elements, thereby facilitating the creation of a contour or surface plot [31].

2. Experimental work

2.1. Material

Used Toyota Quartz 5000 20W-50 engine oil was collected as the base oil from the MOENCO repair shop in Dire Dawa City. To produce a lithium soap thickener, stearic acid, a long-chain saturated fatty acid, was reacted with lithium hydroxide. Formic acid was used to process the used engine oil, and earth bleaching was applied to clean the oil. Odor and color treatments were also applied. Coconut oil fatty acids help prevent rust and serve as anti-wear additives. Graphite and calcium carbonate (CaCO_3) were selected as additives for their established roles in enhancing the tribological and rheological performance of lithium soap-based greases. The addition of CaCO_3 improves mechanical stability while maintaining proper consistency and promoting load distribution [32]. Likewise, graphite offers a sustainable and economical alternative to CaCO_3 and to commercial additives such as MoS_2 or ZDD for lubrication applications [33].

Using Minitab's Extreme Vertices Design, a mixture design experiment was conducted to optimize the LiOH concentration. Three primary goals were targeted for the formulation: complete saponification of stearic acid, adequate formation of the thickener network, and a grease consistency within the balanced NLGI Grade 2-3 range rather than excessive stiffness. The optimum LiOH concentration aligned well with values listed in the NLGI Lubricating Grease Guide and with concentrations used in lithium soap-based greases formulated with recycled or vegetable-based oils. The grease formulation was tested against the ASTM D4950-20 specifications for automotive service greases to guard against over-thickening or underperformance. Values for the core parameters of dropping point, oil separation, and copper corrosion were all above their respective limits stipulated by the standards. There was no evidence of over-thickening; the grease remained thermally stable, with excellent pumpability and spreadability throughout the expected service temperature range. Underperformance was also ruled out, given the grease's relatively high dropping point ($>190^{\circ}\text{C}$) and low oil separation ($<5\%$), both of which indicate strong structural integrity and lubrication-retention performance. Later, details about homogenization control were provided [14].

2.2. Pre-treatment of Used engine oil

The pre-treatment of waste engine oil involves several stages, including settling and filtration, thermal dewatering, acid treatment, alkaline neutralization, and final settling and filtration.

Settling and Filtration: The collected waste engine oil was allowed to settle for 24 hours, then filtered to remove contaminants such as dust, solid particles, and metal shavings. This low-energy process is a standard pre-treatment step in oil recovery, effectively reducing impurities before chemical treatment[34, 35].

Thermal dewatering: According to Mohammed et al. [34], a temperature range of 120°C to 150°C effectively removes moisture and low-molecular-weight volatile chemicals without thermally damaging the base oil. As shown in Fig.1(A), the dewatered waste engine oil was collected in 300 mL batches and then dried at 140°C for about 15 minutes to remove residual moisture.

Acid Treatment with formic acid: Formic acid was chosen for its low molecular weight and high polarity, which help break down polar impurities, including oxidized species, resins, and varnishes. Several studies, including Hamawand et al. (2013) [36], recommend maintaining the reaction at 60°C , as this temperature optimizes reactivity while minimizing acid degradation and impurity breakdown. The preheated waste engine oil is then mixed with formic acid and heated for an additional 20 minutes with stirring, as indicated in Fig.1(B). The temperature is kept at 60°C to allow the mixture to cool and promote sedimentation at the bottom of the beaker (see Fig.1(C)).

Alkaline Neutralization with Quick Lime: The excessively acidic oil was converted into an alkaline solution by reacting it with a 6% quick-lime (CaO) solution. The mixture was heated for 30 minutes at 120°C while stirring continuously.

Post-Treatment Settling and Filtration: The 24-hour settling period after neutralization allows for the complete sedimentation of metal soap and sludge particles, consistent with

conventional waste oil processing methods, such as the D893 standard [37]. After the 24-hour settling period, the oil was filtered and then decanted into a new beaker.

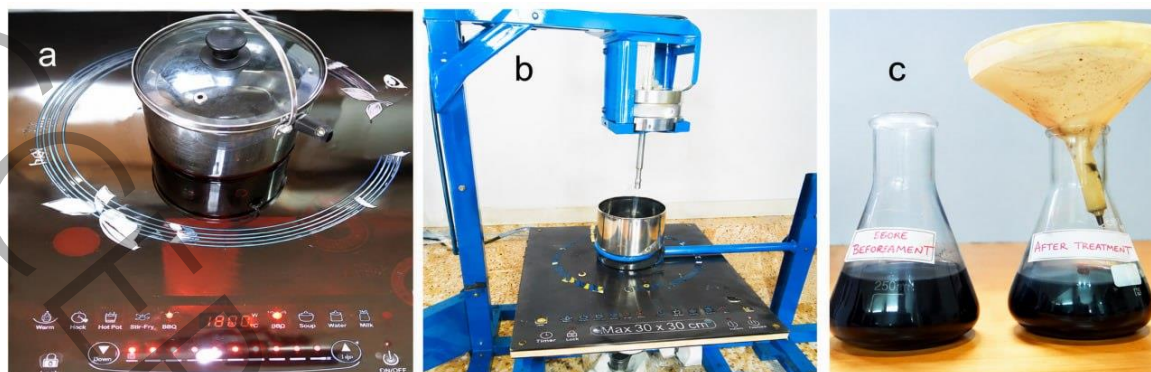


Fig. 1. Heating waste engine oil (A), Acid treatment (B), Pretreated oil and used oil (C)

2.3. Grease formation and manufacturing of test samples

The grease samples were developed through a series of four distinct steps with percentage modifications, as analyzed in Minitab using extreme vertices mixture designs. Extreme vertices mixture designs were selected over standard factorial designs because this research focuses on creating mixtures rather than testing individual factors. In mixture experiments, the response variables are constrained to sum to a fixed total, typically 100%. Soap-thickened greases, including lithium-based types, are produced at temperatures ranging from 160 °C to 250 °C, depending on the base oil composition and reactor configuration. The 160–200 °C production temperature range is standard for manufacturing soap-thickened greases because it promotes phase change without causing thermal degradation [36]. Consequently, the preferred temperature range for lithium grease production operations is 160-200 °C, as this range yields the best chemical and physical conversion results. The reagents, chemicals, and materials were used as indicated in Fig. 2. After processing, the engine oil was heated to 100 °C for 15 minutes, cooled for 10 minutes, then mixed with coconut oil and agitated for 30 minutes. The temperature was subsequently increased to 200 °C to achieve homogenization, then cooled, and finally decreased gradually to 60 °C. Thus, 14 grease samples were formulated following the procedure shown in Fig. 3.

