

Article

Tribological Performance of a Paraffinic Base Oil Additive with Coated and Uncoated SiO₂ Nanoparticles

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Abstract: Electric vehicles (EVs) have emerged as a technology that can replace internal combustion vehicles and reduce greenhouse gas emissions. Therefore, it is necessary to develop novel low-viscosity lubricants that can serve as potential transmission fluids for electric vehicles. Thus, this work analyzes the influence of both SiO₂ and SiO₂-SA (coated with stearic acid) nanomaterials on the tribological behavior of a paraffinic base oil with an ISO VG viscosity grade of 32 and a 133 viscosity index. A traditional two-step process through ultrasonic agitation was utilized to formulate eight nanolubricants of paraffinic oil + SiO₂ and paraffinic base oil + SiO₂-SA with nanopowder mass concentrations ranging from 0.15 wt% to 0.60 wt%. Visual control was utilized to investigate the stability of the nanolubricants. An experimental study of different properties (viscosity, viscosity index, density, friction coefficient, and wear) was performed. Friction analyses were carried out in pure sliding contacts at 393.15 K, and a 3D optical profilometer was used to quantify the wear. The friction results showed that, for the SiO₂-SA nanolubricants, the friction coefficients were much lower than those obtained with the neat paraffinic base oil. The optimal nanoparticle mass concentration was 0.60 wt% SiO₂-SA, with which the friction coefficient decreased by around 43%. Regarding wear, the greatest decreases in width, depth, and area were also found with the addition of 0.60 wt% SiO₂-SA; thus, reductions of 21, 22, and 54% were obtained, respectively, compared with the neat paraffinic base oil.

Keywords: friction; wear; nanolubricants; nanoparticle surface modification



Citation: Liñeira del Río, J.M.; Guimarey, M.J.G.; Somoza, V.; Mariño, F.; Comuñas, M.J.P. Tribological Performance of a Paraffinic Base Oil Additive with Coated and Uncoated SiO₂ Nanoparticles. *Materials* **2024**, *17*, 1993. <https://doi.org/10.3390/ma17091993>

Academic Editor: Csaba Balázi

Received: 15 March 2024

Revised: 11 April 2024

Accepted: 22 April 2024

Published: 25 April 2024



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1. Introduction

Energy needs are constantly increasing; consequently, the natural environment is significantly impacted. This is the case with the transport sector, as it is responsible for a large part of carbon dioxide, CO₂, gas emissions, and climate change due to fuel-powered machinery [1]. For this reason, automotive industries need to develop new technologies to produce highly efficient vehicles for individual and public mobility [2,3]. Thus, electrification designs (hybrid or electric vehicles (EVs)) have emerged as an optimal solution for new propulsion systems to reduce greenhouse gas emissions [4], although a specific analysis of each country is necessary to ensure proper emissions reduction [5]. These greenhouse gas reductions, particularly that of CO₂, greatly depend on the source of the electricity [6]. When electricity originates from renewable energy sources, the CO₂ emissions of an EV are 4.5 times less than those of a combustion engine car [6]. Although EVs are very efficient and produce very low exhaust emissions, they have efficiency and endurance issues that affect the moving components and thus their tribology. Thus, tribological solutions such as new materials or optimized lubricants can help to

increase the driving range of EVs since tribology can help to enhance the efficiency by lowering friction in elements like gears and wheel bearings [7].

Even though EVs exhibit significantly elevated efficiency in terms of energy use, there are still challenges related to the need to further enhance the efficiency; hence, the improvement of new fluids and materials [7] and the progression of new batteries [8] are being promoted. EVs require transmission oil lubricants with greater technical requirements [9] than those of internal combustion motors; this is because during the operation process contact is made with copper wires, sensors, and circuits [10]. Moreover, the high rotation rates of the electric motor require the use of lubricants with very low viscosity. If the oil viscosity is reduced, viscous drag and viscous heating drop and, therefore, the heat transfer is raised [9,11]. However, if the viscosity of a lubricant is lowered a shift from full film to boundary lubrication occurs and a more critical surface contact and wear is produced. This circumstance indicates that enhanced anti-wear and anti-friction properties are required. Therefore, to meet the needs of future EV lubricants, it is fundamental to use advanced additives [12]. The best conventional lubricants used for ICEVs, with the chance to be used in EV automotive elements, are made of mineral-based oils prepared with several additives to meet the rigorous requirements [7].

