



Conversion of used engine oils from agricultural machinery into colloidal graphite greases enhanced lubricating greases

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ABSTRACT

The article examines the development of a stable lubricant enhanced with colloidal graphite, utilizing recycled 15W40 diesel engine oil thickened with calcium soap. The study reveals that increasing the graphite concentration results in a gradual rise in water content, while the amount of thickener remains unchanged. Furthermore, higher graphite levels lead to a reduction in penetration values, decreasing from 440 units to 310 units. This suggests an improvement in grease consistency and stiffness. The dropping point also shows notable changes, increasing from 79°C to 88°C at a graphite concentration of 150 ppm, before declining to 81°C with further graphite additions. Regarding anti-wear performance, the smallest wear scar diameter is observed at the same 150 ppm concentration, measuring 0.215 mm—25.1% smaller than that of the control sample.

1. Introduction

The agro-industrial sector is advancing rapidly with the adoption of modern mechanized agricultural machinery and equipment [1, 2]. Operating such machinery requires process fluids, including plastic lubricants [3], which are particularly essential for suspended trailed implements. However, the growing number of machines has led to a significant environmental issue: the accumulation of used engine oils, exacerbated by the expanding machinery fleet [4]. Recycling these used oils into plastic lubricants (PLM) has become a critical challenge [5, 6]. The recycling process is often complex, as the production of high-quality lubricants from used oils typically involves calcium soap-based thickeners [7, 8]. These lubricants are cost-effective and use safe, readily available components, including plant-based materials [9]. However, their quality can be improved with additives like anti-wear agents, extreme pressure additives, and antioxidants [10]. Recently, nanoparticles have emerged as innovative functional additives due to their unique properties [11]. Among these, colloidal graphite (CG), also known as nanographite or multilayer graphene [12], has garnered substantial interest.

Owing to the considerable importance of graphene-based materials, these systems have been extensively

investigated in diverse fields including adsorption, catalysis, and computational studies [13, 14].

While CG is often used in significant amounts (e.g., starting at 0.1% by weight) [15], its high cost makes this approach economically unfeasible for industrial applications. Research has shown that even minimal concentrations of CG, such as 0.075% by weight (750 ppm), can have structuring effects [16]. Advancements in recycling waste motor oil from agricultural machinery now focus on studying the impact of nanoplatelets on the tribological and rheological properties of lubricants at concentrations below 750 ppm [17]. The increasing reliance on heavy machinery like tractors, harvesters, and seeders in modern agriculture highlights the need for high-performance engine oils to ensure efficiency and sustainability. These oils reduce friction, wear, and heat but degrade over time, becoming contaminated with soot, metal particles, oxidized hydrocarbons, and chemical additives, resulting in used engine oil (UEO) [18–20]. The disposal of used engine oils (UEOs) poses significant environmental and economic challenges. Millions of liters of waste oil are generated globally each year, and improper disposal—such as dumping into soil or water—causes severe ecological harm, including groundwater contamination, soil degradation, and damage to aquatic ecosystems [21].

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In agricultural settings, where machinery is heavily used in remote areas, sustainable management of UEOs becomes even more critical.

These oils contain toxic substances like polycyclic aromatic hydrocarbons (PAHs), heavy metals (e.g., lead, cadmium, zinc), and sludge-forming particles, necessitating safe handling and treatment. Simultaneously, the demand for eco-friendly, high-performance lubricants—especially greases capable of operating under extreme pressure, temperature, and corrosive conditions—is increasing. Researchers have begun exploring innovative methods to recycle UEOs into value-added products, particularly colloidal greases enhanced with nanomaterials like graphene. Graphene, a single-atom-thick sheet of sp^2 -hybridized carbon atoms arranged in a honeycomb lattice, offers exceptional mechanical strength, thermal conductivity, and tribological properties [24, 25]. Its integration into recycled oil-based greases promises superior anti-wear and load-bearing capabilities while addressing environmental concerns through waste oil recycling. This study aims to develop graphene-enhanced colloidal greases from UEOs for agricultural machinery maintenance.