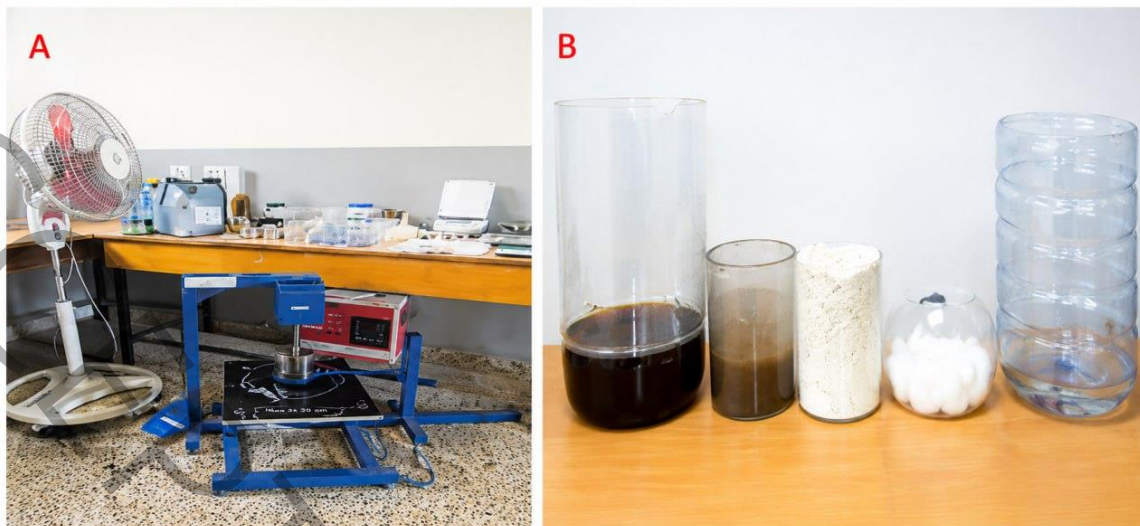


Fig. 2. Materials: (A) Apparatus, and (B) Chemicals

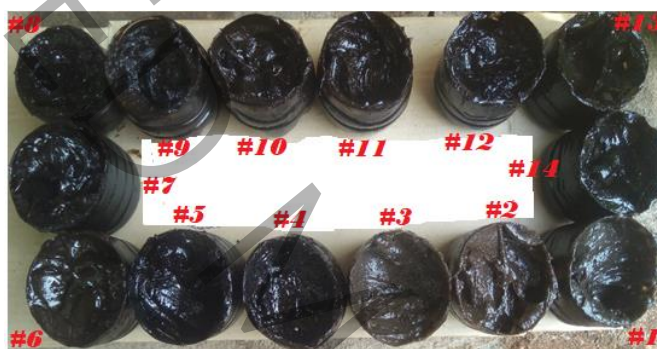


Fig. 3. Prepared greases

2.4. Grease Analysis

To explore the properties of the grease composition, several tests were conducted. These included dropping point, consistency, oil separation, and copper corrosion of the lubricating Grease. Characterizing grease properties solely by thickener type is not straightforward, as the composition and performance additives used have an equal or greater influence on the Grease's properties than the thickener type[38, 39]. The Grease was blended in various proportions of metallic soap and additives, and the resulting formulations were analyzed and tested in accordance with ASTM standards for the treatment of waste motor oil. The dropping point of the Grease is the temperature at which a drop of Grease falls from a cup, marking the beginning of the grease analysis, as outlined in ASTM D-2265/D-566 [40, 41]. Similarly, the Dropping Point of Lubricating Grease test provides a qualitative measure of grease's thermal resistance in applications requiring a semisolid lubricant.

Additionally, oil separation tests were performed in accordance with ASTM D-06[29] [42] in which a weighed grease sample was placed in a wire-cloth filter, suspended in a beaker, and heated for 30 ± 0.25 hours under static conditions. Moreover, per ASTM D 2595, Evaporation Loss of Lubricating Grease [43], a heating device maintains a weighted grease sample in an evaporation cell at the designated test temperature, exposing the grease surface

to heated air for 22 ± 0.1 hours. The weight loss from evaporation is then calculated. Finally, ASTM D 4048 outlines the procedure for copper corrosion testing of lubricating Grease [44]. A copper strip is submerged in Grease and heated at 100°C for 24 hours; upon removal, it is cleaned and compared to the ASTM copper strip corrosion criteria.

3. Results and Discussion

The experimental findings for greases formulated with various base oils (treated waste engine oil) compositions, lithium hydroxide, and additives are shown in Table 1. The quantities of treated waste engine oil, thickener, and additives used in many runs significantly affect the yield and color of the Grease, even though all prepared greases were checked at the time of production. The formulation of these raw ingredients under appropriate temperature conditions resulted in 14 grease samples, and four grease attributes (drop point, oil separation, evaporation loss, and copper corrosion test) were evaluated.

Table 1. Experimental results of the produced Greases

Sample number	Base Oil grams (%)	LiOH gram (%)	additives gram (%)	Mixing temperature ($^\circ\text{C}$)	Drop Point ($^\circ\text{C}$)	Oil Separation (%)	Evaporation Loss (%)
1	80	10	10	160	190	1.1	1
2	85	10	5	160	200	0.12	0.63
3	80	15	5	160	190	2.7	0
4	81.66	11.67	6.67	160	210	0	0.27
5	80.83	10.83	8.34	160	195	0.55	0.2
6	83.33	10.83	5.84	160	180	0.86	0.74
7	80.83	13.33	5.84	160	200	0	0.78
8	80	10	10	200	170	2.2	0.83
9	85	10	5	200	190	0	0.77
10	80	15	5	200	190	0.51	0.27
11	81.66	11.67	6.67	200	180	2.42	0.3
12	80.83	10.83	8.34	200	170	2.47	0.63
13	83.33	10.83	5.84	200	190	1.72	0.58
14	80.83	13.33	5.83	200	205	0	0.28

- % weight percentages

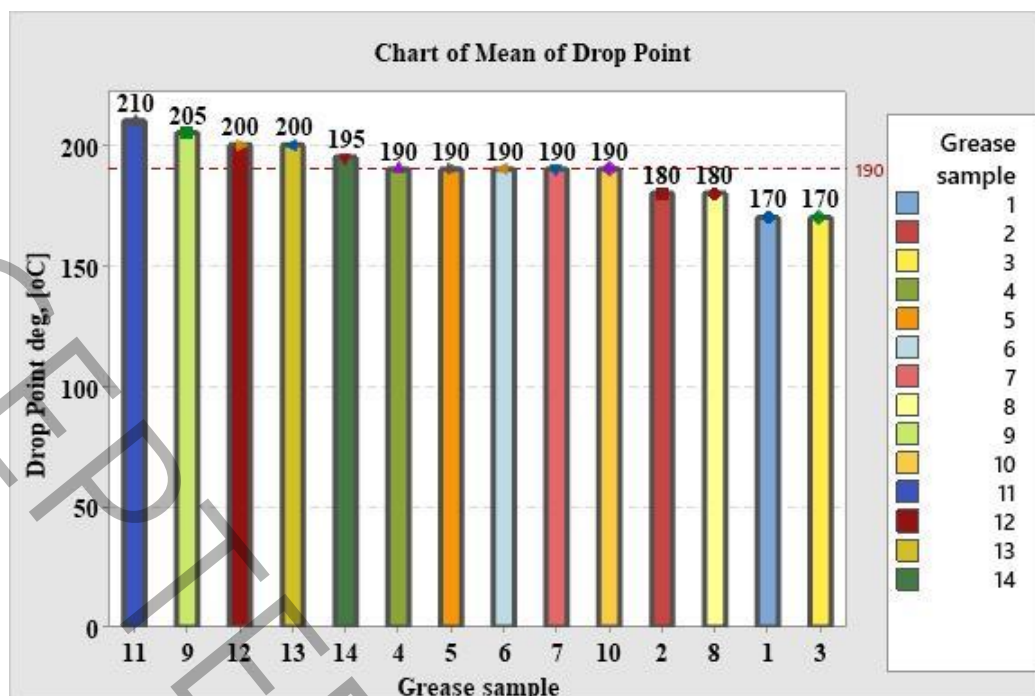


Fig. 4. Dropping point of Grease samples

Table 1. and Fig. 4 indicate that the dropping points of grease samples 11, 9, 12, 13, 14, 5, 6, and 10 are within the permissible range. However, of the fourteen grease samples tested, four (Samples 1, 2, 3, and 8) failed to meet the ASTM D2265 dropping point requirement of at least 200°C for automotive applications. Analyzing the compositions and processing conditions provides useful insights into the main causes of failure. Accordingly, samples 2 and 3 had 5% additives, which is quite low compared with the successful samples (usually 6.67–10%). Sample 3 also contained the highest LiOH level at 15%, which might have disrupted the thickeners' network or induced diluting effects, resulting in a loss of grease structure. Sample 1, although made with an appropriate additive content (10%), was blended at 160°C, which was not high enough to fully saponify, thereby compromising the quality of the soap gel. Conversely, Sample 8, having the same composition as Sample 1, was exposed to a substantially higher blending temperature of 200°C. Overheating may induce structural breakdown of the soap or premature phase separation, thereby decreasing the dropping point [45]. The findings show that maintaining an appropriate balance among additive content (typically 6–8%), LiOH concentration (preferably 10–13%), and mixing temperature (approximately 160°C) is critical for producing Grease with acceptable thermal stability. This proposition conforms to optimal practices described in the NLGI Lubricating Grease Guide (2016) and related literature on lithium soap greases that utilize recycled or vegetable oils. Thus, compared with the other greases examined, Grease Sample 11 has a higher dropping point. The ratios of base oil, thickener, and additives, as well as the mixing temperature, account for the variances in the dropping points. Lithium greases often have a high dropping point in the region of 190–220 °C, which is the greatest among basic soap greases, as specifically mentioned in [28]. The effect of LiOH and additives varies with the Grease's dropping point; the line is not horizontal and shows a higher mean than the other parameters. Moreover, the temperature of 160 °C has a larger

mean dropping point than that of 200 °C. Base oil also impacts the dropping point of the grease lubricant. In general, the impact of LiOH is beneficial compared with additives, base oil, and temperature, as shown in Fig. 5.

