# Nanolubricants applied in the automotive industry

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## ABSTRACT

The growing demand for products with higher performance, lower manufacturing costs, with less impact on the environment and that at the same time are more energy efficient, has encouraged research in various areas of nanoscience and nanotechnology. In the automotive sector, in order to improve engine performance and contribute to fuel economy, nanoparticles have been studied for application in mechanical systems. However, the wide application of nanolubricants is hampered by the tendency of nanoparticles to agglomerate and precipitate.

Based on this context, the present study aimed to develop nanolubricants and evaluate the stability capacity of the formulations, as well as the contribution power of the inserted nanoparticles in significantly reducing friction and wear in moving components. The nanolubricant samples were formulated by incorporating the nanoparticles into base oil by dispersion in an ultrasonic bath. The study was conducted initially through the visual stability test, quantified by absorption spectroscopy in the visible (400-1000 nm) and tribological tests of friction coefficient and wear scar in the Four-ball equipment. After the tribological tests, the test specimens were analyzed under an optical microscope to prove and quantify the data obtained. The results indicated signs of loss of stability of the formulations in one day, with an average percentage precipitation of 35.94 at 54.76 % of nanoadditive, indicating the need for the use of surfactants capable of improving the stability of the formulations. In tribological tests the nanolubricants demonstrated high efficiency in lubricating properties, significantly reducing the coefficient of friction by 26.47 % and the wear scar by 50.97 % for the G100 formulation (100 ppm graphene).

**Keywords:** nanolubricant, graphene, stability, tribology.

## INTRODUCTION

With the development of society and the advancement of science and technology, the transportation, chemical industry, and machinery sectors are expanding, but the waste of energy caused by heat transfer and friction has seriously retarded the development of the industry. Achieving efficient heat transfer to heating components and improving the abrasion resistance of long-term moving components are essential for industrial development [1].

The development of high-performance lubricants is being driven because of the enormous cost caused by energy losses due to friction in systems with surfaces in contact and in relative motion. According to Holmberg et al. [2,3], it is estimated that 23 % of the world's total energy consumption originates from tribological contacts. In general, controlling wear appears to be more critical than friction, as it can result in catastrophic operational failures that can negatively affect productivity, and therefore cost. However, of that amount, 20 % is used to overcome friction and 3 % is used for rework of worn parts and spare equipment due to wear and wear-related failures.

One of the most effective ways to reduce friction and wear is using fluid lubricants. Many of the fluid lubricants used today rely heavily on additives containing elements that can have harmful impacts on the environment and human health, such as phosphorus and sulfur. Consequently, with pressure from government policies, new environmentally friendly additive alternatives are being developed to improve the anti-wear and friction reduction characteristics of lubricants.

The concept of nanolubricants was innovatively proposed by Choi [4], and nanolubricants exhibited enhanced heat transfer and mechanical friction reduction properties by adding nanoscale solid particles to the fluid, aerospace, shipping, automotive, air conditioning, refrigeration, electronics computing, and other fields to meet the high heat transfer and lubrication requirements of the heat exchange system, which is of great importance to improve the economics, sustainability, and miniaturization of the heat exchange system, and has broad application prospects and enormous potential economic value.

However, incorporating nanoparticles into fluids and obtaining a good dispersion is not an easy task: they tend to agglomerate, thus losing their nanometric character. Among the nanoparticles recently studied, graphene deserves to be highlighted.

Graphene consists of a flat monolayer (or single sheet) of carbon atoms tightly packed in a two-dimensional (2D) lattice. Since its discovery in 2004, it has been considered one of the materials of greatest scientific and technological interest due to its excellent physicochemical properties.

Due to its intrinsic properties, graphene has attracted great interest because of its high surface area (2630 $m^2\ g^{-1}$) and its distinctive mechanical and electrical properties, such as mechanical strength (1,060 GPa), high intrinsic mobility (200,000 $cm^2\ V^{-1}s^{-1}$), high modulus of elasticity (~1.0 TPa), optical transparency (~97.7), and excellent thermal conductivity (~5,000 $Wm^{-1}K^{-1}$) combined with gas impermeability [5,6].

The atomically smooth surface and weak Van der Waals force between graphene layers (Figure 1) facilitate sliding between layers and contribute to its self-lubricating characteristic. In addition, the 2D structure of graphene with high specific surface area also favors charge transfer [7].