Key objectives include characterizing UEOs' physicochemical properties, designing purification protocols to remove contaminants, dispersing graphene effectively within purified oil, formulating greases using thickening agents, and evaluating their performance against commercial lubricants. The research aligns with sustainable development goals (SDGs) such as responsible consumption and production (SDG 12) and climate action (SDG 13), promoting circular economy principles by converting hazardous waste into high-value industrial products. Graphene-enhanced greases derived from UEOs exhibit significant tribological benefits, including reduced friction (up to 30–50%), lower wear rates, improved thermal conductivity, and extended operational temperature ranges [26]. These properties are particularly advantageous for agricultural machinery operating under extreme conditions. Beyond agriculture, such greases have applications in mining, construction, transportation, and manufacturing industries. Despite promising results, challenges remain in scaling production. These include variability in UEO feedstock composition, purification costs, high graphene production costs, and ensuring long-term dispersion stability. Addressing these issues requires interdisciplinary collaboration across material science, chemical engineering, and industrial design. In conclusion, transforming UEOs into graphene-enhanced colloidal greases represents a sustainable solution to environmental and industrial challenges. This approach minimizes waste pollution while delivering high-performance lubricants for demanding applications. Continuous research and development, coupled with

policy support and industry collaboration, could pave the way for widespread adoption of this innovative technology.

2. Materials and Methods

Regenerated 15W40 waste diesel engine oil, previously used in a New Holland T8.410 agricultural tractor, was used as the base oil. The oil regeneration was carried out via an adsorption-based purification process using proprietary technology. Colloidal graphite (CG) was synthesized through electrochemical exfoliation, chosen for its efficiency and environmental compatibility [30]. The starting material was thermally expanded graphite foil ("Graflex"). The exfoliation process employed a 0.1 N NaOH aqueous solution as the electrolyte and a current density of 0.11 A/cm². The setup included 150 ml of electrolyte in a thermostatically controlled beaker, with two graphite electrodes (9.5 mm in diameter, 15 mm apart, immersed 55 mm deep). A rectangular alternating voltage at 0.1 Hz was applied for 45 minutes. The electrolyte was continuously stirred using a paddle stirrer. The resulting suspension had a CG concentration of 2.05 g/L. Nanoplatelet morphology was analyzed using scanning electron microscopy (Carl Zeiss Merlin) and transmission electron microscopy (Hitachi HT7700). Lateral dimensions were determined via optical microscopy (OSEELANG BM-500T) and dynamic light scattering (NICOMP-380ZLS). To prepare consistent lubricants, 100 g of regenerated oil was heated to 65 °C under intensive overhead stirring. Subsequently, 80 g of unrefined sunflower oil and 50 ml of lime milk (containing 12 g Ca(OH)₂) were added. The mixture was heated to and maintained between 95–100 °C for 1.5 hours to complete saponification. After this, an additional 100 g of regenerated oil was added. The mixture was cooled to 55–60 °C under stirring and poured into a container. After reaching room temperature, samples were collected. Lime milk was prepared by dispersing CG suspension in water to achieve graphite concentrations of 15, 75, 150, and 750 ppm. The resulting PSM samples were analyzed for dropping point (ASTM D566), cone penetration, calcium thickener content (GOST 5211-85), and residual water content (via distillation). For water content analysis, 50 g of homogenized sample was mixed with 100 ml of Nefras C2 80/120, and the mixture was distilled until no further water was detected. Each test was performed in duplicate. Tribological properties were evaluated using a four-ball friction tester. The steel balls (III15, Ø10 mm) were rotated at 1480 rpm for 1 hour. Wear scar diameters were measured using a HIGH CLOUD S4 microscope, calibrated with a micrometer object (GOST 7513-75), and analyzed using TouPTek TouPView software. Friction coefficients were derived from current measurements using a Holdpeak HP-90EPC multimeter. A commercially available fatty oil (GOST 1033-79)

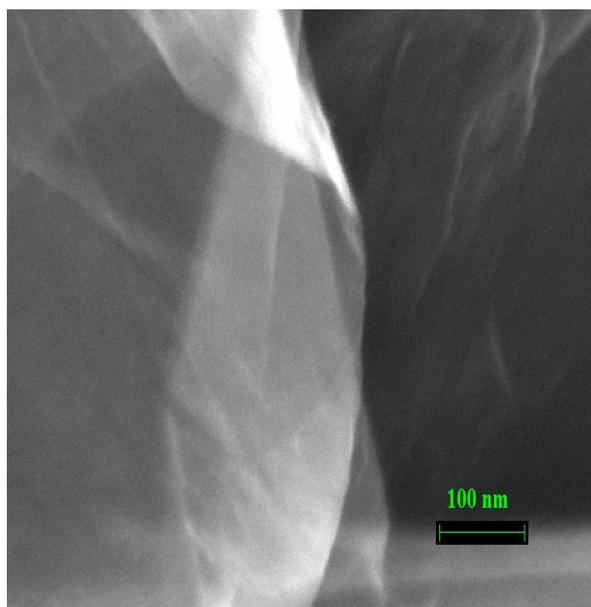
served as the control sample.