Recently, nanotechnology-based anti-friction and anti-wear additives were suggested for transmission fluids for EVs [7]. Then, the research on nanoparticles as oil additives was able to support the advance of a novel production of lubricants with low viscosity that were specifically modified to meet the necessities of EVs (electrified transmissions), owing to their outstanding anti-friction and anti-wear capacities, which can lead to an extended life in the operating conditions of EVs [12–14]. Furthermore, nanomaterials are more ecologically friendly than other conventional additives [15,16]. A crucial part of achieving a proper nanolubricant is the creation of temporal stability for an extended time; the sedimentation of nano-additives can lead to a decrease in efficiency and system damage owing to the abrasive wear [17]. To improve the stability of nanolubricants, different procedures can be carried out, using surfactants, physical treatment, or chemical surface modification [18,19]. The requirement of stable nanolubricants is particularly important for those lubricants composed of oils with low viscosity owing to the poor stability of the nanoparticles in such fluids. Even though lubricants with additives containing nanomaterials have exhibited good anti-friction and anti-wear performances in conventional lubricants [13,20–22], there is scarcely any investigation on nanolubricants with regard to EVs' tribological needs. Mustafa et al. [9] have recently reviewed the tribological performance of several low-viscosity lubricants, based on different polyalphaolefin low-viscosity base oils and water. For instance, Chou et al. [23] analyzed the effect of adding Ni nanoparticles (20 nm) on the tribological activity of PAO6 base oil; they observed a reduction in friction between 7% and 30% and in wear between 5% and 45%, and they achieved the highest friction and wear reductions with the PAO6 + 0.5 wt% Ni nanolubricant. Because of the needs, it is necessary to improve and study potential stable lubricants formed through base oils with low viscosity and nano-additives. In this investigation, a paraffinic G-III base oil was selected to meet those qualities. The nano-additives used in this work, SiO₂ nanoparticles, have exclusive physical and chemical characteristics; therefore, they can be used in several fields, such as in adhesives, the textile industry, and lubrication [24]. In fact, SiO₂ as a lubricant additive usually shows an excellent anti-wear property, due to the fact that SiO₂ has hydroxyl and unsaturated bonds and can form a solid chemical adsorption film to protect the metallic surface, significantly improving the friction performance of the lubricating oil. Furthermore, it has good electrical, optical, and magnetic properties and has received considerable interest in terms of applications such as those of catalysis, pharmaceuticals, drug delivery, and pigments. SiO₂ nanopowder is a solid and colorless crystalline substance, which does not react with water and is resistant to acids. Furthermore, in this research, commercial SiO₂ nanoparticles were chemically functionalized with stearic acid (SA) through an esterification process to enhance their stability in G-III base oil. SiO₂ nanoparticles were studied as lubricant additives and demonstrated good ability as

friction and wear modifiers [25–30]. For instance, Cortés et al. [25] studied the tribological performance of non-coated SiO₂ nanoparticles, such as the additives of a vegetable oil, achieving decreases of up to 77% in terms of friction and 74% in terms of wear volume. Additionally, some authors [17,26] functionalized the SiO₂ nanoparticle surface with the aim of enhancing the temporal stability of the nano-dispersions. For instance, Peng et al. [17] coated the SiO₂ nanoparticles with oleic acid (SiO₂-OA), reaching a temporal stability of about one month for a paraffin oil, with the mass percentages shifting from 0.05 to 1.0 wt% of the SiO₂-OA.

In this article, we focus our attention on the use of commercial SiO₂ and stearic acid-coated SiO₂ (SiO₂-SA) nanoparticles as additives of a G-III base oil, and these nanolubricants were tribologically analyzed at high temperature (393.15 K) in pure sliding contacts.

2. Materials and Methods

2.1. Base Oil and Nanoparticles

The paraffinic G-III base oil was provided by Repsol S.A. (Madrid, Spain); it possesses a dynamic viscosity and density of 28.9 mPa and 0.8234 g·cm⁻³ at 313.15 K, respectively, and a 133 viscosity index. This oil was previously fully characterized through infrared spectroscopy (FTIR) and Raman spectroscopy; peaks associated with CH₃ and CH₂ stretching were observed using FTIR, and others attributed to C-H and C-C stretching were found using Raman spectroscopy [31]. Regarding the nano-additives, two different types of SiO₂ nanoparticles were used. The first ones were commercial SiO₂ nanoparticles provided by the company US Research Nanomaterials, Inc. (Houston, TX, USA), with a purity of 99% and a diameter of 8 nm. The second ones were the same SiO₂ nanoparticles but coated in our laboratory with stearic acid (SiO₂-SA). The SiO₂ nanopowders were characterized by means of transmission electron microscopy (TEM); it can be seen in Figure 1a that the studied SiO₂ NPs have a roughly spherical shape. Through the TEM characterization, the calculation of the average particle size was performed using Image J software (version 1.54h). Thus, as shown in Figure 1b, average sizes of around 11 nm were reached and were similar to the average size provided by the manufacturer (8 nm). Furthermore, in a previous work [32], infrared spectra of SA, uncoated SiO₂ NPs, and SiO₂-SA NPs were also reported; it was observed, among other information, that the characteristic peaks of SA also appear in the spectrum of SiO₂-SA, evidencing a proper SA coating with the SiO₂ nanoparticles.

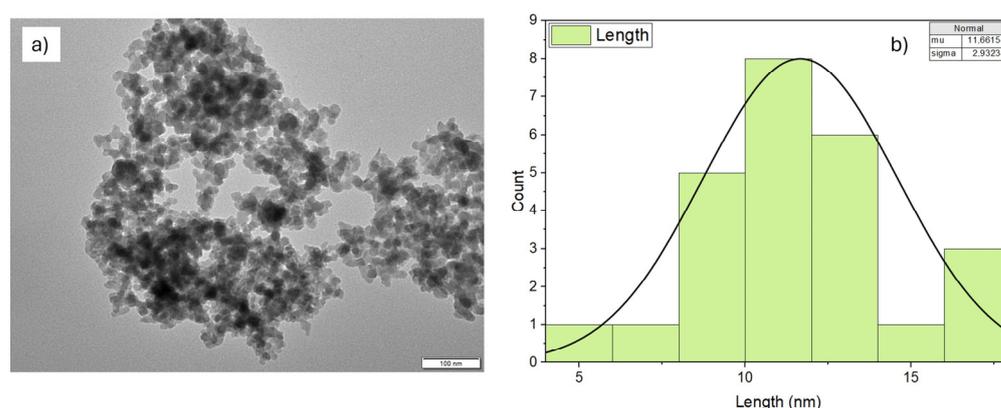


Figure 1. Image of TEM (a) and mean size particle distribution of SiO₂ NPs (b).

