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RESEARCH

# Graphene–Curcumin Reinforced Waste Cooking Oil-Based Biogrease: Physicochemical and Tribological Properties

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A B S T R A C T

*This study examines the influence of a graphene–curcumin hybrid additive on the structural and tribological properties of a biogrease derived from waste cooking oil (WCO). Fumed silica was used as an inorganic thickener, while graphene nanoplatelets and curcumin were introduced at different concentrations to enhance the grease matrix. The formulations were characterized using FTIR spectroscopy, cone penetration, dropping point, copper strip corrosion, and pin-on-disk tribological tests. FTIR analysis confirmed preservation of the triglyceride ester structure of WCO and successful incorporation of silica, graphene, and curcumin without chemical degradation. Cone penetration values ranged from 378 to 292 × 0.1 mm, corresponding to NLGI grades 0–2, indicating adjustable consistency, while dropping point tests showed non-melting behaviour up to above 304 °C. Copper strip corrosion ratings of 1–1a indicated non-corrosive behaviour. The graphene–curcumin-modified grease reduced the steady-state coefficient of friction by approximately 40–50% and significantly decreased the wear depth compared with the base WCO–silica grease. The improved tribological performance is attributed to enhanced graphene dispersion and stabilization of the lubricating film within the grease matrix. These results demonstrate the potential of graphene–curcumin reinforced WCO-based biogrease as a sustainable high-performance lubricant.*

## 1. INTRODUCTION

Lubricating greases are critical tribological materials that enable smooth operation in mechanical systems by reducing friction and wear, particularly under boundary and mixed lubrication regimes [1,2]. Conventional greases are typically formulated from mineral base oils, metallic soap thickeners, and performance additives [3]. However, increasing environmental and sustainability concerns—especially regarding the non-biodegradability and fossil origin of mineral-oil-based greases—have become pressing issues [4]. The depletion of petroleum resources and growing awareness of lubricant-related pollution have driven research into renewable bio-based lubricants, reinforcing the principles of waste valorization and the circular economy [5].

In the pursuit of sustainable lubricant development, waste cooking oil (WCO) emerges as a promising alternative. WCO is an abundant and cost-effective feedstock that can be converted into valuable lubricants or greases [6]. The conversion of WCO aligns with ecological objectives by mitigating waste while also addressing the demand for high-performing lubricants. Various studies have highlighted the successful formulation of bio-lubricants from WCO that exhibit enhanced properties, such as a higher viscosity index and lower volatility, compared to conventional lubricants [6,7]. The use of anti-wear additives and chemical modifications can further optimize the performance of WCO-derived lubricants [8].

The utilization of WCO as a precursor for biogrease production offers significant benefits, particularly by reducing environmental pollution from improper oil disposal and decreasing reliance on petroleum-based lubricants [9]. However, WCO-derived greases often face challenges related to oxidative instability, limited load-carrying capacity, and insufficient thermal resistance. These limitations arise from the presence of free fatty acids, oxidation by-products, and poor molecular packing within the thickener–oil matrix. As a result, such greases may suffer from soft consistency, increased wear, and reduced corrosion resistance, particularly under high-load or high-temperature operating conditions. Without reinforcement additives, the internal gel structure formed by fumed silica or soap thickeners tends to collapse under stress, leading to oil bleeding, surface film breakdown, and accelerated wear. Therefore, reinforcing the grease matrix with advanced additives is essential to enhance both its structural integrity and tribological performance [10]. For example, specific formulations have been shown to interact positively with WCO components, potentially improving the characteristics of greases intended for high-stress mechanical environments.

Nanomaterials have emerged as highly effective performance enhancers in lubricant formulations due to their exceptional surface area, mechanical strength, and ability to form protective tribo-films. Among these, graphene and its derivatives have received considerable attention as friction modifiers and antiwear agents due to their lamellar structure, high load-bearing capacity, and outstanding thermal conductivity [11,12]. Graphene nanoplatelets can form a thin, continuous film on sliding surfaces, reducing metal-to-metal contact and stabilizing temperature distribution within the grease matrix. Furthermore, graphene, when appropriately functionalized, exhibits strong adsorption and excellent dispersion stability in oil systems, thereby reinforcing the internal soap or thickener network that governs grease consistency and rheological performance [13].

In parallel, natural organic compounds such as curcumin have been explored as multifunctional bio-additives due to their antioxidant, corrosion-inhibiting, and polar characteristics. Curcumin, a polyphenolic compound derived from Curcuma longa, contains active functional groups such as β-diketone and phenolic hydroxyl groups that can chelate metal ions, scavenge free radicals, and interact with both polar and nonpolar phases in lubricant systems [14]. When combined with graphene, curcumin can enhance the dispersion and interfacial compatibility of the nanostructured additive within the grease matrix, thereby improving oxidation resistance and film stability. This hybridization approach, integrating an inorganic nanomaterial (graphene) with an organic molecule (curcumin), is proposed to induce synergistic effects that may simultaneously improve structural cohesion and tribological efficiency.