3. Results and Discussion

Scanning electron microscopy images (Figure 1a, b) revealed that the synthesized CG nanoplatelets consisted of stacks containing 15 to 25 graphene layers. The lateral dimensions, determined by both optical microscopy and dynamic light scattering (Figure 2a, b), approximated squares with side lengths of $\sim 6.3 \mu\text{m}$, with strong agreement between methods. Analysis of the PSM samples demonstrated a monotonic increase in water content with higher CG concentrations—from 0.6% to

2.3% by weight. Similarly, penetration values decreased steadily from 440 to 310 units, indicating increased consistency with CG content [27-29].

The dropping point exhibited a non-linear trend, peaking at a CG concentration of 150 ppm. Rheological measurements (Figure 3) and tribological performance tests showed that wear scar diameter (Figure 4) and friction coefficient (Figure 5) were both dependent on the axial load. The CG-enriched lubricants consistently outperformed the control sample, particularly at moderate concentrations, with significant improvements in anti-wear behavior and reduced friction [30, 31].

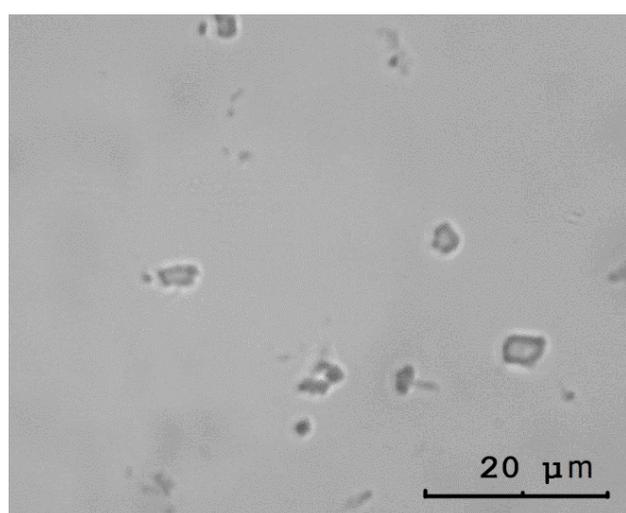


a

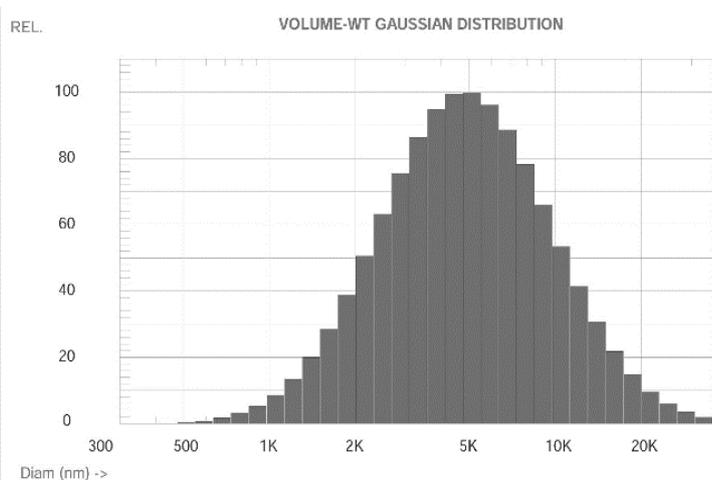


b

Fig. 1. Images of colloidal graphite obtained by methods: a) scanning electron microscopy, b) translucent electron microscopy.



a



b

Fig. 2. Diametric characteristics of graphene nanoparticles measured by techniques of: a) optical microscopy, b) dynamic light scattering.