2.2. Formulation of Nanolubricants

The uncoated SiO₂ nanolubricants were formulated with different mass concentrations of SiO₂ (0.15, 0.30, 0.45 and 0.60 wt%) in G-III base oil. For this purpose, a conventional two-step method and a Sartorius MC 210P microbalance (± 0.00001 g) were utilized. Furthermore, an ultrasonic method (Ultrasonic bath FB11203 Fisherbrand from Fisher Scientific, Hampton, VA, USA) was used for 4 h to homogenize the SiO₂-based nanolubricants. On the

other hand, to prepare the SiO₂-SA nano-dispersions, commercial SiO₂ nanopowders were coated with SA following the chemical reaction given in Figure 2a and then the dispersion method displayed in Figure 2b, to finally obtain a 4 wt% SiO₂-SA nanolubricant. More details about a similar functionalization process can be seen in our previous article [32].

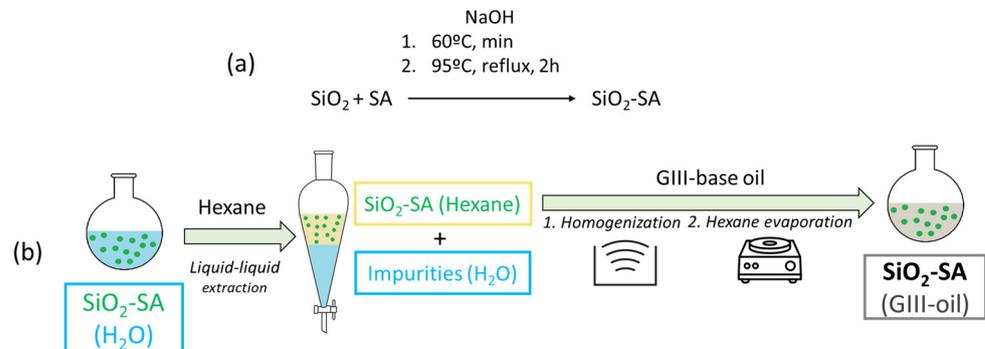


Figure 2. Scheme of the nanoparticle functionalization (a) and dispersion method (b).

Therefore, dilutions of the achieved 4 wt% SiO₂-SA nanolubricant were performed by adding G-III base oil, until reaching the desired (0.15, 0.30, 0.45, and 0.60 wt%) SiO₂-SA nanolubricants. After the dilutions, the nanolubricants were also homogenized via an ultrasonic bath, as in the case of the bare SiO₂ nanolubricants. Furthermore, the temporal stability of the nanolubricants was evaluated by visual control and refractive index evolution of the samples over time.

2.3. Thermophysical Characterization

The density of the nanolubricants was examined from 278.15 to 373.15 K, utilizing a vibrating densimeter Anton Paar (Graz, Austria) SVM 3000 Stabinger. The expanded ($k = 2$) uncertainty of the density measurements was 0.0005 g cm⁻³. The viscosity at atmospheric pressure and the viscosity index (VI) of the nanolubricants were also analyzed with the aforementioned densimeter. This device can measure kinematic and dynamic viscosities between 278.15 and 373.15 K. A relative expanded ($k = 2$) uncertainty of 1% was calculated for the dynamic viscosity.

2.4. Tribological Characterization

Friction tests were carried out in pure sliding contacts with a rheometer MCR 302 from Anton-Paar, kitted with a tribology unit T-PTD 200 and utilizing a Peltier hood H-PTD 200 for an ideal temperature control. In this research, a ball-on-three-pins test disposition was utilized; the ball is put on a shaft and set to turn by the rheometer motor, while being pushed at the same time against the three pins. The rheometer axial force is transferred into a normal force which proceeds perpendicularly to the contact positions on the pins. In this case, the ball turns on the pins below a 20 N normal force, resulting in a load of 9.43 N in each pin, which corresponds to a maximum contact pressure of around 0.8 GPa. Friction experiments were conducted at a constant rotational speed of 213 rpm and for 3400 s at 393.15 K. The specimens tested were polished AISI 52100 (100Cr6) steel balls ($R_a = 20$ nm) and pins ($R_a = 50$ nm) with a hardness of 62–66 HRC. The ball had a 12.7 mm diameter, and the cylindrical pins had a diameter and height that were both 6 mm. The balls and pins were cleaned with acetone/hexane and dried with air prior to the tribological tests. The pins were completely flooded by adding over 1.2 mL of each tested nanolubricant or base oil. At least three replicates were tested for each concentration of lubricant to obtain representative values. More information involving this tribological machine can be obtained from an earlier article [31]. To inspect the worn pins after the tribological studies, a 3D Optical Profiler was employed to measure the wear created in the pins for diverse parameters, such as wear scar diameter (WSD), wear track depth (WTD), or worn area. These parameters were analyzed in the three different pins by means of a confocal

mode (10× objective). Moreover, a WITec alpha300R+ confocal Raman microscope (Oxford Instruments, Abingdon, UK) was utilized to obtain knowledge regarding the spreading of the nanoparticles in the worn pins.