Recent research on nanostructured lubricants highlights the importance of additive–matrix interactions in determining grease performance. For instance, Ong et al. (2020) demonstrated that graphene oxide incorporation improved the strength of the lubricating film and reduced friction coefficients by more than 40% in bio-based systems [15]. Similarly, Cacaci et al. (2023) reported that hybrid nanofluids containing graphene and organic modifiers exhibited superior antiwear performance due to enhanced boundary-layer film formation [16]. Despite these advances, the application of hybrid graphene–curcumin systems in grease formulation remains limited. Existing studies primarily focus on mono-additive approaches, while the combined structural and antioxidative roles of graphene and curcumin have not been systematically evaluated, particularly in waste cooking oil (WCO)-based matrices [17,18]. Moreover, most prior work emphasizes rheological or tribological results without correlating them with chemical bonding characteristics or internal structural stability [14].

This study addresses the gap by examining graphene–curcumin hybrid additives in a waste cooking oil (WCO)-based biogrease, explicitly positioning the proposed system relative to previously reported graphene greases modified with other organic additives and to lubricant systems employing curcumin as a single functional component. Structural interactions among the base oil, fumed silica thickener, and additives were analyzed using FTIR spectroscopy, while cone penetration, dropping point, copper corrosion, and tribological tests were used to link chemical structure to consistency, thermal stability, corrosion resistance, and friction–wear behavior. The novelty of this work lies in the dual-function hybrid strategy, in which graphene provides mechanical reinforcement and load-carrying capacity, while curcumin simultaneously acts as an organic dispersion promoter and an antioxidative stabilizer. Recognizing typical limitations of such hybrids, including graphene agglomeration and the potential thermal sensitivity of curcumin during grease processing, these challenges were mitigated through controlled processing temperatures, sequential additive incorporation, and confinement within a fumed-silica-supported grease network. Consequently, curcumin promotes homogeneous graphene dispersion via π–π stacking, hydrogen bonding, and polar interactions, while retaining antioxidative activity under the applied processing conditions.

## 2. METHODOLOGY

### 2.1 Materials

Waste cooking oil (WCO) collected from the Universitas Negeri Surabaya canteen was filtered using Whatman No. 1 paper and heated at 105 °C for 30 min to remove residual moisture. Physicochemical analysis conducted by PT Petrolab Service showed a density of 907.3 kg $m^{-3}$ at 15 °C, kinematic viscosity of 8.458 cSt (100 °C), free fatty acids of 0.84%, and water content of 3.3%. Fumed silica (primary particle size <50 nm, BET surface area ≈200 $m^2$ $g^{-1}$) was used as the inorganic thickener. Graphene nanoplatelets with >99% purity, lateral size of 3–7 µm, thickness <10 nm, and no surface functionalization were used as received. Curcumin (≥98% purity) was employed as a natural organic additive and antioxidant.

### 2.2 Biogrease formulation and homogenization

The formulation design employed symmetric graphene-to-curcumin loadings (1:1 mass ratio, 1–4 wt% each relative to base oil), as shown in Table 1. This ratio was selected based on preliminary screening experiments (single-factor variation) comparing asymmetric and symmetric loadings, which indicated that equal graphene–curcumin content provided the most stable dispersion and reproducible tribological response while minimizing graphene agglomeration at higher loadings. A full factorial design of experiments was not conducted; however, the selected ratios represent an optimized compromise between dispersion stability and additive efficiency.

**Table 1.** Composition of WCO-based biogrease.

| Code | Composition [wt%] | | | |
|---|---|---|---|---|
| | WCO | Fumed Silica | Graphene | Curcumin |
| G0 | 87 | 13 | 0 | 0 |
| G1 | 85 | 13 | 1 | 1 |
| G2 | 83 | 13 | 2 | 2 |
| G3 | 81 | 13 | 3 | 3 |
| G4 | 79 | 13 | 4 | 4 |

Fumed silica was first dispersed into WCO (Table 1) by heating the mixture to 120 °C and mechanically stirring at 500 rpm (corresponding to an estimated shear rate of ~300–400 $s^{-1}$) for 60 min to form a stable three-dimensional gel network. Particle size analysis confirmed uniform silica dispersion with minimal agglomeration. Graphene nanoplatelets were then gradually added under continuous stirring at 500 rpm for 30 min at 120 °C to promote wetting and deagglomeration within the silica–WCO matrix. Subsequently, curcumin was introduced stepwise and homogenized for an additional 30 min at the same temperature and stirring speed to ensure complete dissolution and uniform distribution.

The sequential addition strategy and controlled processing temperature were specifically chosen to mitigate graphene agglomeration and to preserve curcumin's antioxidative functionality. A schematic representation of the graphene–curcumin WCO biogrease preparation process is shown in Fig. 1. After homogenization, all grease samples were conditioned at room temperature (25 ± 2 °C) for 48 h prior to characterization.