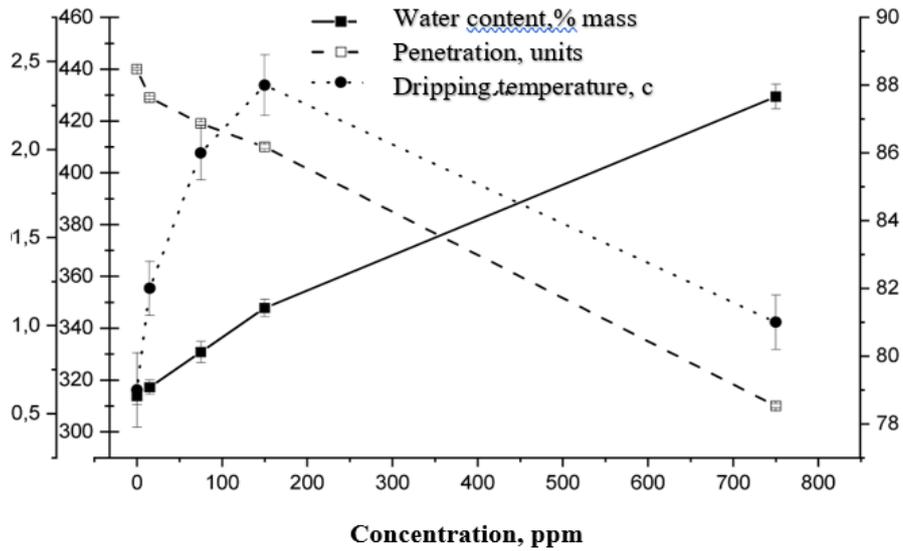


Fig. 3. Dependence of rheological characteristics of lubricants on the concentration of colloidal graphite

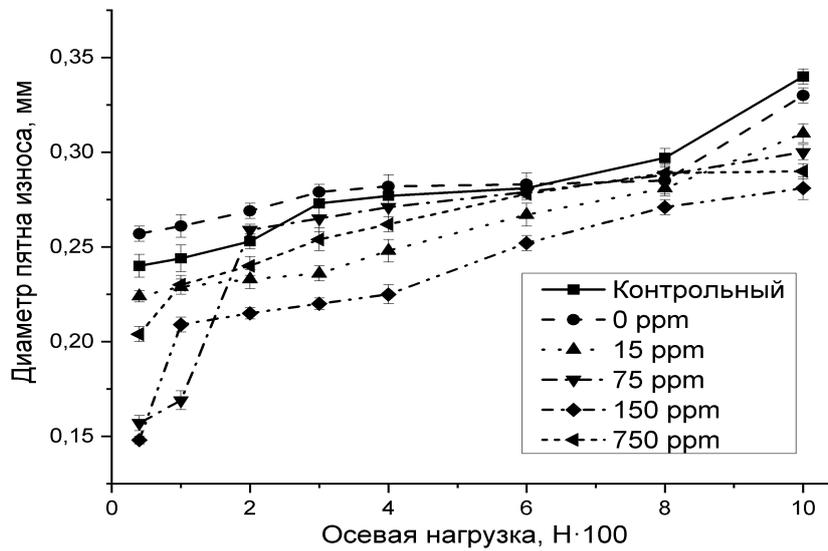


Fig. 4. Effect of KG Concentration on the Diameter of Wear Scar under Increasing Axial Load.

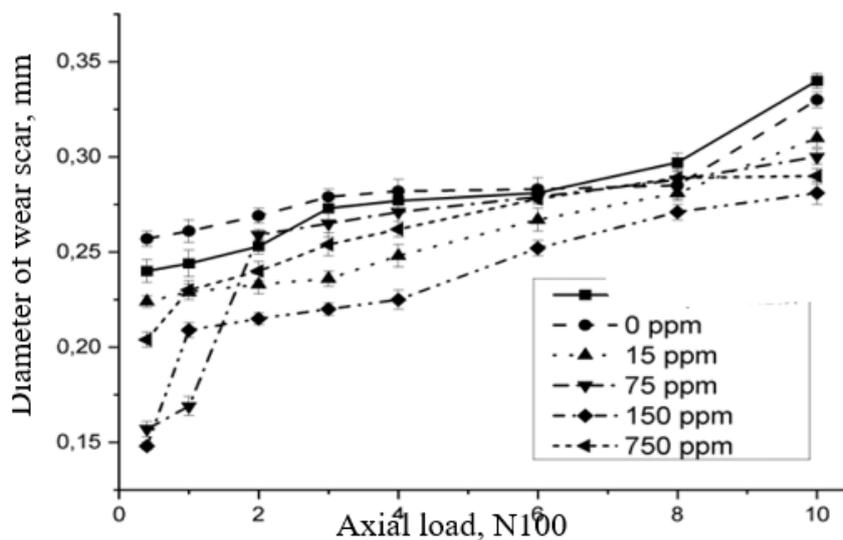


Fig. 5. Wear Performance of Lubricants with Varying KG Concentrations under Axial Load Conditions.