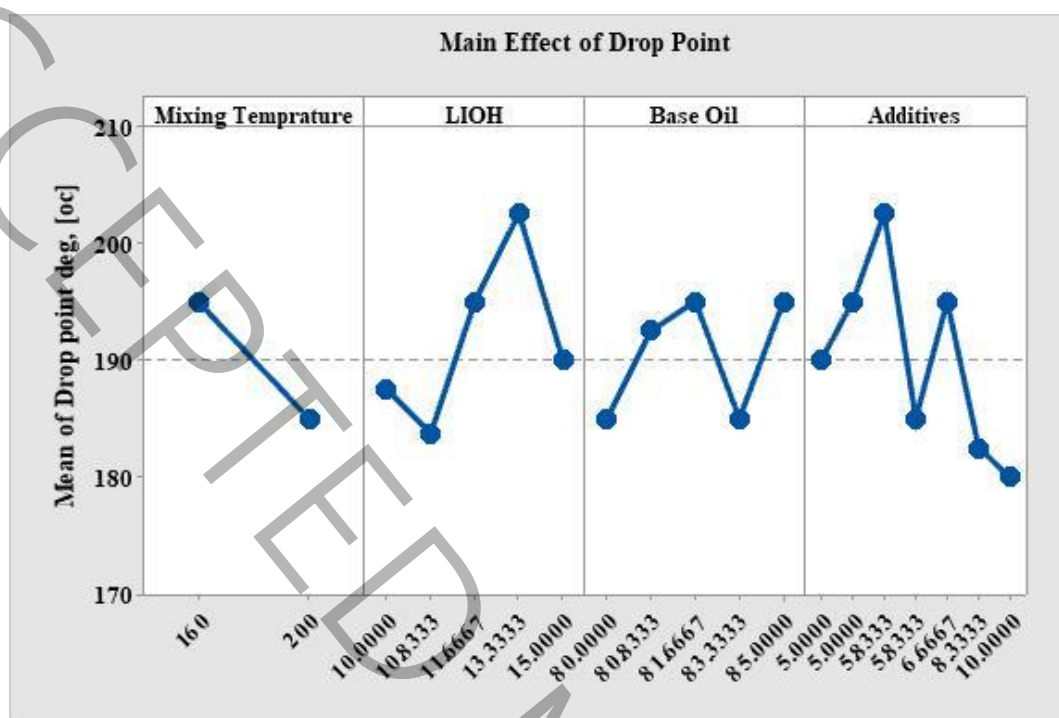


Fig. 5. Effect plot of Drop Point

The response trace plot in Fig. 6 shows how each component affects the response relative to the reference mixes. The reference mix represents the centroid of the design vertices, and each component is shown as a percentage of the total. The acceptance rating of the drop point decreases slightly as the proportion of base oil in the combination increases. At the same time, as the proportions of the other mixture components increase, the acceptance rating rises. The acceptance rating in the reference blend is nearly optimal; therefore, reducing the base oil percentage relative to it may be beneficial.

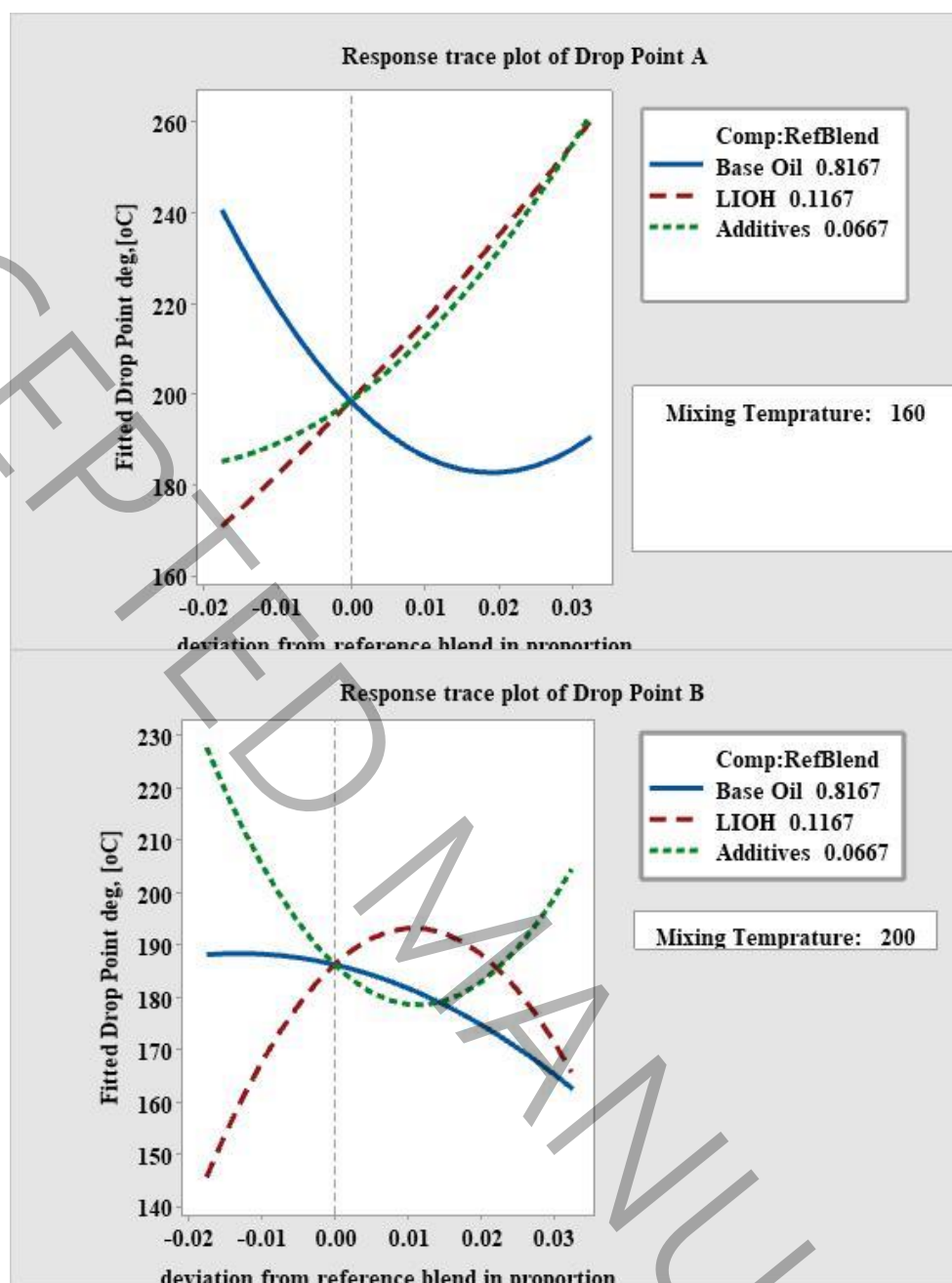


Fig. 6. Response Trace Plot of Drop Points (A) and (B)

LiOH significantly affects the Grease's drop point, with its acceptability rating increasing steeply as the LiOH percentage in the mixture rises and decreasing sharply as LiOH levels fall. The drop point's acceptability rating also rises slightly with increased additives and decreases with decreased additives. Fig. 6(B) shows that the Grease's drop-point acceptability rating decreases with increasing base oil and other components, but increases marginally when base oil decreases. The drop point's acceptability rating also declines as the LiOH percentage increases, indicating that the LiOH level in the reference blend is nearly ideal. Additionally, the drop point's acceptability rating increases with decreasing additive percentage and increasing other components. In contrast, it marginally increases with a reduction in additives and a rise in other components. Reducing the additive

percentage relative to the reference mix can improve the acceptability rating of the drop point.