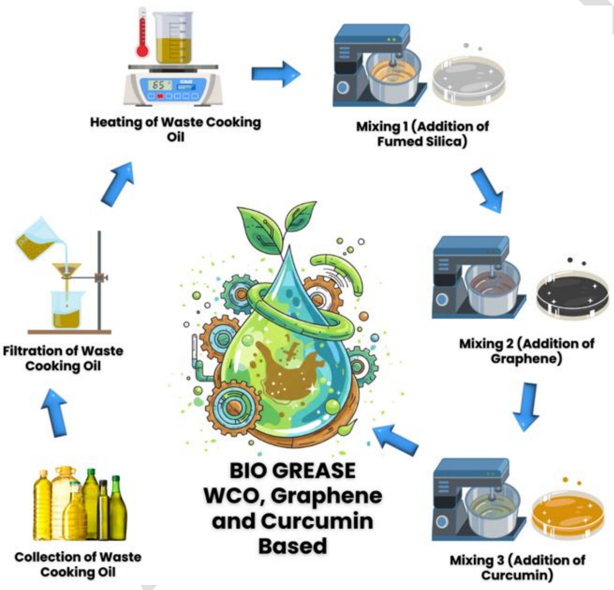


**Fig. 1.** Schematic of graphene–curcumin WCO biogrease preparation.

## 2.3 Fourier transform infrared spectroscopy (FTIR)

In this study, Fourier Transform Infrared (FTIR) spectroscopy was used to analyze functional groups and chemical interactions in the formulated greases comprising base oil, thickener, and additives. The FTIR spectra were acquired using a UATR Spectrum Two FTIR spectrometer (PerkinElmer, USA), operating over the wavenumber range of 4000–450 $cm^{-1}$ with a spectral resolution of 4 $cm^{-1}$. The spectrum of each sample was averaged over 32 scans to ensure the reliability and reproducibility of the results, as detailed in the literature on FTIR methodologies [19].

For the grease samples, analysis was performed directly in the universal attenuated total reflectance (UATR) mode, eliminating the need for solvent-based preparation. This approach is critical for preserving the structural integrity of the samples, which harsh solvents or preparation techniques can otherwise compromise. The effectiveness of FTIR in preserving sample characteristics is supported by previous studies showing that UATR techniques yield reliable data while minimizing sample alteration [19,20].

The spectral analysis focused on identifying characteristic absorption bands. Notably, signals corresponding to C–H stretching indicative of triglycerides, Si–O–Si vibrations characteristic of a fumed silica network, as well as functional groups related to graphene and curcumin, were meticulously evaluated. The presence of these functional groups supports the assertion that chemical bonding and molecular interactions result from the incorporation of the graphene–curcumin hybrid additive into the waste cooking oil (WCO)-based matrix. The interactions and potential hydrogen-bonding between curcumin and other components, as reported by other studies, were also taken into consideration [21,22].

## 2.4 Cone penetration test

The measurement of grease consistency in accordance with ASTM D217-21a requires a systematic methodology to ensure accurate and reliable results. This methodological approach adheres to the procedural guidelines established by the National Lubricating Grease Institute (NLGI) for determining the mechanical strength and workability of lubricants suitable for various industrial applications.

In the conditioning phase, each grease sample is maintained at 25 ± 0.1 °C, followed by working the grease with 60 double strokes using a grease worker, operating at 60 ± 10 strokes per minute [23]. Such a controlled mechanical process ensures uniform distribution of the grease's constituents, promoting a consistent texture for testing. The double-stroke test effectively assesses the behavior of grease under shear, simulating conditions it would encounter in practical applications [24]. The next step involves measuring penetration with a cone penetrometer to quantify depth in tenths of a millimeter (0.1 mm). This approach, which is vital for classifying the grease's NLGI grade, relies heavily on a standardized cone penetration test (CPT), often regarded as a direct measure of grease hardness [25]. The NLGI grading system categorizes greases, with lower grades indicating a more fluid consistency and higher grades indicating a firmer, more stable structure [25,26]. The results from this process reflect the grease's suitability for functional excellence in specific applications, such as bearing lubrication, where it must balance fluidity with mechanical resilience [24].

### 2.5 Dropping point test

The methodology for determining the dropping point of grease samples is essential for assessing their thermal stability and thickener integrity. The procedure was conducted in accordance with ASTM D2265-20 using a dropping-point apparatus. A measured amount of grease is packed into a standardized cup, and a polished thermometer is positioned above the sample. The temperature is then increased at a controlled rate of 1-2 °C per minute until the first drop of melted grease forms and falls through the orifice, with the observed temperature recorded as the dropping point [27].

The dropping point serves as an indirect measure of the grease's thermal stability and thickener integrity. Higher dropping-point values indicate better thermal performance and the ability to withstand elevated temperatures without transitioning to the liquid state [28]. This temperature-dependent behavior is essential for applications where lubricants are exposed to varying thermal conditions, as it influences the selection of suitable greases for specific operating environments [29]. Accurate dropping-point measurements also enable comparative analysis of various grease formulations, ultimately facilitating improved material design and performance predictions [30].

### 2.6 Corrosion test

Copper corrosion resistance was evaluated according to ASTM D4048, using polished copper strips (99.9% purity) as test specimens. Each copper strip was immersed in approximately 20 mL of the test grease within a sealed glass tube and maintained at 100 °C for 24 h inside a thermostatic bath. After exposure, the strips were removed, gently wiped clean, and visually compared with the ASTM Copper Corrosion Standard Chart to assign a classification rating (1a-4c). Lower ratings correspond to superior corrosion protection and indicate that the grease additives, particularly the antioxidant and metal-chelating functionalities of curcumin, inhibit the formation of copper oxides or sulfides.