3. Results

3.1. Stability of the Dispersions

The stability of the SiO₂ and SiO₂-SA nanolubricants was checked using two different techniques: visual observation and temporal evolution of the refractive index using a Mettler Toledo RA-510 M refractometer (Columbus, OH, USA). Figure 3a reveals that sedimentation does not happen for the first 96 h after the nanolubricant formulation, for the coated SiO₂-SA nanolubricants. Conversely, in the case of the uncoated SiO₂ nanolubricants, it can be observed in Figure 3a that 24 h after the preparation, the sedimentation takes place. Thus, through the SA coating of SiO₂ nanoparticles a better stability is reached. Similar stability behavior was observed in other studies using the stearic or oleic acid as the coating of the NPs, and stability improvements were achieved [19,33]. Figure 3b shows the temporal evolution of the refractive index (*n*) for the base oil and 0.6 wt% SiO₂-SA nanolubricant. As can be seen, the tendencies of the refractive index evolution for the SiO₂-SA nanolubricant and base oil are very similar, confirming a good stability against sedimentation.

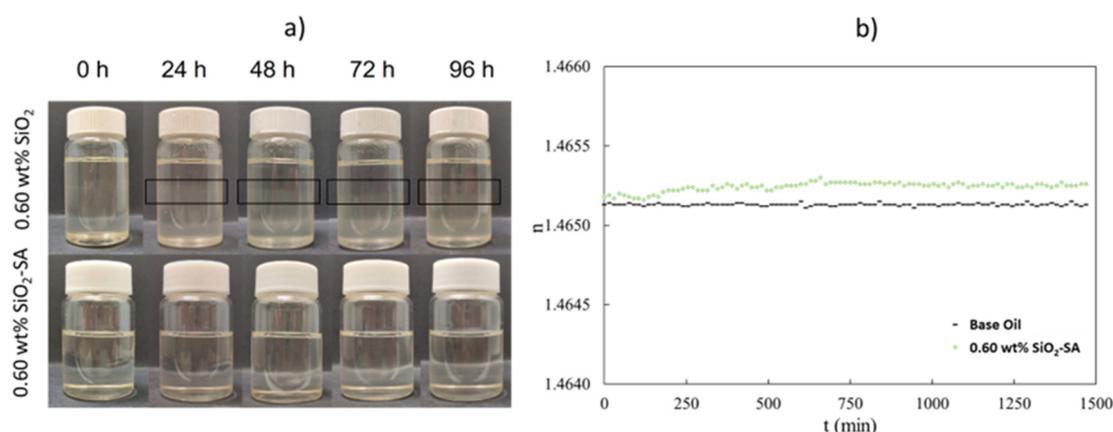


Figure 3. (a) Visual stability observation and (b) temporal evolution of the refractive index for SiO₂-SA nanolubricant and base oil.

3.2. Thermophysical Results

The experimental densities and dynamic viscosities acquired for the base oil and SiO₂ and SiO₂-SA nanolubricants are reported in Tables S1 and S2 (Supplementary Materials). Figure 4a shows the relative variation in the densities of the nanolubricant concentration with respect to the neat paraffinic base oil. For the SiO₂ nanolubricants, a clear increase in density variation is observed as a function of the mass concentration of the nanoparticle; the higher the concentration, the higher the density of the nanolubricant. Thus, the SiO₂ nanolubricants at 0.15, 0.3, 0.45 and 0.6 wt% increase relatively with respect to the neat base oil densities of 0.10, 0.20, 0.29 and 0.34%, respectively. The rise in nanolubricant density with the nanoparticle concentration is attributable to the agglomeration phenomenon [34]. However, for the SiO₂-SA nanolubricants, the relative density increase is similar (around 0.03%) for all the concentrations of the functionalized nanoparticles. The relative viscosity variation in the SiO₂ and SiO₂-SA nanolubricants compared to the neat G-III paraffinic base oil is shown in Figure 4b. The dynamic viscosity rises as the concentration of SiO₂ nanoparticles grows from 1% to 12%. Regarding the SiO₂-SA nanoparticles, the growth in viscosity varies between 12 and 18% for the 0.15 and 0.3 wt% SiO₂-SA nanolubricants, respectively.

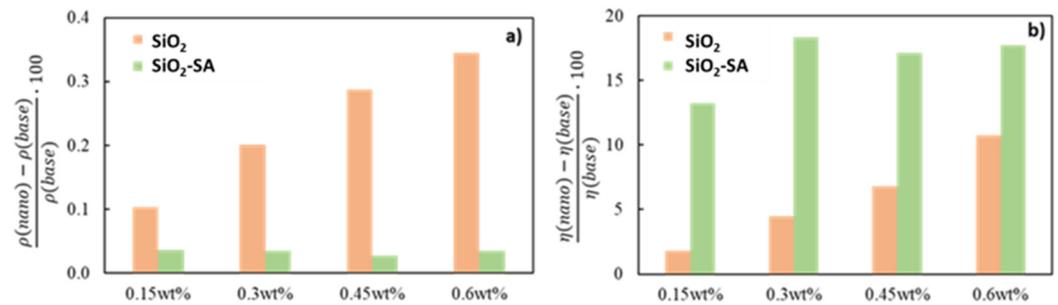


Figure 4. Relative increase in the densities (a) and viscosities (b) with the mass concentration with respect to the neat paraffinic base oil.