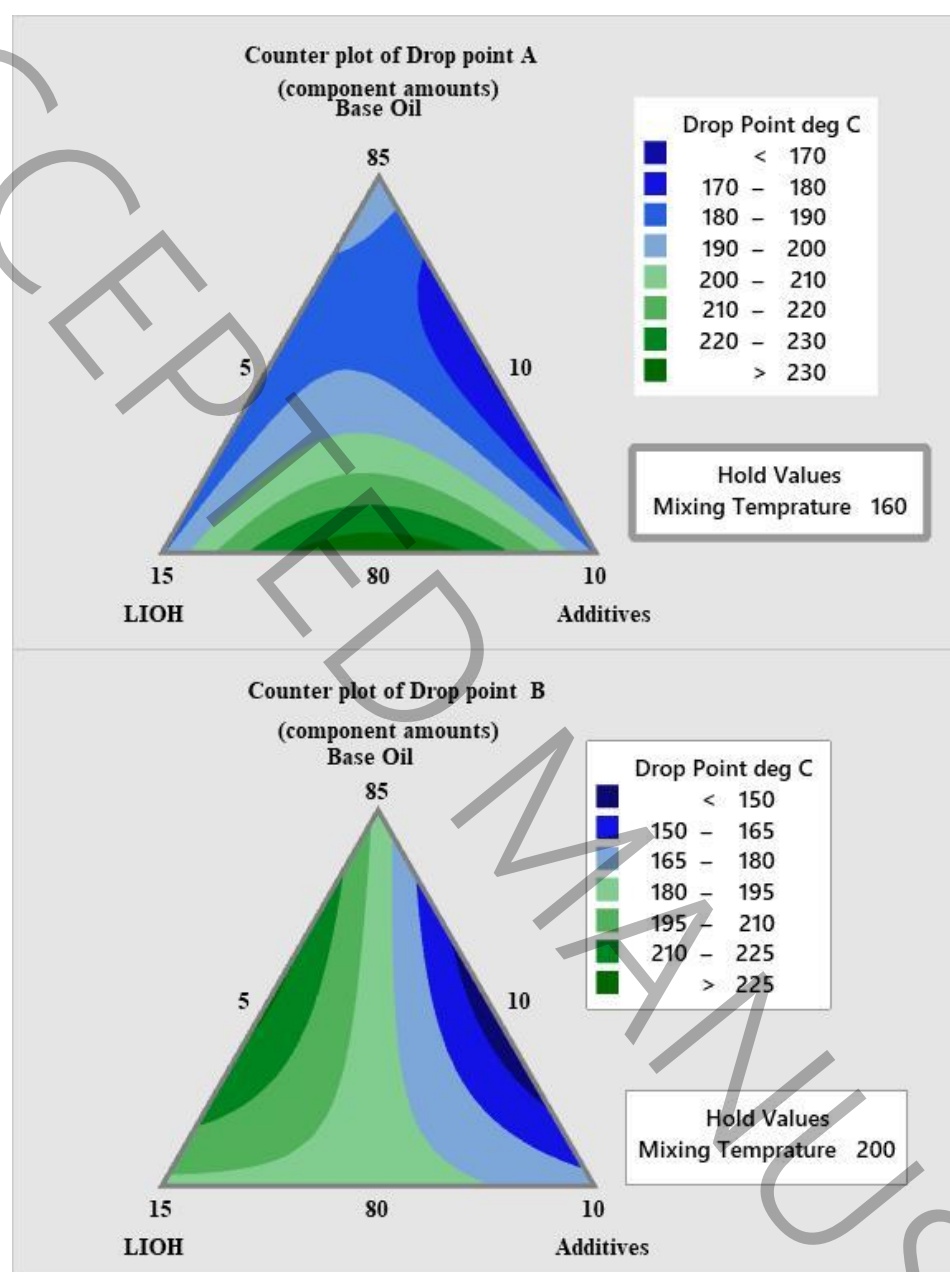


Fig. 7. Counter Plot of drop points (A) and (B)

Fig. 7(A) presents the contour plot of the drop point, dividing the feasible area into eight ranges for drop point effects: less than 170°C, 170-180°C, 180-190°C, 190-200°C, 200-210°C, 210-220°C, 220-230°C, and more than 230°C. The plot shows that the drop point ranges from 200°C to 230°C, influenced by the base oil and LiOH content, and remains constant at 230°C when the base oil and additive content are unchanged. The darkest area signifies the impossible region above 230°C, while the lighter area indicates the feasible areas for each range. Similarly, Fig. 7(B) displays the contour plot of the drop point, categorized into seven ranges for drop point effects: less than 150°C, 150-165°C, 165-

180°C, 180-195°C, 195-210°C, 210-225°C, and more than 225°C. This plot shows the change in drop point from 180°C to 195°C as a function of LiOH content and base oil, with additives held constant. The darkest area again denotes impossible regions above 225°C, while the lighter area indicates the feasible areas for each range. This understanding aids in predicting temperature changes. These temperature effects can be attributed to interactions among different factors within the study's given range.

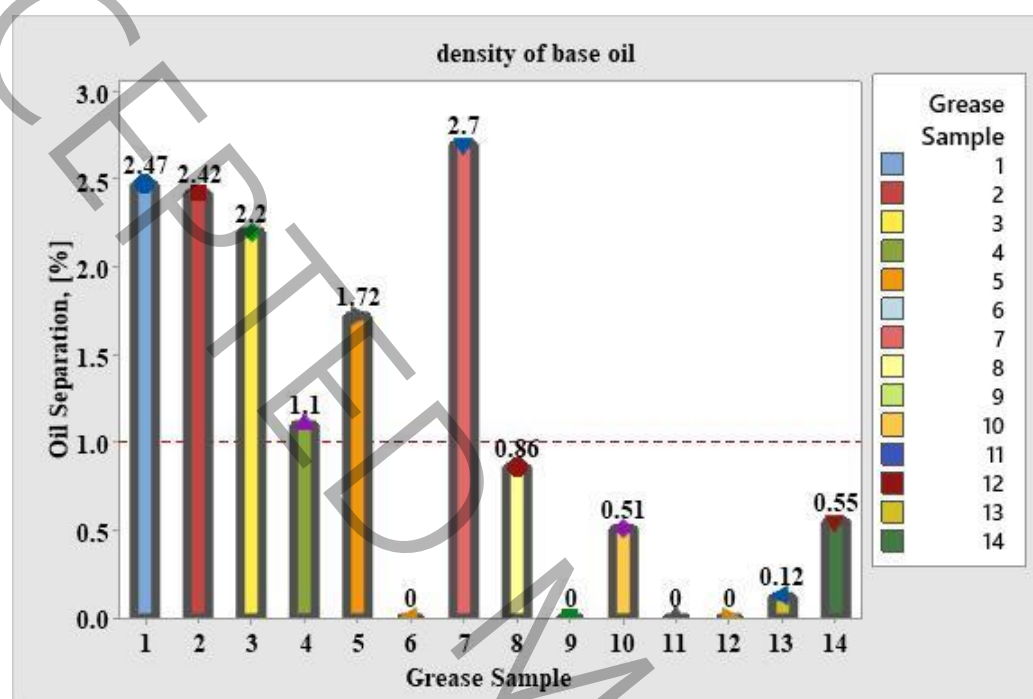


Fig. 8. Oil Separation of Grease Samples

Oil separation during storage can alter grease consistency, reducing effectiveness when base oil separates from other ingredients. Fig. 8 and Table 1 show that grease samples 6, 9, 11, and 12 exhibited 0% oil separation, whereas samples 8, 10, 13, and 14 showed oil separation comparable to samples 1, 2, 3, 4, 5, and 7. In contrast, other samples exhibited oil separation greater than 1%. While 0% oil separation would be ideal, some separation may be necessary and beneficial, depending on the application. The variation in oil separation from sample to sample (0% to 2.7%) is due to differences in structure and composition, particularly the percentages of lithium hydroxide (LiOH), base oil, additive ratios, and mixing temperatures. The highest oil separation rates were observed in samples 3 (2.7%), 11 (2.42%), 12 (2.47%), and 8 (2.2%), which tend to share common characteristics: either low amounts of LiOH (5–6.67%), high processing temperatures (200°C), or both. Low thickener concentration can compromise the soap matrix's ability to retain base oil, leading to increased oil bleed. Excessive mixing temperatures, as seen in samples 8 and 12, can destabilize the thickener's microstructure or lead to incomplete dispersion, contributing to long-term oil separation. Samples with an even proportion of LiOH (6.67–8.34%) and moderate amounts of additives (10–13%), such as samples 4, 5, and 7, displayed 0% oil separation, indicating an optimal grease microstructure. Values are generally within or near acceptable ranges; commercial lithium soap greases typically have oil separation values below 3% according to ASTM D1742, with high-performance

varieties aiming for below 1% to ensure sufficient oil retention and mechanical stability [3]. These findings highlight the need to carefully balance saponification and formulation variables to achieve structural integrity and reduce oil bleed, in line with commercial grease manufacturing practices.

The LiOH and additives appear to affect oil separation in the Grease, as the effect line is not horizontal and has a lower mean than the temperature. Additionally, the base oil has a lower mean at 85%, and at 160°C the mean oil separation is lower than at 200°C. Generally, LiOH and additives positively influence oil separation as temperature increases, and the base oil also has a slight impact on oil separation, as shown in Fig. 9.