### 2.7 Tribological test

The tribological performance of the formulated greases was evaluated using a Pin-on-Disk Tribometer (ASTM G99). Tests were conducted under ambient laboratory conditions (25 ± 2 °C, RH ≈ 50%). A hardened steel pin (6 mm diameter, Ra < 0.1 µm) was subjected to a polished steel disk (Ra < 0.05 µm). The normal load was maintained at 10 N, and the sliding speed was set to 0.1 m $s^{-1}$, resulting in a linear velocity of approximately 0.0628 m $s^{-1}$. Each test was conducted for a total sliding time of 1800 s. Before testing, a thin, uniform layer of grease was applied to the disk surface to ensure consistent film thickness. In situ sensors integrated into the tribometer data acquisition system continuously recorded the coefficient of friction (COF) and linear wear displacement over time. The evolution of wear and COF over time was used to assess the film-forming stability, antiwear capability, and lubricating efficiency of each graphene–curcumin formulation.

## 3. RESULTS AND DISCUSSION

### 3.1 FTIR analysis

Figure 2 presents the FTIR spectra of the base wco grease (G0) and the greases containing fumed silica with different combinations of graphene and curcumin (G1–G4). Fourier Transform Infrared (FTIR) spectroscopy analysis of waste cooking oil (WCO), and formulated greases indicates that the ester structure of the triglycerides in WCO is preserved during grease preparation and additive incorporation.

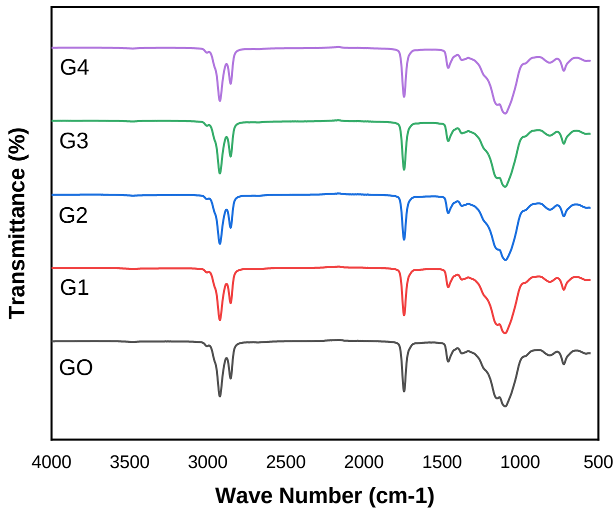


**Fig. 2.** FTIR spectra of the WCO-based grease.

Specifically, the strong C–H stretching bands associated with methylene and methyl groups are consistently observed in the 2920–2850 $cm^{-1}$ region, along with an intense ester carbonyl band at around 1740 $cm^{-1}$. This aligns with the findings of Chowdhury et al. (2014) [31], who have identified similar bands indicative of WCO-derived esters and triglyceride systems. The presence of additional bands at approximately 1460 $cm^{-1}$ and 1377 $cm^{-1}$ due to $CH_2/CH_3$ bending, as well as C–O stretching in the 1230–1100 $cm^{-1}$ range, further corroborates the characteristic signatures of WCO-based biolubricants. The absence of strong absorption bands in the carbonyl region (1700–1726 $cm^{-1}$) suggests minimal thermo-oxidative degradation, affirming that the synthesized greases are stable [32].

Moreover, the incorporation of fumed silica into the greases yields additional features within the silicate region of the FTIR spectrum while maintaining the lipid-related peaks observed in the neat WCO. Notably, a Si–O/Si–O–Si stretching band appeared within the 1080–1000 $cm^{-1}$ range, supported by Si–O and Si–H related bands near 960 and 800 $cm^{-1}$, corresponding to the fumed silica thickener network [33]. These findings are consistent with previous studies on WCO-based fumed-silica greases, which confirmed the successful formation of the silica framework without altering the base oil's fundamental chemistry [34].

The greases containing graphene and curcumin show minimal deviation from the FTIR profile of fumed-silica greases, indicating that these additives are likely physically dispersed rather than chemically altered within the WCO/fumed-silica matrix. The subtle changes observed in the unsaturated C=C stretch around 1600 $cm^{-1}$ and in the O–H stretching near 3400 $cm^{-1}$ suggest that the additives did not undergo transesterification or significant covalent interactions with the triglycerides [35]. This behavior further aligns with the characteristics reported in prior studies of WCO-based grease systems, in which FTIR spectra predominantly reflect the thickener and base ester functional groups, highlighting the compatibility of the additives while preserving essential grease properties [36].