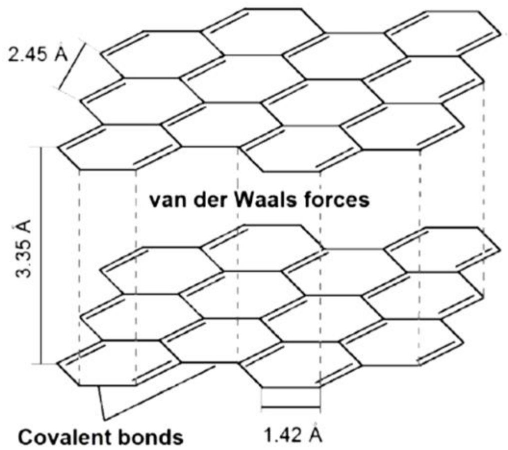


Figure 1. Graphene structure and Van der Waals interactions between graphite layers

Its potential for industrial and commercial applications is impressive, it is estimated that by 2024, the graphene industry will grow its market value from $32 million (year 2016) to over $390 million [2]. In recent years, based on the excellent lubricating properties of graphene, using it as an additive in a base fluid can provide synergistic enhancement of wear scar and friction reduction. This paper explores the comprehensive performance of nanolubricants for wear scar and friction reduction.

## EXPERIMENTAL SECTION

MATERIAL CHARACTERIZATION – Commercially available nanoparticles were purchased from University of Caxias do Sul (UCS). The graphene was characterized by Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM-EDS) (Japan Electron Optics Ltd, Model JSM 7200F), as shown in Figure 2 and the mineral base oil ISO VG 32 was characterized as to physical properties: flash point (Pensalab, Model Optiflash TAG), density (Anton Paar Digital Densimeter, Model DMA-4500) and kinematic viscosity and viscosity index (Multi-Gamma viscometer, Model HVM 472), the results are shown in Table 2.

METHODS IN NANOFLUID PRODUCTION – The preparation of nanolubricants, the graphene nanoadditive was mixed directly into the base oil and homogenized using a sonifier bath (Ultra cleaner, Model 14000A) for 2 h at a temperature of 60 °C. The following formulations were prepared: G0, G100, G500 and G1000 containing 0, 100, 500 and 1,000 ppm graphene respectively.

VISUAL STABILITY EVALUATION OF NANOLUBRICANTS – After complete dispersion of the graphene in the base oil, a 20 ml fraction was added to transparent glass vials, which were kept in static regime, at room temperature, for visual monitoring of the dispersion's stability. The follow-up was recorded by means of photographic images for a period of 0, 1 and 7 days, with time zero referring to the period immediately after dispersion.

QUANTITATIVE ANALYSIS OF NANOLUBRICANT STABILITY – The stability of the formulations was quantified by the UV-Visible Spectroscopy technique, for absorbance analysis, a Mettler Toledo spectrophotometer, Model UV5, was used. The analysis was performed in the 400 to 1000 nm range and the formulations, in static regime, were evaluated in the periods of 0.1 and 7 days. For this study, the absorbance values were considered in the wavelength of 600 nm, since this is an average value of the visible range and more used for this type of analysis [8]. The cuvette used for storage of the nanolubricant was a glass cuvette with an optical path of 10 mm and a capacity of 2 ml.

TRIBOLOGICAL STUDY – Tribological testing was performed to determine the coefficient of friction and wear scar diameter between the contact surfaces using a Four-ball tribometer (Falex Corporation, Model Four-ball Wear Test), as shown in Figure 2. The test procedure followed the guidelines of ASTM D4172-21 [9].

