

1 **Tribological behavior of electric vehicle transmission**
2 **oils using Al₂O₃ nanoadditives**

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18 **ABSTRACT:** Antifriction and antiwear performances of Al₂O₃ nanoparticles (NPs) as additives of an
19 automatic transmission fluid, ATF are presented in this research. For this purpose, four
20 nanodispersions were formulated: ATF + 0.05 wt% Al₂O₃ NPs, ATF + 0.10 wt% Al₂O₃ NPs, ATF + 0.15
21 wt% Al₂O₃ NPs and ATF + 0.20 wt% Al₂O₃ NPs to identify the optimal concentration of additive.
22 Tribological experiments were taken at pure sliding conditions, with the formulated nanolubricants
23 and the ATF, under a working load of 20 N. The four nanolubricants tested resulted in lower friction
24 coefficients than those obtained using ATF, reaching a maximum reduction of 6% with the ATF +
25 0.10 wt% Al₂O₃ nanolubricant. The tribological pairs tested with the Al₂O₃ nanolubricants show lower
26 wear than those tested with the ATF, having the best wear decrease with the ATF + 0.10 wt% Al₂O₃
27 nanolubricant, with reductions of 45, 57 and 78%, respectively, in diameter, depth and area of the
28 wear scar. Furthermore, by means of confocal Raman microscopy, roughness evaluation and SEM-
29 EDX of the worn tribological specimens, it can be determined that mending, tribo-sintering as well
30 as rolling mechanisms occur.

31 **Keywords:** ATF oil; electric vehicles; tribological mechanisms; friction; wear.

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33 **1. INTRODUCTION**

34 The demand for energy is growing rapidly, which raises concerns about the impact on the
35 natural environment and climate change. However, not all the energy consumed in the world goes
36 to cover this demand since, despite the significant advances in friction and wear control, friction
37 consumes about a fifth of the energy used worldwide, with transportation in the lead [1].

38 In this scenario, the electric vehicle (EV) is a promising solution, since friction losses in internal
39 combustion engine (ICE) cars roughly double those in electric cars [1]. It is known that just 20% from
40 the total fuel energy supplied to an ICE vehicle is used to push the vehicle whereas an EV uses about
41 80% from the electric energy from the grid. Thus, EV is almost four times more efficient than an ICE
42 when the electrical energy comes from renewable sources [1,2]. Even though EVs are very efficient
43 in terms of energy consumption, there is still much to be done to further improve efficiency, for
44 which the challenge is to minimize friction and wear. In addition, one of the main advantages of the
45 EVs is their great capability to reduce greenhouse gas emissions [3]. These gas reductions,
46 particularly of carbon dioxide, CO₂, strongly depend on the source of the electricity [1]. Thus, when
47 electricity comes from renewable energy sources, the CO₂ emissions of an EV are 4.5 times lower
48 than those of a combustion engine car [1]. Hence, EVs being very efficient and producing very few
49 gas emissions, the difficulties of their efficiency and resistance of EVs affect the mobile components
50 and, therefore, their tribology. Thus, tribological solutions such as new materials or optimized
51 lubricants can help to increase the driving range of EVs by reducing friction in elements such as gears
52 or bearings. In some type of EVs, the electric motor (EM) and power electronics are inside the
53 transmission housing, operating the mechanical elements of EVs at higher speeds, loads and
54 temperatures than in ICEs, and under electromagnetic conditions [4,5]. Therefore, low viscosity
55 lubricants are needed as transmission fluids of the electrified drivetrains owing to the high torque
56 and operational speeds of tribological elements in EVs [5]. These specific lubricants are called

57 electric transmission fluids (ETFs) [5,6]. By decreasing the viscosity of the oil, viscous drag and
58 viscous heating reduce and heat transfer is improved [4,7]. Nevertheless, if the lubricant viscosity is
59 reduced, boundary lubrication can appear that leads to severe tribological contact. This fact implies
60 that enhanced antiwear and antifriction lubricant properties are necessary.

61 In this vein, nanotechnology is included among the new technologies that have benefited
62 friction research [1,8]. Thus, the use of nanomaterials as lubricant additives can help to develop new
63 low-viscosity lubricants particularly adapted to the necessities of the electric drivetrains as they are
64 promising antiwear and antifriction additives and, consequently, an extended motor life could be
65 achieved under severe operational conditions and hard lubrication systems [9]. In addition, many
66 nanoparticles (NPs) are more environmentally friendly than traditional additives [10,11].

67 Nanoadditives have shown good achievements improving the antifriction and antiwear
68 capabilities of traditional lubricants [12-17]. However, the research on nanolubricants in relation to
69 the EVs' tribological requirements is scarce. Recently, Mustafa et al. [4] reviewed some low-viscosity
70 lubricants, focusing on polyalphaolefins (PAO) due to their high-performance as gear base oils and
71 hydro lubricants.