Overall, the FTIR results confirm that the formulated greases retain the advantageous chemical structure of WCO, effectively incorporate the silica thickener, and accommodate graphene and curcumin additives without inducing adverse chemical changes. To provide a semi-quantitative assessment of these interactions, peak areas were integrated for the ester carbonyl band (~1740 $cm^{-1}$) and the Si–O–Si stretching region (1080–1000 $cm^{-1}$). The integrated ester peak area showed no significant reduction across G0–G4, confirming preservation of the triglyceride backbone and minimal thermo-oxidative degradation. In contrast, a systematic increase in the relative Si–O–Si peak area was observed with increasing additive content, indicating reinforcement of the silica network and enhanced interfacial interactions within the grease matrix. Minor variations in the O–H stretching region (~3400 $cm^{-1}$) further suggest hydrogen bonding contributions from curcumin without the formation of new covalent bonds. These quantitative FTIR observations support the proposed physical interaction mechanism and provide a robust chemical basis for the improved tribological performance discussed subsequently.

### 3.2 Cone penetration analysis

Figure 3 shows the cone-penetration values of the WCO-based greases at 25 °C, determined in accordance with ASTM D217. The results reveal a trend of decreasing penetration from 378 (0.1 mm) for the softest formulation (NLGI 0) to 292 (0.1 mm) for the stiffest formulation (NLGI 2). This reduction emphasizes increased consistency, transitioning from a semi-fluid to a more structured material suited for bearing lubrication [37,38]. Such behavior aligns with established principles of the NLGI classification, wherein lower penetration numbers are associated with more complex greases [39].

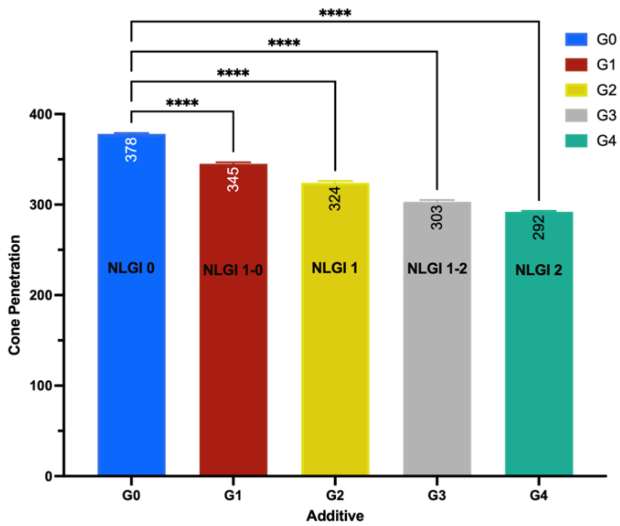


**Fig. 3.** Cone penetration of the WCO-based grease.

This decrease in cone penetration primarily correlates with an increase in effective thickener content, particularly fumed silica, along with a corresponding reduction in the free base-oil fraction throughout the series. As the fumed silica content increases, a denser three-dimensional silica network forms, effectively immobilizing a greater volume of WCO and elevating the apparent yield stress of the grease [32]. This observation is consistent with findings from previous studies on greases, in which higher silica concentrations and reduced oil content contributed to the transition from softer (NLGI 0–1) to harder grades (NLGI 2–3) [40,41].

Moreover, studies of lubricating greases have shown that increasing thickener concentration strengthens the fibrous/particulate network, thereby reducing penetration [8]. The contributions of graphene and curcumin additives further enhance this stiffening effect. Graphene nanosheets serve as rigid, high-aspect-ratio fillers that bridge adjacent silica aggregates and limit the mobility of triglyceride chains, thereby reinforcing the grease's elastic skeleton [38,42]. Research supports this notion, indicating that incorporating graphene can reduce penetration and shift the NLGI grade toward more complex classifications while improving load-carrying and anti-wear performance [43].

Curcumin, featuring multiple polar and aromatic groups, interacts with silica surfaces and ester functionalities through hydrogen bonding and π–π interactions, introducing additional cross-links into the matrix. This structural reinforcement is comparable to that observed in other polar organic additives used to strengthen bio-based greases [44]. Consequently, the cone-penetration results indicate a trend toward increased hardness, signifying a progressively dense, interconnected microstructure that, as discussed subsequently, underpins mechanical stability and tribological performance.

### 3.3 Dropping point analysis

The dropping point of the WCO-based greases was evaluated in accordance with ASTM D2265. As summarised in Table 2, all formulations (G0–G4) showed no detectable drop up to the upper limit of the apparatus (304 °C) and are therefore reported as “>304 °C”. These results indicate non-melting behaviour of these greases within the specified temperature range, suggesting a robust thickener-oil network that maintains structural integrity well beyond typical operating temperatures—thereby ensuring adequate lubrication in industrial applications where drop-out points need to be significantly higher than the maximum service temperature [45].

**Table 2.** Dropping point results of WCO-based greases.

| No. | Code | Method | Unit | Dropping point |
|---|---|---|---|---|
| 1 | G0 | ASTM D2265-20 | °C | >304 |
| 2 | G1 | ASTM D2265-20 | °C | >304 |
| 3 | G2 | ASTM D2265-20 | °C | >304 |
| 4 | G3 | ASTM D2265-20 | °C | >304 |
| 5 | G4 | ASTM D2265-20 | °C | >304 |

The elevated drop points, particularly when using a fumed silica thickener, are critical to the thermal stability of the formulated greases. Fumed silica creates a three-dimensional inorganic network that does not melt in the conventional sense; rather, it facilitates the gradual volatilization of the base oil at high temperatures while maintaining the grease's overall structure. Studies have shown that greases formulated with fumed silica can exhibit dropping points above 350 °C, suggesting these formulations can withstand substantially higher temperatures, significantly surpassing the typical ranges reported for other greases using lithium or mixed soaps, which generally report dropping points around 87 – 150 °C [36].