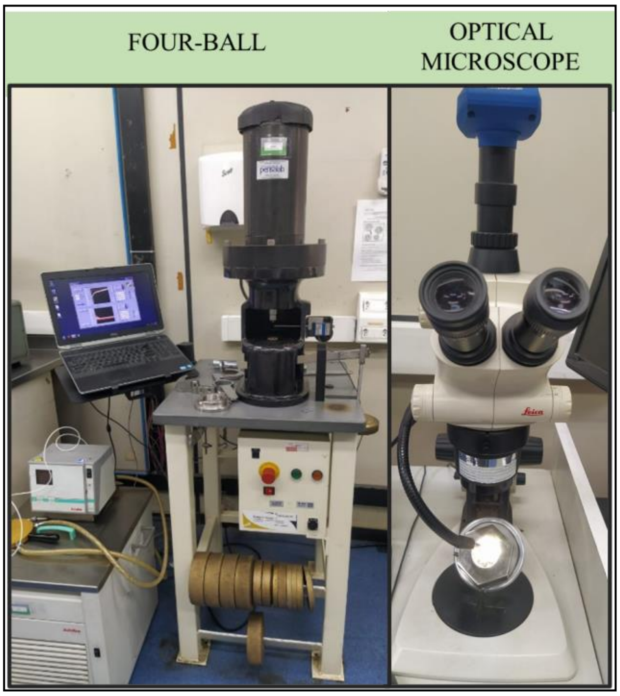


Figure 2. Four-ball wear equipment and optical microscope.

The Four-ball equipment provides data such as coefficient of friction, wear scar diameter, and contact temperature that are available in a computer coupled to the system. The test conditions of ASTM D4172-21 are described in Table 1.

Table 1. Tribological test parameters of the Four-ball.

| ASTM | ASTM TEST METHOD | Test Conditions | | | |
|---|---|---|---|---|---|
| | | Speed (rpm) | Load (Kg) | Time (min) | Temperature (°C) |
| D4172-21 | Typical preventive wear of lubricating fluid | 1200±60 | 15±0,2 | 60±1 | 75±2 |

After the test, the balls were cleaned with heptane and taken to the optical microscope (Leica Microsystems) at 100x magnification for evaluation of the wear mark generated by the relative movement with the balls. The wear dimensions in the x and y axes are measured and averaged, resulting in the wear scar diameter (WSD) value.

## RESULTS

MATERIAL CHARACTERIZATION – Scanning electron microscope (SEM) analysis was performed to evaluate the microstructure of the graphene surface (roughness, morphology, and porosity). The Figure 3 presents the micrograph, using SE secondary electrons (500-10,000x), of the sample surface. As a result, the nanoadditive presents morphology of agglomerated sheets (nanoplatelets) with planar dimension varying between 1-10 µm. With respect to thickness, they presented an average of 10-50 nm, but considering that they were coated with a thin gold film of approximately 5 nm, it is expected that the thicknesses of the nanoplatelets are less than those observed by SEM analysis. Both the thickness of the nanoplatelets and the interplanar distance were obtained with the SEM-FEG software itself, confirming the multilayer nature of the evaluated graphene. The Energy-dispersive X-ray Spectroscopy (EDS) result showed only the presence of the carbon element, ruling out contamination by other chemical elements.

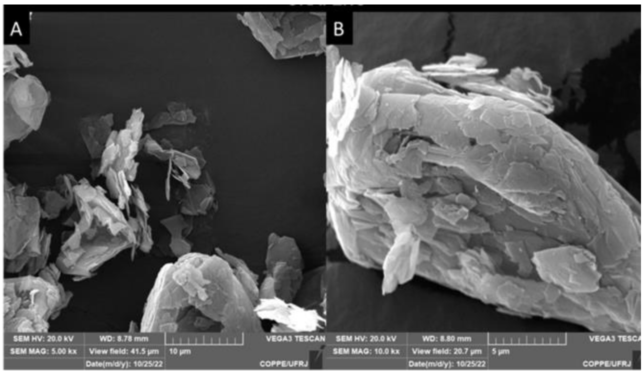


Figure 3. Graphene micrograph: (A) 5,000x zoom; (B) 10,000x zoom.

The VG 32 ISO mineral oil used to prepare the nanolubricants was characterized in terms of its physical properties, and the results are shown in Table 2. The results confirm the physical characteristics of a light neutral oil, as expected.

Table 2. Physical properties of the mineral oil ISO VG 32.

| PHYSICAL PROPERTIES OF MINERAL OIL ISO VG 32 | | | | |
|---|---|---|---|---|
| **Density (g/ml)** | **Dinamic viscosity at 40°C (cSt)** | **Dinamic viscosity at 100°C (cSt)** | **Viscosity index** | **Flash point (°C)** |
| 0.8697 | 29.02 | 5.09 | 102.10 | 220.00 |

VISUAL STABILITY EVALUATION OF NANOLUBRICANTS – The stability of nanolubricants is a critical factor that must be considered since nanoparticles tend to agglomerate over time due to their high surface energy [10]. With agglomeration, a sedimentation process occurs, causing the separation of the solid/liquid phases, thus extinguishing the existence of a nanofluid and its properties.