Additionally, with the aforementioned SiO₂ and SiO₂-SA nanolubricants, the impact of concentration on the viscosity index (VI) was analyzed, as shown in Figure 5. A suitable viscosity index (VI) is essential in a lubricant since it helps to avert collisions and friction among the mechanical device components during operation, while also enhancing the machine's efficiency [35]. It can be observed that all the samples have a higher viscosity index (VI) than the neat base oil, which confirms that the nanolubricant remains useful even at elevated temperatures with the preservation of the thickness of the oil film. The results show that VI increased from 3% to 13% and from 11% to 15% for the SiO₂ and SiO₂-SA nanolubricants, respectively, compared with the neat base oil.

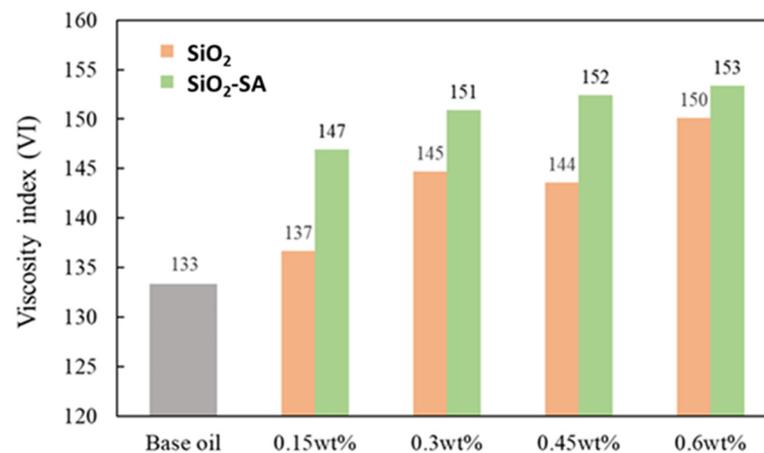


Figure 5. Viscosity index (VI) for the neat paraffinic base oil and for the SiO₂ and SiO₂-SA nanolubricants.

3.3. Tribological Results

Figure 6 and Table 1 present the mean values of the coefficient of friction (μ) for all the tested lubricants based on G-III paraffinic base oil. The friction coefficients found for all the uncoated SiO₂ nanolubricants are quite similar to that reached for the neat G-III base oil (without additives). Nonetheless, for the coated SiO₂-SA nanolubricants the obtained friction coefficients are much lower than that previously reported using the neat G-III base oil [31]. Specifically, the optimal nanoparticle concentration was attained for the 0.60 wt% SiO₂-SA nanolubricant, with a friction decrease of around 43% (μ of 0.077 was found against 0.134). This promising friction performance can be explained by the synergetic effect between the SiO₂ nanoparticles and the coating of stearic acid.

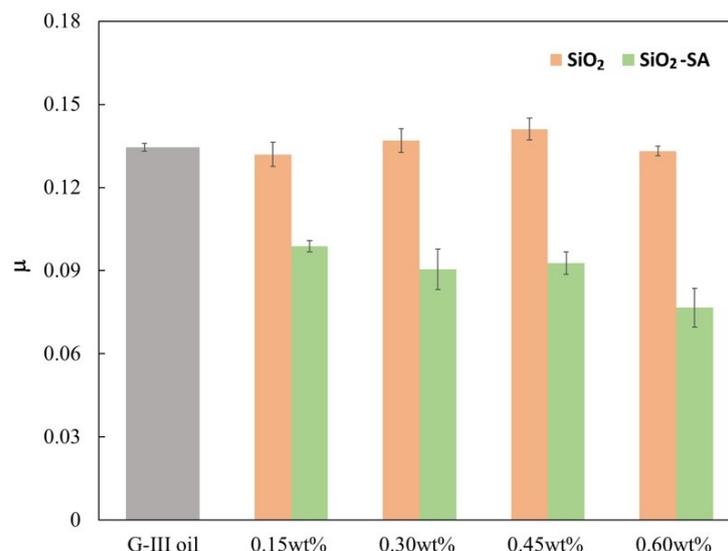


Figure 6. Mean friction coefficients, μ , for the prepared SiO₂ and SiO₂-SA nanolubricants and for the neat G-III base oil [31].

Table 1. Average coefficients of friction, μ , and mean parameters of wear with their standard deviations for the studied G-III base oil nanolubricants at 393.15 K.