Furthermore, the consistency of the results (all formulations reported as ">304 °C") implies that the incorporation of various additives such as graphene and curcumin does not substantially affect the performance of the silica network. Observations suggest that the dropping point of grease formulations is predominantly determined by the thickener type and concentration, with most additives contributing little to the grease's overall thermal class. For example, Padgurskas et al. (2015) reported that functionalized graphene additives could modestly affect the dropping point of lithium greases, suggesting that the addition of these nanomaterials does not compromise the thermal stability imparted predominantly by the thickening agent [33].

### 3.4 Corrosion behaviour

The evaluation of copper strip corrosion is essential for understanding the corrosion resistance properties of lubricating greases, particularly those formulated with waste cooking oil (WCO). As shown in Fig. 4, the corrosion testing performed in accordance with ASTM D4048 indicated that the freshly polished copper strip retained its metallic sheen. In contrast, the strip exposed to the base grease G0 developed a dark-orange film indicative of slight tarnish, classified as grade 1 per the ASTM copper strip scale [43]. This observation aligns with studies on WCO-based greases, in which both treated waste cooking oil (TWCO) and untreated WCO exhibited minimal corrosive behavior toward copper, generally falling within corrosion classes 1a to 1b [46].

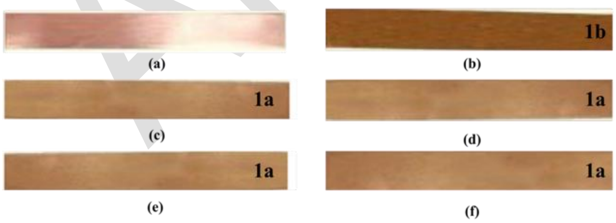


**Fig. 4.** Copper strip corrosion results of WCO-based grease.

Supporting WCO's non-corrosive nature as a base stock, the incorporation of fumed silica, a common thickener, did not significantly alter the corrosion classification. Hairunnaja et al. (2025) reported that greases formulated with fumed silica achieved consistent class 1 tarnishing (1a–1b), reinforcing the inertness of fumed silica towards copper corrosion [46]. This phenomenon is likely due to the absence of aggressive contaminants, such as active sulfur or chlorine, in the formulations, consistent with corrosion mechanisms identified in related studies that emphasize the corrosive potential of certain additives [34].

The modifications resulting from greases G1–G4, which involved the addition of graphene and curcumin, correlated with a similar or enhanced corrosion rating of 1a. These additives are recognized for their environmentally friendly corrosion-inhibition properties, primarily because they form protective layers on metal surfaces and impede anodic and cathodic reactions. Established literature supports the use of curcumin as a natural antioxidant, thereby reinforcing its efficacy in forming protective barriers against corrosion [35].

Moreover, the role of graphene and graphene oxide in corrosion protection has been well documented. These materials are known for forming impermeable coatings that extend the diffusion pathway for corrosive agents, thereby providing enhanced protection to underlying substrates. The findings that all tested greases maintain corrosion ratings of either 1 or 1a validate that the WCO–fumed-silica–graphene–curcumin blend effectively meets the non-corrosive requirements necessary for applications in bearing and gear systems, as corroborated by multiple studies [46].

### 3.5 Tribological behaviour

The performance of greases, particularly in tribological applications, relies on their coefficient of friction (COF) and wear characteristics. Studies on greases G0–G4 indicate that the progression of COF over sliding duration is influenced by additives such as graphene and curcumin. Figure 5 shows the evolution of the coefficient of friction (COF) for greases G0–G4 during 2000 s of sliding. All samples exhibit a short run-in stage (0–200 s) with relatively high, noisy COF, as asperity contacts dominate before a stable film is established. The base grease G0 (WCO–FS without additive) stabilises at the highest COF, about 0.07, whereas all additive-containing greases reach lower plateaus: ~0.06 for G1 and G3, ~0.045–0.05 for G2, and ~0.035–0.04 for G4. Thus, the optimized formulation G4 yields roughly a 40–50% reduction in steady-state friction relative to G0.

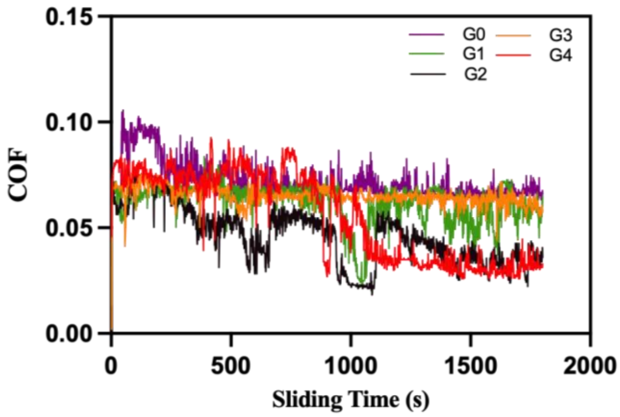


**Fig. 5.** Coefficient of friction (COF) for WCO-based grease.

All samples exhibited an initial run-in period characterized by higher, unstable COF values, a phenomenon attributed to dominant asperity contacts before the formation of a stable lubricating film [47]. The base grease G0 displayed the highest steady-state COF (approximately 0.07). In contrast, the greases with additives achieved notably lower plateaus—G4 reached a COF reduction of about 40–50% relative to G0, supporting the efficacy of graphene-curcumin formulations [48].