The stability evaluation was carried out through the visual observation method of sedimentation by gravity. This methodology is related to the density difference between the nanoparticles and the base fluid. In addition, the nanoparticles dispersed within the lubricating oil suffer random movements resulting from the Brownian force that neutralize the sedimentation and help keep them dispersed [10-12]. The nanolubricant formulations were left to rest in the same environment for a period of 7 days for stability evaluation.

Figure 4 shows the photographs of the dispersions as a function of time immediately after preparation, called day 0, and after 1 and 7 days.

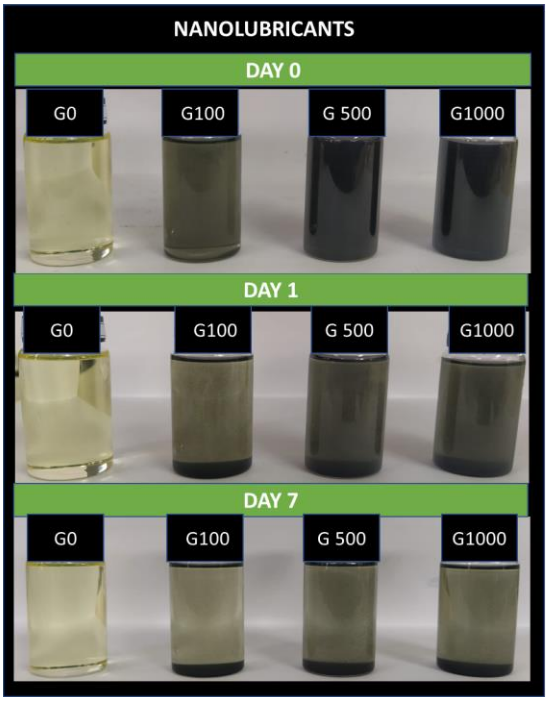


Figure 4. Stability test of formulations G0, G100, G500 and G1000, containing graphene 0-1,000 ppm, in the period of 0-7 days.

It can be seen from Figure 4, that there is an apparent stability of the nanolubricants after preparation (day 0). After the period of one and seven days, the suspensions decant, presenting a lighter coloration and background body, a result of instability. This destabilization phenomenon was observed by several authors [10-16], according to these, graphene sheets tend to bond and bind by weak Van der Waals bonds, generating agglomerations that facilitate destabilization and precipitation of the nanoadditive.

QUANTITATIVE ANALYSIS OF NANOLUBRICANT STABILITY – In the spectral absorption method, absorbance measurements by spectrophotometry in the ultraviolet-visible are used to characterize the colloidal stability of dispersion. Yu et al. [10] have shown that nanomaterials dispersed in fluids have characteristic absorption bands at the wavelength 190-1100 nm and, in general, there is a correlation between the absorbance intensity and the concentration of the nanoparticles in the fluid.

Therefore, it was possible to determine absorbance values of G100 and G1000 formulations over time, these formulations were chosen because they were the first concentrations studied. Table 3 shows the absorbance values in the visible range (600 nm) of the suspensions, 1 and 7 days after preparation. A comparative analysis was performed by the difference between the absorbance values obtained in the formulations in duplicate. As previously presented, from this information, it was possible to obtain a percentage of the absorbance change in the suspension after the analyzed period.

The results show that all formulations of nanolubricants showed a reduction in absorbance values over time, this occurs due to agglomeration and sedimentation of nanoparticles, leaving the nanolubricant less concentrated in the supernatant part, reducing the absorption of light waves.