Sample	μ	σ	WSD/ μm	$\sigma/\mu\text{m}$	WTD/ μm	$\sigma/\mu\text{m}$	Area/ μm^2	$\sigma/\mu\text{m}^2$
G-III base oil [31]	0.1351	0.0014	366	18	2.11	0.19	607	44
+0.15 wt% SiO ₂	0.1319	0.0011	380	23	2.76	0.72	571	31
+0.30 wt% SiO ₂	0.1370	0.0010	422	53	3.37	0.67	806	76
+0.45 wt% SiO ₂	0.1410	0.0011	352	31	2.61	0.45	476	55
+0.60 wt% SiO ₂	0.1332	0.0012	372	8.3	2.67	0.46	726	49
+0.15 wt% SiO ₂ -SA	0.0989	0.0014	356	23	2.00	0.49	439	99
+0.30 wt% SiO ₂ -SA	0.0905	0.0011	371	18	1.60	0.19	401	62
+0.45 wt% SiO ₂ -SA	0.0927	0.0010	296	54	1.80	0.38	279	98
+0.60 wt% SiO ₂ -SA	0.0766	0.0011	289	25	1.64	0.47	281	84

As cited previously, the wear formed in the pins after the friction tests was estimated through many parameters of the wear track: width, depth, and area. For this goal, cross-section profiles and 3D mappings of the wear tracks were taken. The WSD, WTD, and transversal area mean values were taken from the profiles of the worn tracks on the pins tested with the nanolubricants and base oil. The values are reported in Table 1.

As with the friction results, the SiO₂ nanolubricants revealed similar wear results to those of the G-III base oil. Nonetheless, for all the SiO₂-SA-based nanolubricants, the produced wear was greatly inferior to that achieved with neat G-III base oil, particularly in the case of the worn areas (Figure 7). Furthermore, the additive mass concentration used in the nanolubricant design considerably influenced the lubrication performance. Specifically, the greatest decreases in width and area were reached with the G-III base oil + 0.60 wt% SiO₂-SA nanolubricant (Table 1), with reductions of 21 and 54%, respectively (Figure 8).

Similar improved tribological performances with SiO₂ NPs were previously obtained by other authors. Thus, Sanukrishna et al. [29] studied the tribological properties of SiO₂ NPs as additives of a PAG lubricant, observing friction reductions of around 38% and wear reductions of 41%. Also, Rastogi et al. [30] studied the effect of SiO₂ nanoparticles on the tribological characteristics of jatropha oil, obtaining important friction and wear reductions for different normal loads.

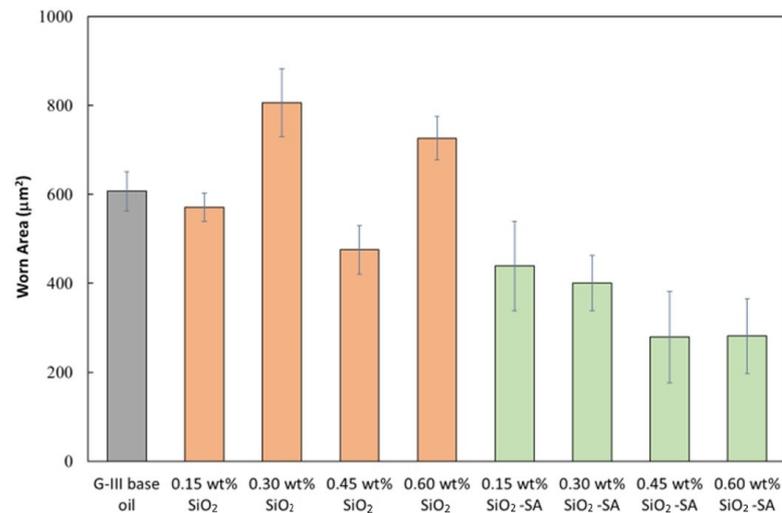


Figure 7. Mean worn areas, found for all the tested SiO₂ and SiO₂-SA nanolubricants and for the neat G-III base oil.

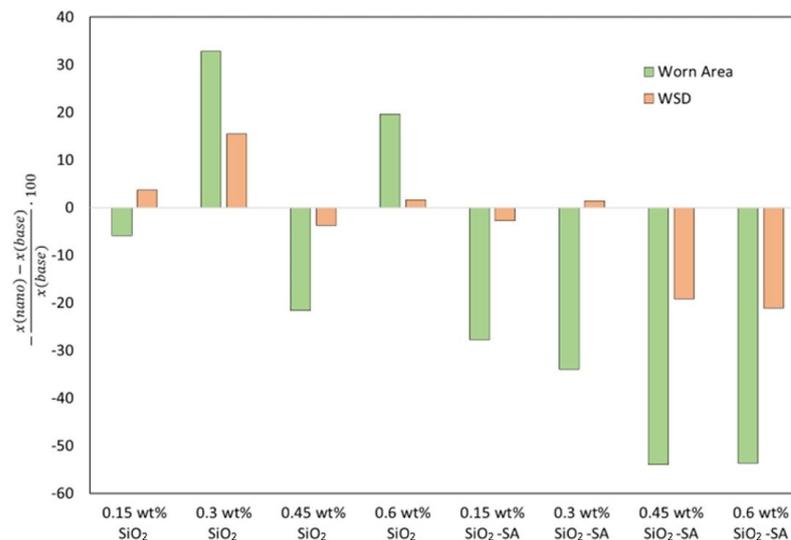


Figure 8. Mean reductions in WSD and worn area, obtained for all the tested SiO₂ and SiO₂-SA nanolubricants.