Tribological theory suggests that lower COF values correlate with effective lubricating films that mitigate wear [47,48]. This is evidenced by studies demonstrating that graphene additives significantly reduce COF by facilitating tribochemical reactions and forming physical films on sliding surfaces [49,50]. In the present study, this mechanism is supported by FTIR observations, where subtle band shifts and changes in relative peak intensities in the ester (C=O), O–H, and Si–O–Si regions indicate physical adsorption, hydrogen bonding, and interfacial interactions among graphene, curcumin, and the silica–WCO matrix. These spectral features provide direct chemical evidence for the formation of a hybrid tribofilm rather than simple mechanical lubrication. Graphene adsorption onto steel surfaces contributes to a load-bearing lamellar film, while curcumin, through its aromatic and polar functional groups, enhances film adhesion and stability, collectively improving load-carrying capacity.

The coefficient of friction trends for the WCO-based greases are presented in Fig. 5, showing a pronounced reduction in COF for formulations containing graphene curcumin additives compared to the base grease G0. Wear analysis further supports these trends, as shown in Fig. 6. The base grease G0 exhibited a high wear depth of up to ~16 μm, whereas all additive-containing greases showed substantially reduced wear. The optimized formulation (G4) stabilized at approximately 3–4 μm, indicating the formation of an effective and durable protective boundary layer over time [48,51].

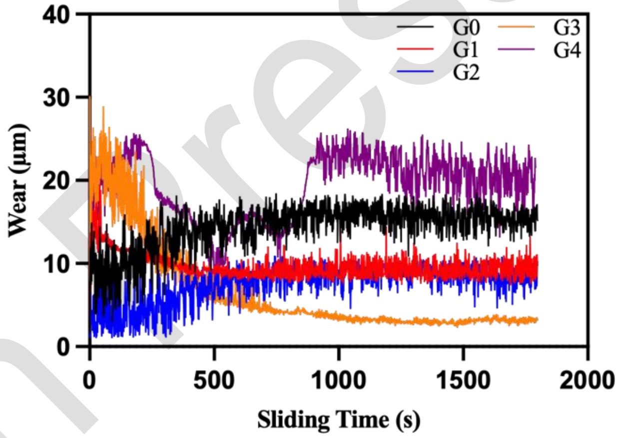


**Fig. 6.** Wear depth for WCO-based grease.

However, the improvement does not increase linearly with increasing additive content. At higher loadings, tribological performance tends to plateau or show marginal gains, which can be attributed to several competing mechanisms. First, additive saturation may occur at the contact interface, where excess graphene and curcumin no longer contribute to further film strengthening. Second, higher graphene content increases the likelihood of nanosheet agglomeration, reducing the effective surface area available for load sharing and disrupting uniform tribofilm coverage. Third, increasing solid additive concentration raises grease consistency and apparent viscosity, which may hinder lubricant replenishment in the sliding contact and promote third-body abrasion. Similar non-linear trends have been reported for graphene-laden greases and bio-lubricants containing polar additives [52,53]. These combined effects explain why an optimal additive loading window exists, beyond which further increases do not proportionally enhance wear resistance, despite the presence of a durable boundary film [54,55].

Moreover, the unique structure of the fumed-silica (FS) thickener in the formulation helps uniformly disperse graphene and curcumin, thereby improving the retention of the lubricating film under shear forces [48]. Various tribological studies have shown that the effectiveness of grease formulations depends primarily on maintaining a balanced thickener structure to ensure efficient film formation without risking starvation or excessive softness [48,55]. In contrast, G0 exhibits inferior performance, indicating that compromised interactions among the base oil, thickener, and additives can lead to unstable lubrication and increased wear [56].

Particle Size Analysis (PSA) was conducted to characterize the dispersion state and size distribution of the fumed silica (HDK) used as the grease thickener, as these parameters strongly influence network formation, oil entrapment, and ultimately grease consistency and tribological behavior. The PSA measurements were performed using a dynamic light scattering (DLS) particle size analyzer (BIOBASE BK-802N) with water as the dispersion medium at 20 °C, and the resulting particle size distribution is presented in Fig. 7. The results show a volume-based median particle diameter (D50) of 22.15 nm, with D10 and D90 values of 8.89 nm and 55.14 nm, respectively, and an average hydrodynamic diameter (Xav) of 49.80 nm. The dispersion index (PI) of 0.6604 indicates a moderately broad but stable particle size distribution, which is typical for fumed silica due to its fractal, aggregated primary particle structure.