Table 3. Absorbance values at 600 nm of the nanolubricant formulations at 0.1 and 7 days of evaluation

| | | WAVELENGTH - 600 $cm^{-1}$ | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | Absorvance (A) | | | Percentagem | | Average (%) | | Standart | |
| NANO LUBRICANTS | Test | Day 0 | Day 1 | Day 7 | Day 1 | Day 7 | Day 1 | Day 7 | Day 1 | Day 7 |
| G100 | 1 | 0.46 | 0.30 | 0.17 | 35.76 | 63.28 | 35.94 | 63.56 | 0.00 | 0.00 |
| | 2 | 0.47 | 0.30 | 0.17 | 36.14 | 63.84 | | | | |
| G1000 | 3 | 229.95 | 103.90 | 0.49 | 54.82 | 99.79 | 54.76 | 99.78 | 0.22 | 0.02 |
| | 4 | 234.09 | 107.61 | 0.53 | 54.03 | 99.77 | | | | |

At one day of stability evaluation, the formulations G100 and G1000 showed an average percentage of destabilization of 35.95 % and 54.68 %, respectively, i.e., with one day 35.9 5% of the additive added to the formulation G100 precipitated. After 7 days, formulations G100 and G1000 showed an increase in the average percentage destabilization of 63.56 % and 99.78 %, respectively. With these quantitative results there is evidence that the increase in the concentration of nanoadditive in base oil favors the instability of the suspension. It is noteworthy that the calculated standard deviations were low for all formulations, but with a slightly higher value for formulation G1000, for one day of stability testing.

TRIBOLOGICAL PERFORMANCE – Tribological tests were performed to determine the friction and wear properties of graphene dispersed in base oil, subjected to mechanical contact in the Four-ball equipment. The formulations G0, G100, G500 and G1000, equivalent to concentrations of 0, 100, 500 and 1000 ppm of graphene, were used. In addition, two Four-ball tests were performed for each formulation and the results were compared in order to obtain the real effect of different nanoadditive concentrations on the tribological response. Based on the visual stability test, it was noted that there is evidence of the onset of precipitation of the nanoparticles after 2 hours of dispersion, so the tests were performed immediately after dispersion of the nanoparticles in the oil. The formulations were subjected to 60 minutes of testing in the Four-ball at a constant temperature of 75±2 °C, according to ASTM D4172-21 (Table 1).

The Table 4 shows the wear results (average of the ball wear scar diameters) of the tests for the base oil (G0) and the formulations of nanolubricants G100, G500 and G1000. It is observed that the addition of nanoadditive reduced the wear scar for all the nanolubricant formulations evaluated (50.97 to 26.47 %) when compared to formulation G0 (base oil, no additives added). Zhang et al. [11] and Kim et al. [12] describe that this lubricity phenomenon occurs due to the mechanisms of nanoparticle deposition in the pores of the ball surface with the flow of the oil throughout the test. This allows the formation of a lubricating film, avoiding direct contact between the surfaces of the ball and test-oil cup device, which reduces frictional forces and increases the lubricating capacity.

Table 4. Tribological test results with formulations G0, G100, G500 and G1000 after testing on the Four-ball.

| NANO LUBRICANTS (ppm) | Test | WSD (mm) | Average (mm) | WSD Reduction (%) | STANDARD DEVIATION |
|---|---|---|---|---|---|
| G0 | 1 | 0.991 | 0.983 | -------- | 0.0113 |
| | 2 | 0.975 | | | |
| G100 | 3 | 0.484 | 0.482 | 50.97 | 0.002 |
| | 4 | 0.480 | | | |
| G500 | 5 | 0.528 | 0.523 | 46.80 | 0.0078 |
| | 6 | 0.518 | | | |
| G1000 | 7 | 0.734 | 0.733 | 25.43 | 0.0011 |
| | 8 | 0.732 | | | |

It is important to note that the best result was obtained for the formulation with the lowest additive concentration (100 ppm). Nevertheless, the results for G100 and G500 formulations are within the repeatability of the method (0.12 mm scar diameter). This result corroborates what is found in the literature, where many authors observe that graphene can be very promising in improving the tribological behavior when applied at low concentrations (100-500 ppm) in lubricants [13,14]. This event may be related to the excess of graphene nanosheets that generate agglomeration in the contact areas, and consequent reduction of the lubricating effect in these regions.

Rapoport et al. [15], describe that this phenomenon is associated with the formation of roughness on the contact surface during the tribological test. The smooth surfaces of the metal spheres become rough, forming valleys and trails around 150-300 nm. In this scenario, if the size of the thickness of the stacked nanoparticles, are larger than cracks generated in certain zones or regions, the lubricating action may be impaired.