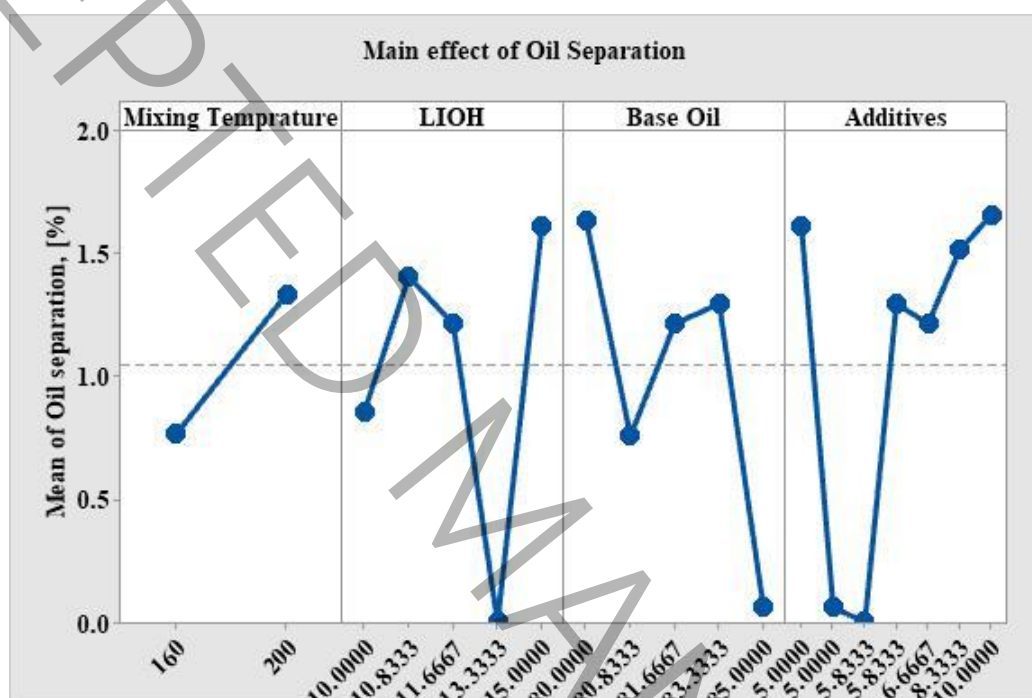


Fig. 9. Main Effect Plot of Oil Separation

In Fig. 10(A), the oil separation rating increases as the percentage of base oil in the mixture rises, while the other components decrease. It declines as the base oil content lessens. The rating decreases slightly with increasing LiOH, but rises as the LiOH level decreases. The oil separation rating drops as the proportion of additives in a grease lubricant mixture increases, and it decreases slightly as that proportion decreases. Base oil significantly affects oil separation, and reducing its percentage could enhance the acceptance rating of the reference blend, which is already close to ideal.

In Fig. 10(B), the acceptance rating for Grease's oil separation decreases as the base oil percentage rises, and it also falls when the base oil proportion decreases. The diagonal LiOH percentage in the mixture does not affect oil separation and may even have a negative impact. As the proportion of additives increases and the other components decrease, the mixture's acceptance rating improves; however, the oil separation rating declines as the additive proportion in the reference blend approaches optimal levels.

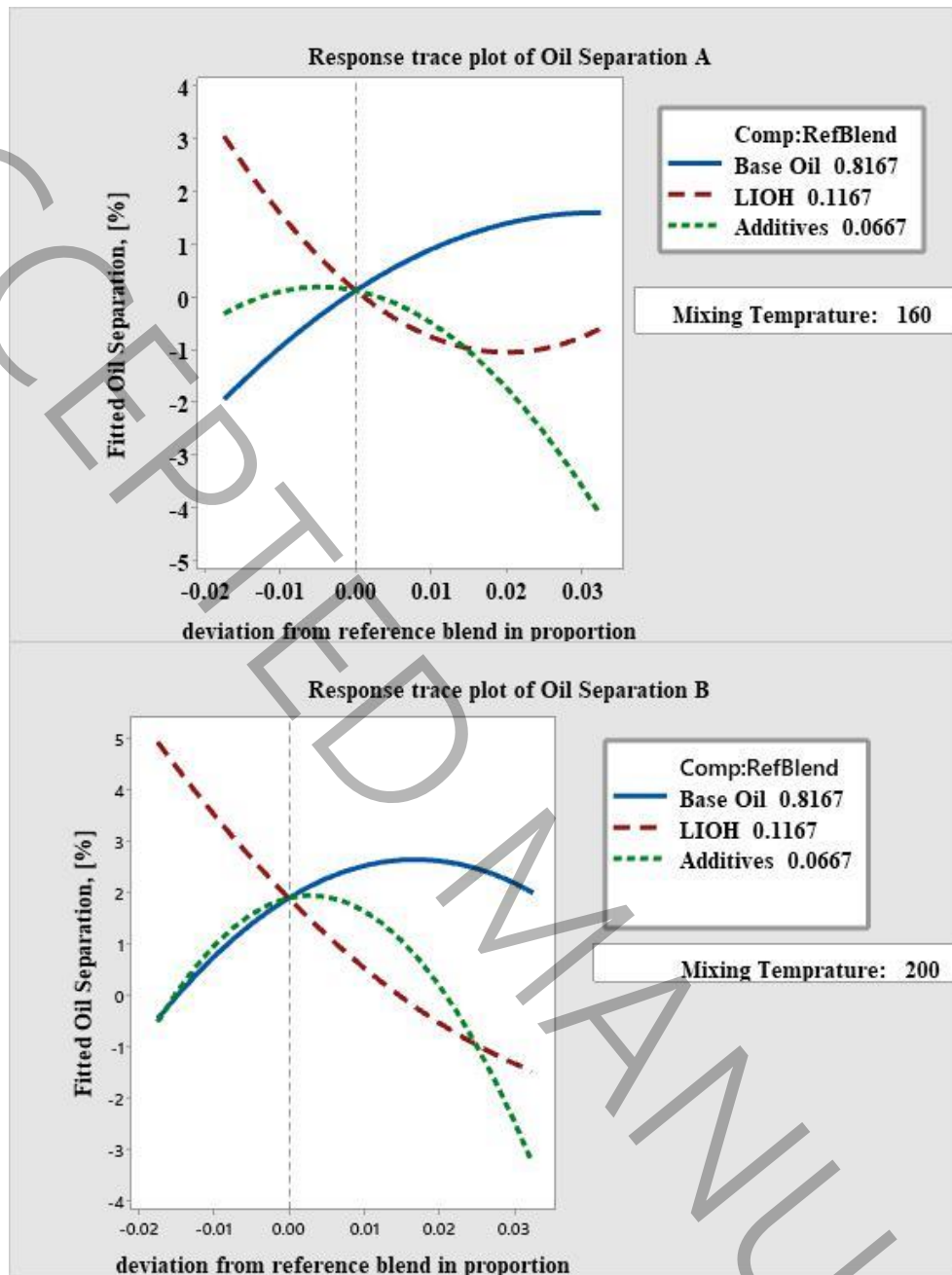


Fig. 10. Response Trace Plot of Oil Separation (A) and (B)

Fig. 11(A) shows the contour plot of oil separation, which divides the feasible area into six ranges for oil separation effect: less than -1%, -1% to 0%, 0-1%, 1-2%, 2-3%, and more than 3%. This plot offers insight into oil separation, particularly the change in oil separation between 0 and 1% (indicated by the Limon color) in two plotted areas. The first area represents a decrease in base oil content and an increase in additive content, with a corresponding oil separation of 0-1% at constant LIOH content and decreasing additive content. The lighter (<0) and darkest (>3) regions indicate impossible areas (unacceptable), as the desired oil separation range should be between 0 and 2%.