| Test Condition | | |
|---|---|---|
| Test temperature: 20°C | Dispersion medium: Water | Medium viscosity: 1.002E-03Pa·s |
| Medium refractive index: 1.333 | Angle: 90.0Deg | Delay Unit Time: 20us |
| LightColor: Red | | |
| **Test Information** | | |
| Test company: LIHTR | | |
| Tester: LIHTR | Test time: 7/4/2025 3:48:59 PM | |
| **Test Result:** | | |
| Dav Diameter: Xav=49.80 nm | Dispersion Index: PI=0.6604 | Photon Count: 3 |
| D10= 8.89 nm | D50= 22.15 nm | D90= 55.14 nm |

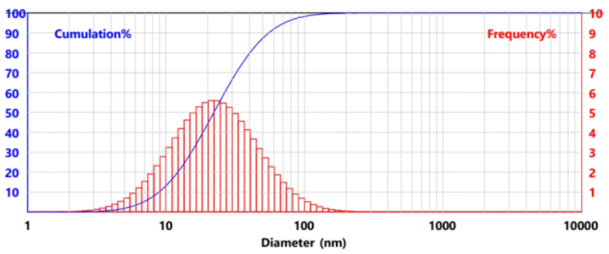


**Fig. 7.** Particle size analysis (PSA).

The narrow nanoscale distribution confirms that the silica exists predominantly as nanoaggregates rather than large agglomerates, which is favorable for forming a continuous three-dimensional thickener network within the WCO matrix. Such a network enhances oil immobilization and mechanical stability, supporting the observed NLGI 0–2 consistency range and high dropping points. The presence of a limited fraction of larger aggregates (D90 ≈ 55 nm) is attributed to reversible physical aggregation in aqueous dispersion and does not indicate poor material quality. Instead, this hierarchical particle structure is advantageous in grease systems, as it promotes strong thickener–oil interactions while maintaining sufficient gel network flexibility under shear. Overall, the PSA results provide quantitative support for the effective role of fumed silica as a nanostructured thickener, complementing the tribological and rheological performance observed in the formulated biogreases

### 3.6 Lubrication mechanism of graphene-curcumin biogrease

The performance of greases, particularly in tribological applications, is heavily influenced by their coefficient of friction (COF). The schematic in Fig. 8 illustrates the lubrication mechanism inferred from the FTIR, rheological, and tribological results. In bulk grease, WCO triglycerides are immobilized within the fumed silica network. Curcumin, on the other hand, interacts with both the oil and graphene through polar interactions and π–π stacking, contributing to the homogeneous dispersion of graphene.

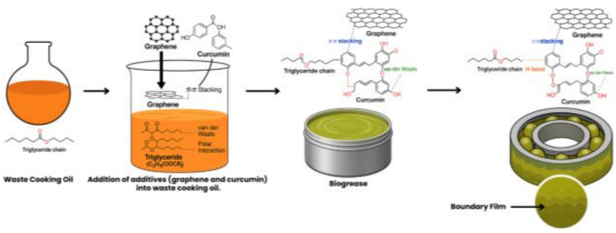


**Fig. 8.** Schematic representation of the lubrication mechanism of graphene–curcumin WCO biogrease in the sliding contact.

During sliding, these species accumulate at the contact point, forming a hybrid boundary film on the steel surface. This film comprises graphene sheets, which provide a lamellar, low-shear solid lubricant with high load-carrying capacity.

Curcumin, through its phenolic and aromatic groups, adsorbs onto the film surface, acting as a green film-former and a corrosion inhibitor. This proposed synergistic effect is suggested to account for the lower, more stable coefficient of friction, reduced wear, and non-corrosive behavior observed in the graphene–curcumin formulations compared with the base WCO–silica grease. This finding aligns with earlier reports on graphene-based nanoadditives and curcumin-containing green lubricants.

## 4. CONCLUSION

This study demonstrates the successful formulation of a waste cooking oil (WCO)-based biogrease reinforced with a graphene–curcumin hybrid additive and a fumed silica thickener, offering a sustainable alternative to conventional mineral-oil-based greases. FTIR analysis confirmed the preservation of the triglyceride ester structure of WCO. Additionally, characteristic Si–O–Si and additive-related bands indicated the effective incorporation of the silica network and hybrid additives without any undesirable chemical degradation, supporting predominantly physical and interfacial interactions within the grease matrix. The formulated biogreases exhibited a practically relevant consistency range (NLGI grades 0–2) and high thermal stability, with all samples showing non-melting behaviour and dropping points exceeding 304 °C, while copper strip corrosion ratings of 1–1a confirmed non-corrosive performance toward copper surfaces. Tribological testing further demonstrated a reduction in steady-state coefficient of friction of approximately 40–50% and a decrease in wear depth from ~16 μm to ~3–4 μm relative to the base WCO–silica grease. These improvements are attributed to a proposed synergistic lubrication mechanism involving a mechanically stable silica–WCO gel matrix, lamellar graphene sheets, and curcumin-derived boundary films, which are hypothesized to collectively enhance the stability of the lubricating film under mechanical and thermal stress. Analysis of formulation trends indicated an optimal graphene–curcumin concentration window, beyond which additive saturation, agglomeration, and increased grease consistency may limit further performance gains, highlighting the need for future statistical analysis, advanced surface characterization, and standardized biodegradability assessments.

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