In other words, nanoadditive-based lubrication systems need to remain in the contact zone during movement to protect surfaces from shear. From this point of view, the effect of nanoadditive size may come into play when we consider that the roughness of the shear surfaces can act as physical barriers that keep the nanoadditives within the contact zones. To illustrate this effect, one can consider two different conceptual cases in Figure 5. The first case (Figure 5A) refers to when the roughness length scale (h) characteristic of shear surfaces is smaller than the radius of the nanoadditive (r). Seen this, the ratio of roughness to nanoparticle radius is an indicator of how much lateral force is required to dislodge a nanoparticle from a roughness barrier (Figure 5B).

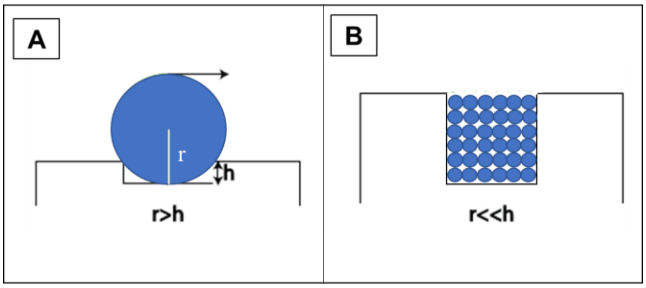


Figure 5. The effect of surface roughness on the organization of nano-charges between the asperities of the shear surfaces: A) the ratio of the roughness <radius of the nanoparticle; B) the ratio of the roughness ≥ the radius of the nanoparticle.

It is relevant to mention that graphene has been considered a promising additive for lubricant fluid due to the following characteristics: i) weak interaction between the nanosheets (Van der Waals forces) allows the structure to shear at low shear stress levels; ii) possibility of the nanosheets adhering to moving surfaces (formation of tribofilm during contact) also causing surface regeneration; iii) ability to resist load application (up to 20N).

Figure 6, shows the micrographs of the three spheres after the tribological tests, along with the x- and y-axis wear diameter markings, which resulted in the WSD values for the four formulations. It can be seen that Figure 6A, referring to formulation G0 (without nanoadditive) the wear scar is noticeably more prominent and deeper, and is characterized as adhesive wear and some abrasive wear. However, the worn surfaces of the material lubricated by the nanoadditives (Figure 6B-D) show smoother wear scar surfaces, showing signs of fine and less deep scratches compared to the base oil. This behavior may be associated with the penetration of graphene nanosheets into the worn regions, causing surface regeneration.

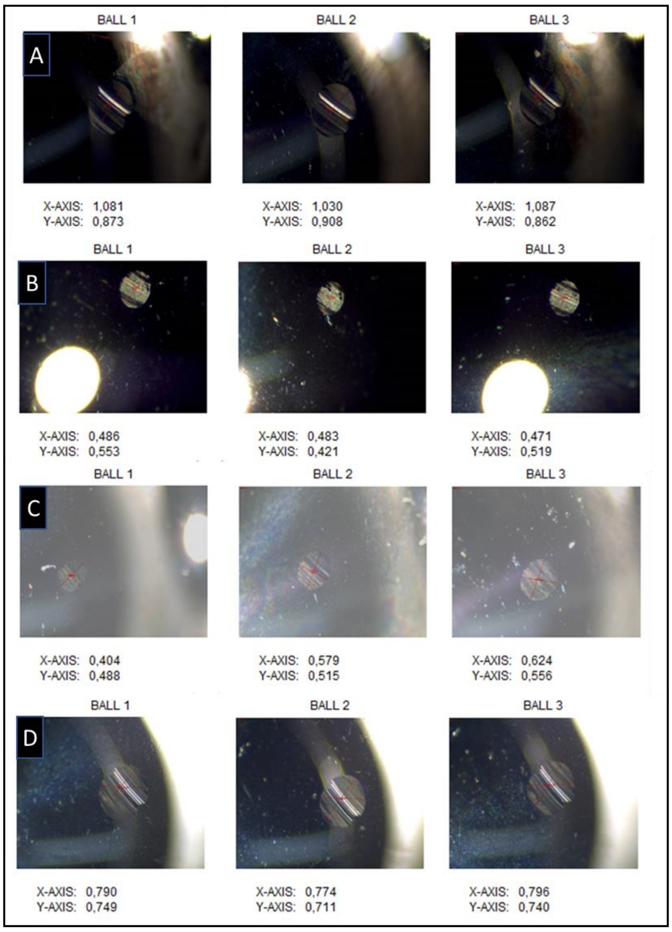


Figure 6. Micrographs of the wear scar of the three beads containing the formulations: A) G0; B) G100; C) G500 and D) G1000 after testing in Four-ball.