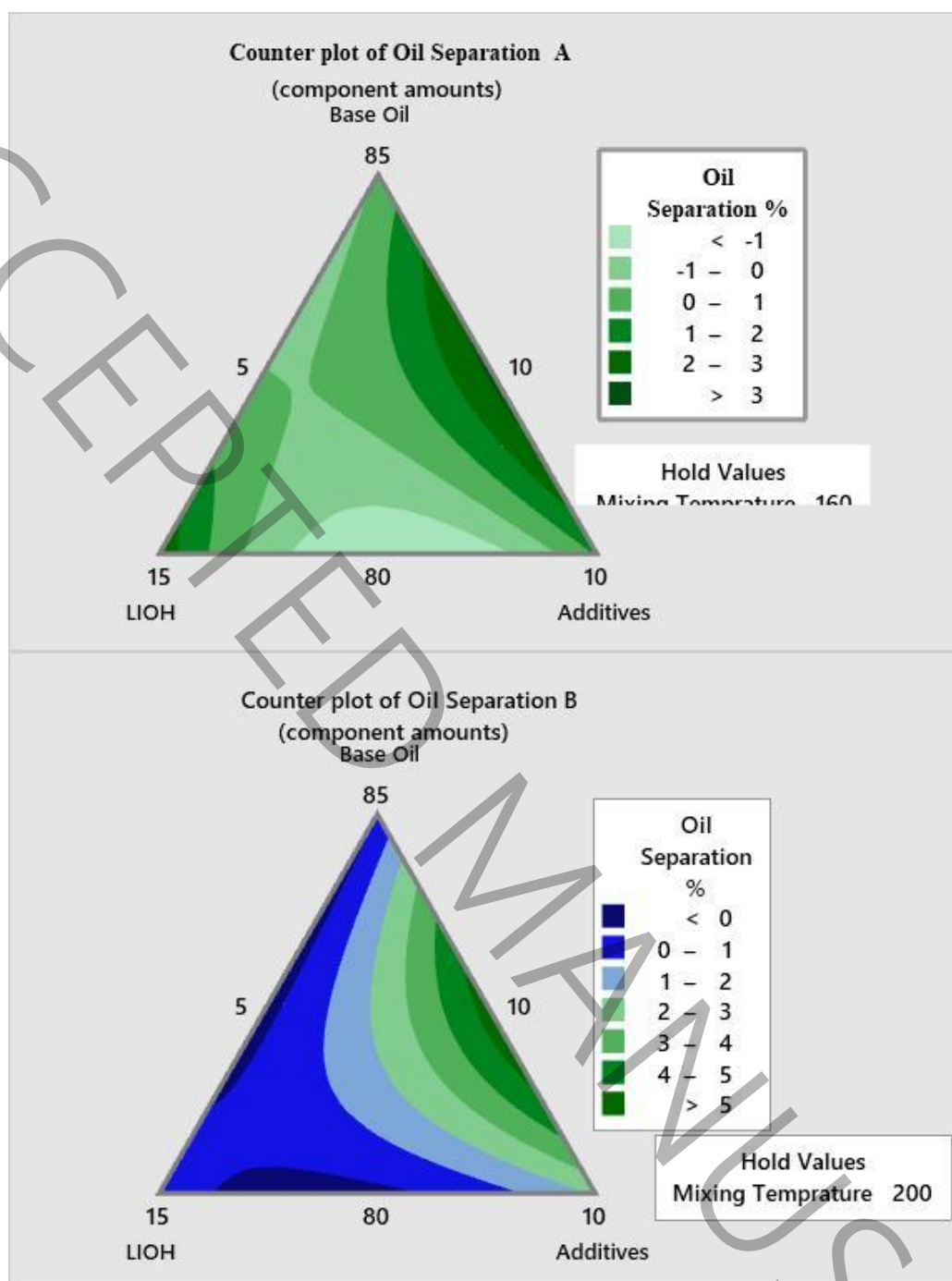


Fig. 11. Counterplot of oil separation (A) and (B)

Fig. 11(B) shows the contour plot for oil separation, dividing the feasible area into seven ranges for the oil separation effect: less than 0%, 0-1%, 1-2%, 2-3%, 3-4%, 4-5%, and more than 5%. This plot enhances understanding of oil separation, particularly the shift from 0% to 1% when the LiOH content in the base oil remains constant and additive content is minimized. The two darkest areas (<0 and >4) indicate unacceptable regions, as the desired range for oil separation is between 0% and 1%. Contour plots analyze the optimal response across multiple response variables, specifically drop point, oil separation, and evaporation loss. Minitab maintains a constant temperature of either 160 or 200°C to compute fitted

response values, without considering the third predictor, mixing temperature. This method helps determine the direction of improvement based on the required qualities.

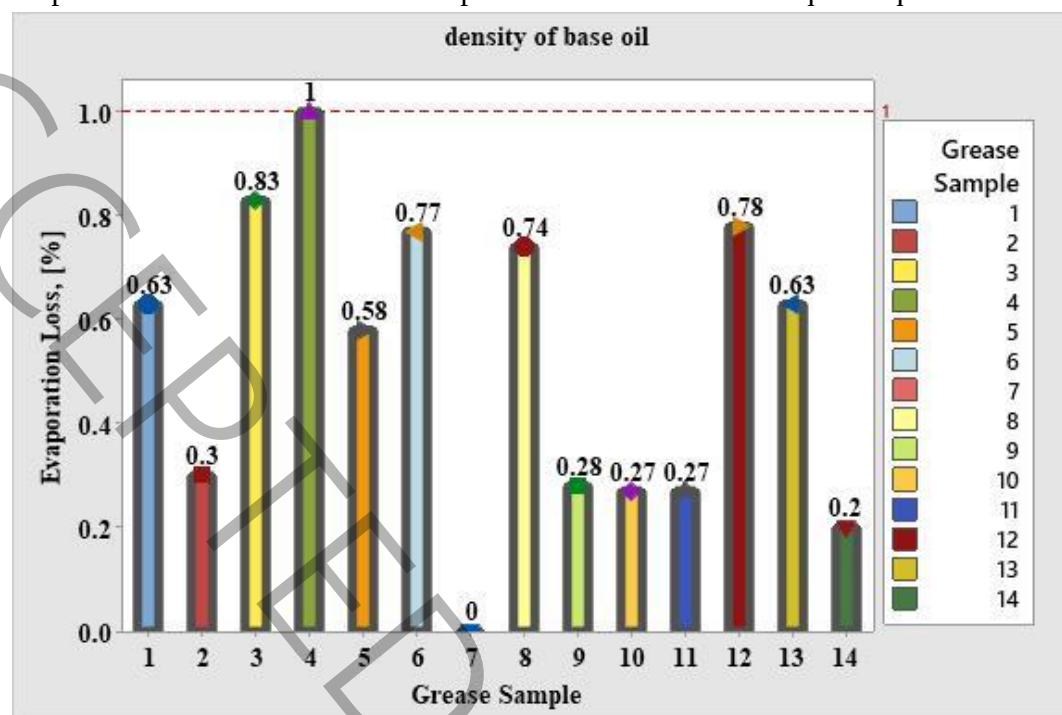


Fig. 12. Evaporation Loss of Grease Samples

Fig. 12 shows that sample grease 8 had a lower evaporation loss percentage than the other grease samples. Nearly all sample greases exhibited similar evaporation loss, with results below 1%. When a significant amount of oil evaporates from Grease, it becomes thicker or the oil viscosity changes, which in turn affects the properties of the grease lubricant and ensures that the mass loss of Grease remains within the product application specifications.

Table 2. Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Mixing Temperature	1	0.00011	0.000114	0.00	0.973
Error	12	1.13649	0.094707		
Total	13	1.13660			

Observational analysis of experimental data trends, particularly the interpretation of response traces and contour plots produced during the mixture design study, led to the conclusion that temperature (between 160 and 200 degrees Celsius) had a negligible influence on the evaporation loss of Grease (see Table 2). These graphical tools provided a visual representation of how changes in component ratios (LiOH, additives, and base oil) and process conditions (temperature) affected performance characteristics, including evaporation loss. In all fourteen created samples, the evaporation loss graphs showed no strong directional slope or curvature as temperature rose from 160 °C to 200 °C; while mixing temperature changed, the mean evaporation loss values stayed within a limited and steady range; other factors, such as oil separation, showed much stronger sensitivity to

temperature increases, especially at 200 °C. However, LiOH and additives appear to influence the evaporation loss of the Grease, as their effect line is not horizontal and shows a lower mean than the base oil, which has a mean of 81.6%. Typically, LiOH and additives positively impact evaporation loss compared to base oil. Nonetheless, base oil also has a slight effect on evaporation loss, as shown in Fig. 13.

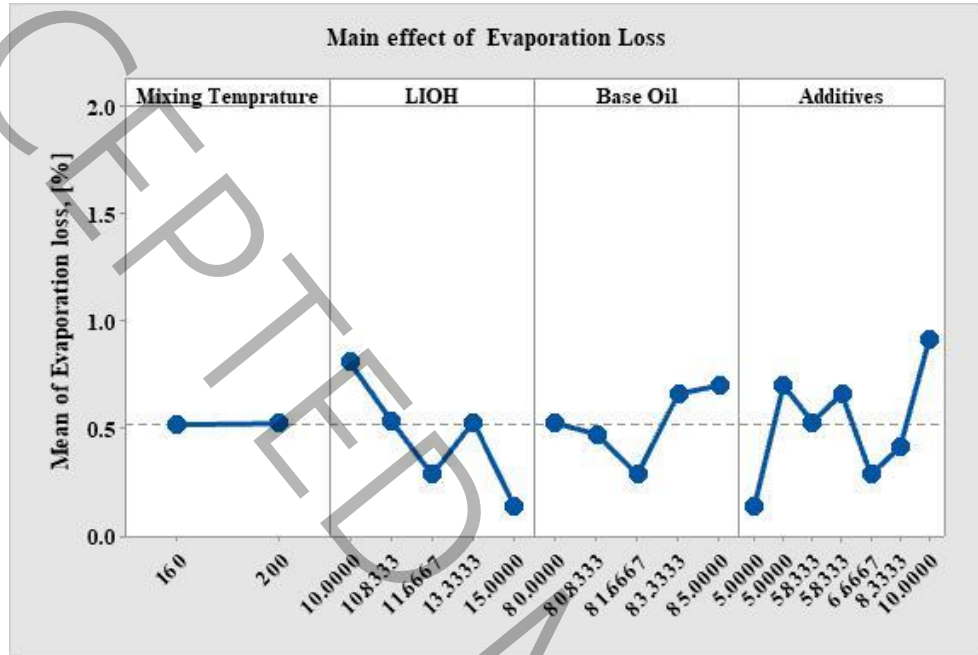


Fig. 13. Main Effect Plot of Evaporation Loss

For this study, the reference blend for generating response trace and contour plots was selected as the centroid of the feasible design space defined by the extreme-vertices mixture design in Minitab®. This blend, containing approximately 81.66% treated waste engine oil, 6.67% lithium hydroxide, and 11.67% additives (Graphite and CaCO_3 , with a graphite-to- CaCO_3 blending ratio of 1:1), represents the average composition within the design space. Using this centroid, the influence of each component on evaporation loss can be assessed while holding the others constant, as in an optimal mixture design. However, the study recognizes that for multi-component mixtures, such interpretations were limited by potential interactions among components that cannot be fully captured by one-dimensional trace plots[46].