Additionally, it can be clearly observed in the worn profiles in Figure 9 that the optimal SiO₂ nanolubricant that contains the stearic acid coating presents considerably better anti-wear capacities with respect to the G-III base oil and the optimal uncoated SiO₂ nanolubricant.

Furthermore, the Raman spectra of the nanolubricant components evidence the fact that characteristic areas of these elements appear in the worn surfaces of the pins (Figure 10). Thus, in Figure 10a blue areas of Raman mapping are associated with iron oxides, green areas with the base oil, and red areas with the burned oil. In Figure 10b, it can be seen from the presence of blue areas associated with the SiO₂-SA nanoparticles that the spectrum of this area coincides with the SiO₂-SA Raman spectrum [32]. Considering these Raman analyses, a protective tribofilm from the SiO₂-SA in the tribo-contact can be a possible tribological mechanism that participates in the decrease in friction and wear. Thus, the spherical SiO₂ NPs that are dispersed in the paraffinic lubricant with the high contact pressure can enter into the interspace of contact surfaces and progressively deposit on surfaces, causing the creation of a physical film. This tribofilm can separate the two metal surfaces and prevent direct contact [36]. Furthermore, some tribochemical reactions can

occur, boosted by the high temperatures and pressures caused by the friction process. Hence, these conditions could cause the breaking of the bonds between SA and the coated SiO₂ NPs, as was previously pointed out by Zhang et al. [37] for SA-modified TiO₂ NPs. SiO₂ NPs can easily be adsorbed on the worn surface, generating a boundary-lubricating film, whereas the SA can also be physically adsorbed on the steel surface during the tribotests, generating good lubricant properties [38,39]. Likewise, owing to the spherical nature of SiO₂ NPs, they are more likely to roll between two surfaces, reducing the friction coefficient and wear. Therefore, rolling and tribofilm formation are the two possible tribological mechanisms. Similar results were previously obtained by Xie et al. [40] for SiO₂ NPs dispersed in engine oil.

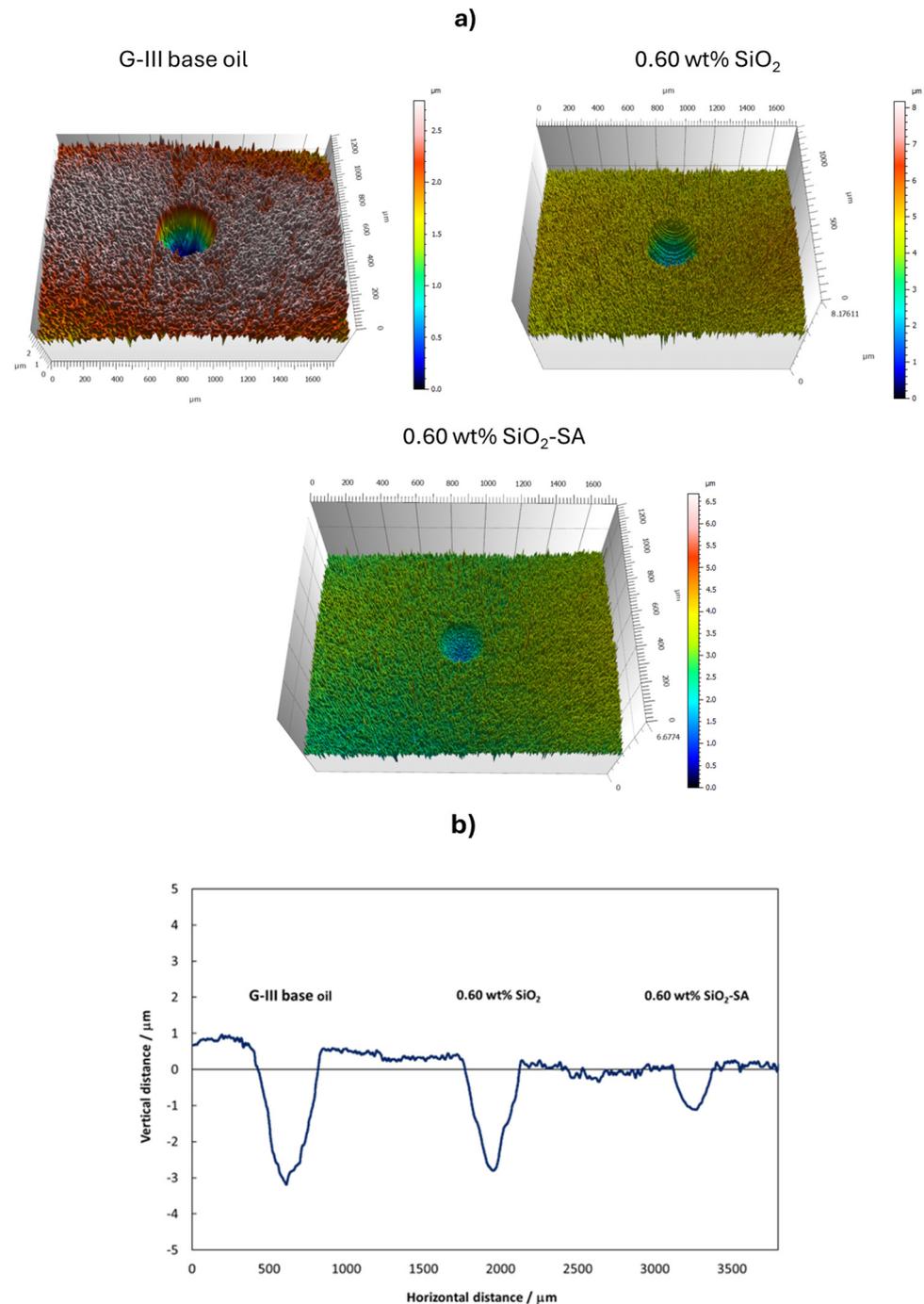


Figure 9. Three-dimensional (a) and two-dimensional (b) profiles of worn pins tested with neat G-III base oil [31], 0.60 wt% SiO₂, and 0.60 wt% SiO₂-SA.