The coefficients of friction (COF) obtained in the tribological tests, resulting from the formation of a lubricating film of the pure oil and the nanolubricants are presented in Figure 7. The average COF resulting from duplicate tests for G0, G100, G500 and G1000 formulations were 0.35, 0.25, 0.27 and 0.30 respectively, with a very low standard deviation in the range of 0.005 to 0.003. The results show that the average coefficient of friction of all nanolubricants were lower than that of the pure oil for all concentrations. The lowest coefficient of friction found was 0.26 for the G100 formulation containing 100 ppm graphene. This represents an improvement in tribological behavior due to friction of 26.47 % when compared to the result for the pure base oil. It is worth noting that the calculated standard deviations were very low for all formulations.

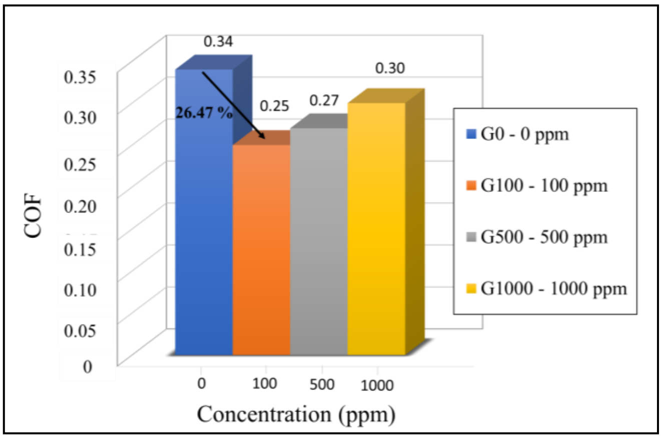


Figure 7. Average coefficients of friction (COF) obtained in the Four-ball test for the formulations: G0, G100, G500 and G1000.

The friction reduction and anti-wear mechanism of nanoadditives in lubricants have been extensively investigated, and can be described as follows: rolling effect, protective film, splicing effect and polishing effect [16]. Some mechanisms of lubrication by nanoadditives illustrated in Figure 8 are proposed by researchers [16-19].

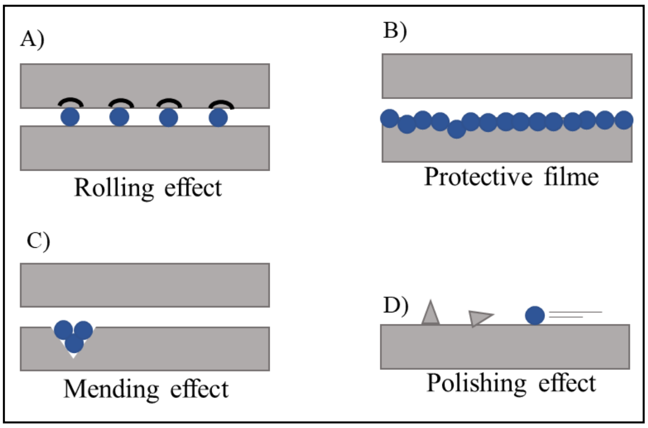


Figure 8. Possible lubrication mechanisms of nanoparticles as friction modifiers: a) rolling effect; b) protective film; c) mending effect; and d) polishing effect.

In view of this, graphene is believed to be a promising nanoadditive capable of improving the performance of nanolubricants for the automotive industry, when applied at low concentrations.

## CONCLUSIONS

Considerable effort has been made to explore and develop new types of lubricating oil nanoadditives to reduce wear and friction in tribological systems and improve surface morphology. It was observed that there is a need to develop a technology capable of dispersing and stabilizing graphene in lubricating oil.

Moreover, this paper presented the results of graphene nanoparticles as a potential friction modifier and anti-wear in oil. In summary, the present study shows that nanolubricants can reduce the coefficient of friction and increase wear resistance compared to conventional base oil. The significant reduction in wear can be attributed to the fact that nanoparticles in the contact regions are able to protect the surfaces from severe friction and adhesion wear. These phenomena proved the importance of uniform dispersion of nanoparticles to increase wear protection.

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