Linear regression models were further used to assess the influence of blending temperature on the quality features of Grease. The regression equation for predicting the drop point is shown in Eq. (1).

$$\text{DropPoint} = 190 + 5(T_{160} - T_{200}) \quad (1)$$

Where: *DropPoint* = predicted drop point (°C),

T_{160} = coded variable for the mixing temperature at 160 °C,

T_{200} = coded variable for the mixing temperature at 200 °C.

Similarly, the evaporation loss was predicted using the regression equation given in Eq.(2).

$$\epsilon = 0.520 - 0.0029T_{160} + 0.0029T_{200} \quad (2)$$

Where: ϵ = predicted evaporation loss

T_{160} = coded variable representing the first mixing temperature in (°C).

T_{200} = coded variable representing the second mixing temperature in (°C).

The positive coefficient for T_{160} and the negative coefficient for T_{200} in the drop point model indicate that increasing the first mixing temperature raises the drop point, whereas increasing the second mixing temperature lowers it. Conversely, the evaporation loss model indicates that increasing the first mixing temperature reduces evaporation loss, whereas increasing the second mixing temperature slightly increases it within the experimentally investigated region.

Fig.14(A) shows that the evaporation loss rating decreases slightly as the base oil content in the combination increases. In contrast, the other mixture components decrease, while the base oil proportion increases slightly as the other components increase. The evaporation loss rating decreases with increasing LiOH; however, it also decreases with decreasing LiOH. The rating increases with the addition of LiOH but decreases with the addition of additives, indicating that these factors do not affect the grease lubricant's evaporation loss. In fact, in Fig.14(B), the grease's oil loss by evaporation rating increases as the matrix increases in the mixture combination, while it decreases slightly as the matrix decreases. With increasing LiOH, evaporation loss decreases, whereas with decreasing LiOH, ratings increase. The evaporation loss rating increased with higher additive levels, while the acceptability rating decreased with lower additive levels. During base oil reduction, the increase in LiOH and the constant additives result in a nearly ideal evaporation loss.

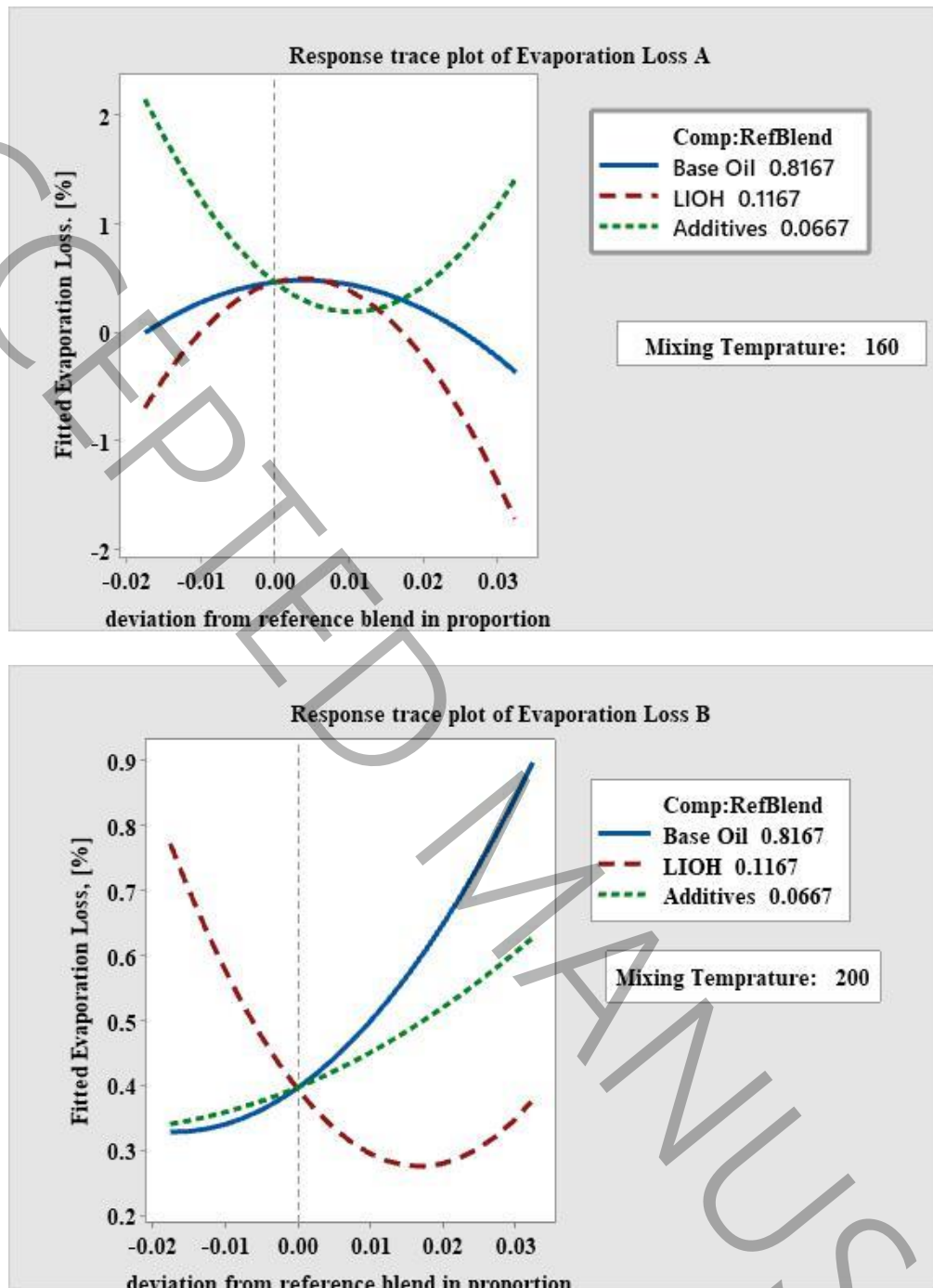


Fig. 14. Response Trace Plot of Evaporation Loss (A) and (B)

The drop-point function, which defines the feasible range of drop-point effects from 190 °C to 240 °C, is shown in the Overlaid Contour Plot (A) in Fig.15. However, the data in Table 1 indicate that the lower and upper result ranges are 170- 210 °C, and the lithium grease drop point falls between 190 and 240 °C. The white area in the figure represents the potential combinations that can achieve a drop-point effect between 190 °C and 240 °C through the interplay of oil separation and evaporation loss. Optimal responses are highlighted within this white area, particularly near the surface, where LiOH concentrations are higher. Specifically, the region where LiOH is at 15% concentration tends to yield the

most favorable outcomes when combined with 6% additives. By establishing these two coordinates, the base oil proportion is inherently determined.

Table 3: Copper Corrosion Result

Run Order	Base Oil in grams	LiOH in grams	Additives in grams	Mixing Temperature (°C)	Copper Corrosion
1	80.00	10.00	10.00	160	1b
2	85.00	10.00	5.00	160	1b
3	80.00	15.00	5.00	160	1b
4	81.66	11.67	6.67	160	1a
5	80.83	10.83	8.34	160	1b
6	83.33	10.83	5.84	160	1a
7	80.83	13.33	5.84	160	1b
8	80.00	10.00	10.00	200	1a
9	85.00	10.00	5.00	200	1a
10	80.00	15.00	5.00	200	1a
11	81.66	11.67	6.67	200	1a
12	80.83	10.83	8.34	200	1a
13	83.33	10.83	5.84	200	1b
14	80.83	13.33	5.83	200	1b

ASTM D4048 is the standard test method for assessing the corrosiveness of lubricating Grease to copper under static conditions. It is a critical quality control test used by lubricant manufacturers and industries such as automotive and aerospace to ensure that Grease will not chemically attack components made of copper or its alloys, such as bearings and bushings. The method involves preparing a copper strip, fully immersing it in a Grease sample, and heating it in an oven at 100 ± 1 °C for 24 ± 5 min. At the end of this heating period, the strip is removed, washed, and compared with the Copper Strip Corrosion Standard (color scale). Most industrial and automotive specifications require a Grease to achieve a 1a or 1b score.