The dropping point, penetration, thickener fraction, and residual water content of the lubricants were measured. Additionally, the relationships between wear scar diameter and friction coefficient with axial load values were determined. These parameters provide a comprehensive understanding of the tribological and rheological characteristics of the PSM. The proportion of free water in the lubricant steadily increases from 0.6% to 2.3% by weight as the graphite concentration rises. However, the addition of KG does not influence the formation or concentration of calcium soaps, which remain consistent at $18.5 \pm 1.3\%$ by weight across samples. This phenomenon can be attributed to structural changes in calcium soap caused by the interaction between graphite plates and the filiform crystals of the thickener. Although the exact nature of this interaction remains unclear, it is hypothesized to be driven by dispersion forces. It is believed that KG nanoplatelets serve as centers that create physical bonds between individual threads formed by calcium soap. Future research aims to investigate this interaction mechanism using computational chemistry methods, particularly molecular dynamics simulations [32]. The variation in water content further supports this hypothesis, as a more branched thickener structure appears capable of retaining a greater number of water molecules. As illustrated in Figure 3, water concentration directly correlates with penetration values—the higher the water content, the lower the PSM penetration value.

The impact of KG concentration on the dropping point value suggests alterations in the thickener's structure, which exhibits a robust configuration when the carbon nanoplatelet content reaches 150 ppm. This is further corroborated by the wear scar measurements obtained during four-ball friction machine testing under an axial load of 200 N. If KG functioned solely as an additive with a functional effect [33], it would influence only the tribological properties. However, in this case, the rheological properties also underwent changes, which are primarily dictated by the thickener's structure. This observation is indirectly supported by the analysis of the trends illustrated in Figure 4.

If graphite served solely a functional role, an increase in its concentration would result in a reduction in the size of the wear scar. However, observations reveal that the base solid oil (without KG) performs worse than the standard sample. When carbon nanoplatelets are added, the wear scar size decreases, albeit not consistently, and the standard sample begins to lag behind the modified PSM. The most favorable results were achieved with a concentration of 150 ppm, except under a load of 100 N. Analysis of the trends depicted in Figure 5 suggests that at lower axial loads (up to 300 N), all samples exhibit similar behavior, but as the load increases, qualitative differences emerge. Both the control and base solidol samples display comparable trends, whereas the standard

sample has a slightly lower friction coefficient [34].

Samples containing up to 150 ppm KG show higher friction coefficients after an axial load of 800 N compared to PSM without graphite. This can be attributed to the unfavorable configuration of the thickener caused by KG's structuring effect. As anticipated, the sample with 750 ppm demonstrates the lowest friction coefficient, likely because graphite at this concentration contributes not only to thickener structure formation but also directly to the friction process. Solidol with 150 ppm KG exhibits similar trends but with higher absolute friction coefficient values. This observation may be explained by hypothesizing that at this level of colloidal graphite, an optimal concentration is achieved for structuring the thickener effectively. Beyond this concentration, further increases lead to graphite's direct involvement in the friction process, resulting in a functional effect.

4. Conclusion

Colloidal graphite produced through an electrochemical method at concentrations below 750 ppm influences the flow behavior and frictional properties of plastic materials and lubricants thickened with calcium soap. The optimal performance is observed at a colloidal graphite concentration of 150 ppm, where the smallest wear scar diameter and the highest dropping temperature are achieved. Increasing the concentration of graphite nanoplates results in higher water content and a decrease in penetration value. Furthermore, tribological analysis indicates that graphite nanoplates form a thread-like structural framework with calcium soaps, establishing physical links between individual chains.

Research findings demonstrate that introducing colloidal graphite at a concentration of 150 ppm into consistent lubricants derived from secondary motor oils containing calcium soaps enhances tribological performance. Specifically, the maximum wear scar under a 200 N axial load is reduced by 20.1%, and the friction coefficient under a 1000 N axial load decreases by 1.14 times compared to the control sample. Additionally, the increase in dropping point to 88 °C suggests expanded application potential for modified lubricants.

These studies have identified optimal conditions for producing lubricants modified with colloidal graphite, which can be recommended for industrial processes involving the recycling of used motor oil from agricultural machinery. This technology is suitable for implementation both by specialized enterprises and within agricultural organizations.

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