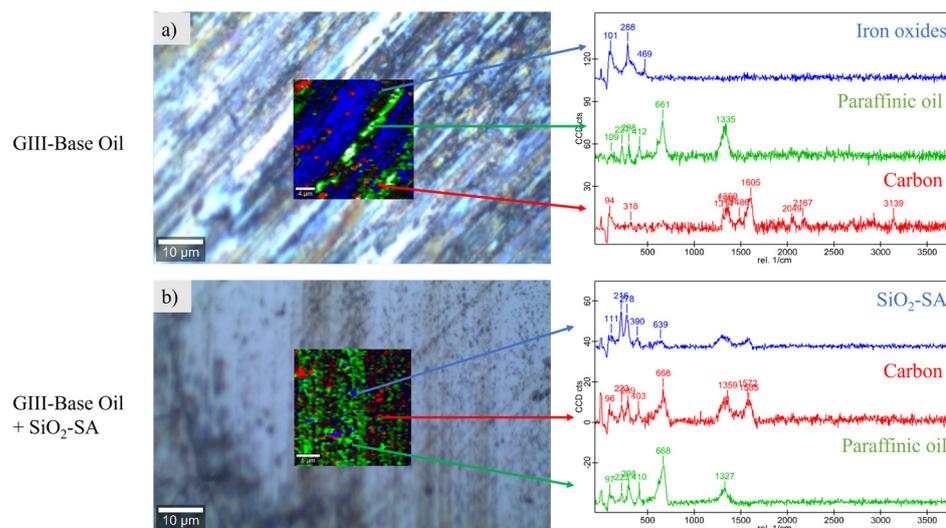


Figure 10. Mapping of Raman with worn pins tested with G-III base oil and 0.60 wt% SiO₂-SA.

4. Conclusions

The formulation and tribological characterization of modified SiO₂ nanolubricants was performed with the aim of contributing to the EV industry demands for developed lubricants that can be used in the future as EV transmission fluids, leading to an improvement in their energy efficiency. Therefore, the main conclusions of this investigation are:

- Good stability against sedimentation was achieved with SiO₂ nanoparticles, coated with stearic acid and dispersed in a paraffinic base oil.
- Friction coefficients found with the SiO₂-SA nanolubricants are reduced in comparison with the neat G-III base oil.
- The wear produced in the pins is lower than that found with G-III base oil; the optimal nanolubricant is the G-III base oil + 0.60 wt% SiO₂-SA, with reductions of up to 21, 22, and 54% in terms of width, depth, and area, respectively.
- The tribological lubrication mechanisms can be justified by the rolling and tribofilm formation of the SiO₂-SA nanoparticles.
- Before applying these nanolubricants in EVs, more studies are needed, such as tribological tests for extended times to estimate their possible degradation and the study of other key properties, such as thermal or electrical conductivity, and the compatibility and synergies with other additives used in the lubricant formulation.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/ma17091993/s1>, Table S1: Experimental density determined with Stabinger densimeter for the paraffinic base oil and the nanolubricants at different temperatures and at 0.0991 MPa, Table S2: Experimental viscosity determined with Stabinger rotational viscometer for the paraffinic base oil and the nanolubricants at 0.0991 MPa at different temperatures.

Author Contributions: Conceptualization, J.M.L.d.R.; methodology, J.M.L.d.R., V.S., F.M. and M.J.G.G.; validation, M.J.P.C.; formal analysis, M.J.P.C.; investigation, J.M.L.d.R. and V.S.; writing—original draft preparation, J.M.L.d.R. and M.J.G.G.; writing—review and editing, J.M.L.d.R., M.J.G.G., M.J.P.C. and F.M.; supervision, M.J.P.C.; project administration, M.J.P.C.; funding acquisition, M.J.P.C. All authors have read and agreed to the published version of the manuscript.

Funding: This research is supported by Xunta de Galicia (ED431C 2020/10), by the State Research Agency (AEI) of Spain, and by the European Regional Development Fund (ERDF, FEDER in Spanish) through the PID2020-112846RB-C22 project. J.M.L.d.R. acknowledges the grant of the Margarita Salas program, funded by MCIN/AEI/10.13039/501100011033 and “NextGenerationEU/PRTR”. M.J.G.G. also acknowledges the Xunta de Galicia (Spain) for the postdoctoral fellowship (reference ED481D 2023/016).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Data are contained within the article and Supplementary Materials.

Acknowledgments: The authors also thank Repsol Lubricants for providing the G-III base oil.

Conflicts of Interest: The authors declare no conflicts of interest.

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