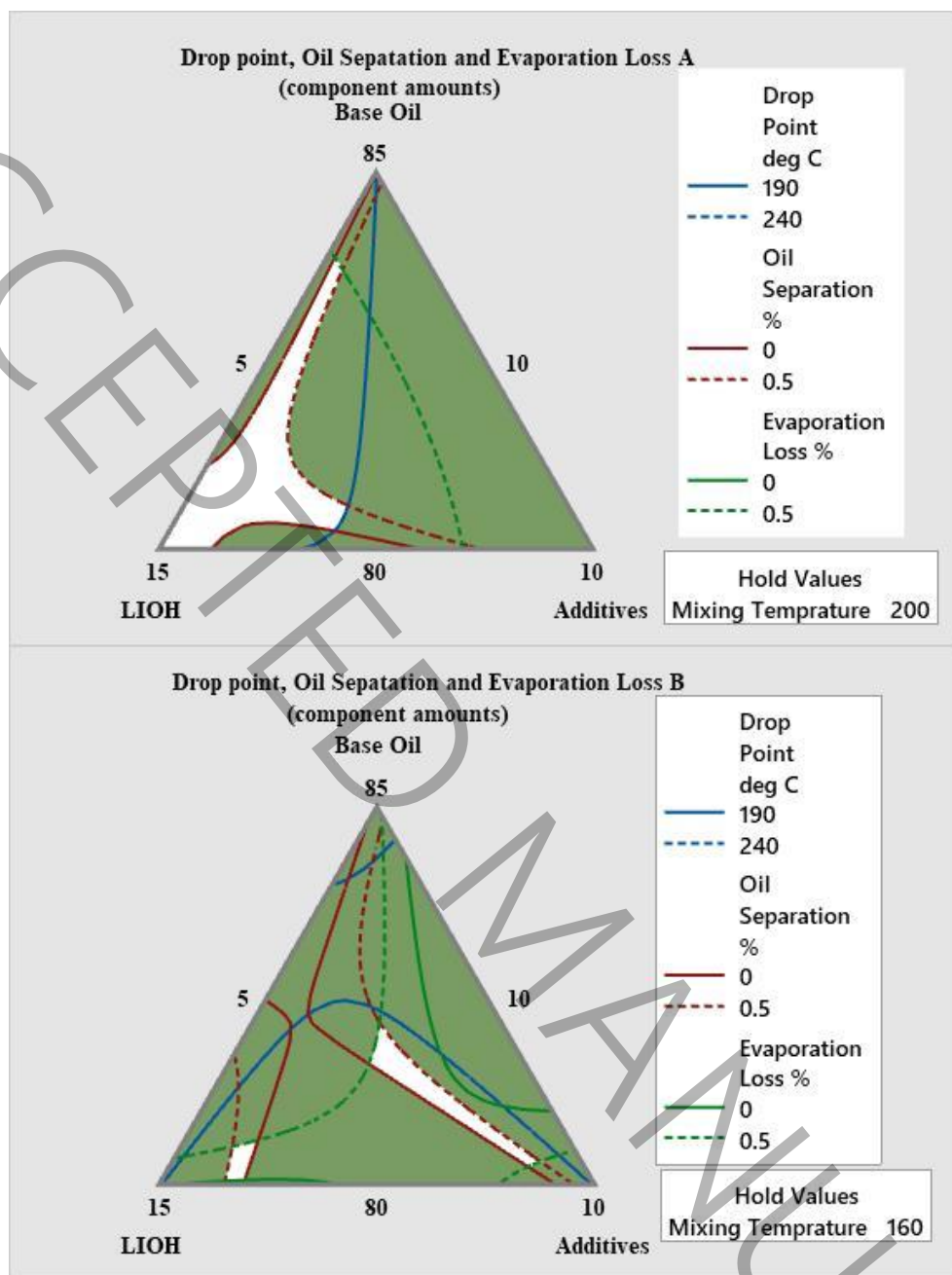


Fig. 15. Overlaid contour plot of a drop point, oil separation, and evaporation (A) and (B)

The overlaid contour plots were derived from experimental data on fourteen systematically formulated grease samples, with key properties such as drop point, oil separation, and evaporation loss measured in accordance with relevant ASTM standards (e.g., ASTM D566/D2265 for drop point). Reproducibility was assessed through triplicate testing of optimal formulations, particularly those near 15% LiOH and 6% additives, yielding a standard deviation of $\pm 2.5^{\circ}\text{C}$ for drop point, indicating high repeatability at the lab scale. The response surfaces, validated by an $R^2 > 90\%$ for drop-point prediction, confirm the model's robustness. However, industrial-scale production may introduce variability due to factors such as feedstock inconsistency, mixing dynamics, and environmental conditions.

To address this, we recommend applying operational tolerances in accordance with ASTM D4950–19 to ensure consistency and performance during scale-up.

The drop point is low, and oil separation and evaporation losses are large in the near-surface region with a low concentration of LiOH. Fig. 15(B) shows the plot for the drop point, outlining the permissible region for the effect between 190 °C and 240 °C. Two white areas, farther from the surface, represent the best-performing LiOH and base oil toward the upper part of the generated plane and, from the centroid toward the additives, the highest performance as well.

Automobile manufacturers conduct copper corrosivity tests to ensure that lubricants do not corrode copper or copper-alloy bearings. Although this test does not measure the Grease's stability or resistance to external corrosion, it ultimately ensures that the lubricant will protect the metal surfaces it contacts. Table 2 represents the corrosivity results obtained with the basic oil-and-grease formulation incorporating LiOH and additives. The results indicate that treated used engine oil (Classes 1a and 1b) caused only minor tarnishing of copper strips compared with strips that had recently been polished. The Overview also notes that copper strips exhibited little tarnishing when mixed with Grease made from treated waste engine oil, calcium carbonate, and Graphite, which could help protect metals against corrosion.

During grease production and preparation, the product evolved from the desired oily state to a semisolid state as the reaction proceeded in the beaker while being stirred. The color of the final product also varied depending on the base oil, ranging from light gray to black, including shades of dark and light brown. Greasability also became more complex, with higher amounts of thickener or a base oil ratio closer to the existing one, which affected LiOH production. The yield of the final grease product also varied with the base oil content: a high base oil ratio usually yielded the expected amount from the starting material, whereas a low base oil concentration combined with a high thickener concentration resulted in a lower-than-expected grease yield. It was also observed that increasing the base oil proportion made the final grease product greasy (similar to frozen oil), while increasing the thickener proportion resulted in a stiffer final product.

The evaluation of experimental uncertainty depends on the nature of each test. For the Drop Point determination, reproducibility was primarily limited by the instrument's resolution, resulting in a systematic uncertainty of at least $\pm 5^{\circ}\text{C}$. The oil separation and evaporation loss tests depended critically on mass determination, with a controlled analytical uncertainty of only $\pm 0.01\%$. This magnitude difference shows that the study's overall variability stems from thermal control and the visual/physical detection of the dropping point. At the same time, mass changes were maintained under strict statistical rigor.

The ASTM D2265 standard allows for acceptable repeatability up to 7°C . The evaluation of experimental uncertainty was structured according to the methodology and nature of each test. For the Drop Point determination, the tests were conducted in accordance with ASTM D2265. Reproducibility in this case was limited by the system's physical resolution, assuming an instrumental uncertainty of $\pm 5^{\circ}\text{C}$.

4. Conclusions

This study demonstrated that treated waste engine oil can be recycled into lithium-based Grease. An optimal blend of 81.66% treated waste oil, 11.67% lithium hydroxide, and 6.67% additives (Graphite and calcium carbonate), identified using Minitab's Extreme Vertex Mixture Design, exhibited excellent thermal properties. The developed Grease met ASTM criteria for drop point, oil separation, evaporation loss, and copper corrosion resistance. Both LiOH and additives reduced oil separation and evaporation loss, but the concentration of lithium hydroxide significantly influenced thermal stability. Mixing at 160°C yielded better structural stability than at 200°C. Notably, evaporation loss remained consistently low across all samples, confirming formulation stability. Recycling waste engine oil reduces hazardous waste and conserves resources, offering clear financial and environmental benefits. This work supports the sustainable manufacture of lubricants and the principles of the circular economy by converting waste oil into high-performance Grease.

Data availability

The data supporting this study's findings are available from the corresponding author upon reasonable request.

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