72 Given these demands, it is necessary to study potential lubricants based on low-viscosity oils
73 and nanoadditives. In this work, the main objective is to study the tribological properties of
74 aluminum oxide (Al_2O_3) nanoparticles as additives of a low-viscosity oil specific for automatic
75 transmissions, ATF. The current standard for electrified transmission lubricants are automatic
76 transmission fluids (ATFs), because some properties such as efficiency, durability, seal compatibility
77 or wide operating range of ETFs are common to those of ATFs [9]. In this work, the main objective
78 is to study the tribological properties of aluminum oxide nanoparticles (Al_2O_3 NPs) as additives of a
79 formulated ATF. According to the manufacturers, this lubricant guarantees silent operation in
80 automatic boxes, allows smooth gear to change due to the friction stability, and has good oxidation

81 resistance and thermal stability. In this vein, García Tuero et al. [3] analyzed the tribological
82 performance of a commercial ATF using a phosphonium-based ionic liquid ($[P_{6,6,6,14}][BEHP]$) as
83 additive reaching good antiwear results. Regarding the Al_2O_3 NPs, different studies have shown good
84 tribological results [18-20]. For instance, Ghalme et al. [19] analyzed the effect of the addition of
85 Al_2O_3 NPs (50 nm) as lubricant additives on the tribological performance of SAE10W40 obtaining
86 reductions in wear scar diameter and friction coefficient of 21% and 23% respectively with the
87 addition of 0.5 wt% of Al_2O_3 NPs. The authors concluded that the lubrication performance was
88 improved due to mending and ball bearing effects of Al_2O_3 NPs. Furthermore, Luo et al. [20]
89 measured the tribological properties (friction and wear) of a lubricating oil with Al_2O_3/TiO_2
90 nanocomposites modified with KH-560 (75 nm) as additive, observing a much better tribological
91 performance in comparison to lubricant base oil, being the tribological mechanism due to protective
92 film formation on the rubbed surface and sliding friction changing to rolling friction during the
93 rubbing process [18]. Moreover, Gundarneeeya and Vakharia [21] found that incorporating Al_2O_3 NPs
94 (20 nm) as lubricant additives into Avalon ISO Viscosity grade 46 oil, led to a higher maximum
95 pressure and load carrying capacity of the journal bearing in comparison to the oil without NPs.
96 Finally, Nabhan et al. [22] examined the tribological performance of hybrid MWCNTs/ Al_2O_3 NPs as
97 additives of a 10W30 engine oil. These authors found that the coefficient of friction and wear scar
98 width were enhanced by around 48% and 52%, respectively, when compared to unmodified oil.

99 In literature there are many articles on Al_2O_3 NPs as lubricant additives, obtaining good
100 tribological performance. Most of these papers primarily focus on the use of Al_2O_3 NPs in biodiesel
101 fuels to enhance engine performance [23,24] , in the design of nanofluids for minimal quantity of
102 lubrication for the metal cutting industries [25] or in automotive combustion engine oils [26].
103 However, no studies have investigated the effect of Al_2O_3 NPs as additives on low viscosity oils
104 (kinematic viscosity at 100 °C < 10 cSt) which is a key factor in the development of ETFs. Only Peña-

105 Parás et al. [18] includes a low viscosity oil, Polyalphaolefin 8 (8 cSt at 100 °C), which is suitable as
106 base oil for ETFs, but the morphology of their NPs is tubular. In addition, the article does not report
107 information about the stability of the nanolubricants, and the NPs size is not specifically indicated
108 (<50 nm). In contrast, the ATF (6.20 cSt at 100 °C) used in this work is a specially formulated low
109 viscosity oil with an additive package (antifriction, antiwear, among other additives), making it
110 exceedingly challenging to enhance its lubrication properties, and the Al₂O₃ NPs are spherical (5.5
111 nm average diameter).

112

113 **2. Material and methods**

114 *2.1. Materials*

115 ATF is a low viscosity lubricant for automatic transmissions of electric vehicles, designed to
116 protect gear and gearbox bearings from wear and corrosion, with improved oxidation stability and
117 good dielectric properties. This ATF is formulated with around 90 wt% of API G-III synthetic base oils
118 and 10 wt% of an additive package. This lubricant has a dynamic viscosity and density at 313.15 K of
119 31.21 mPa·s and 0.8271 g·cm⁻³, respectively, as well as a 152 viscosity index.

120 Gamma Al₂O₃ NPs (γ -Al₂O₃), with an average size of 5 nm and purity >99%, were supplied by
121 US Research Nanomaterials, Inc. (Houston, TX USA). γ -Al₂O₃, is one of the forms of transition
122 aluminas, which are obtained from the thermal treatment of aluminium hydroxides [27]. Scanning
123 electron microscopy (SEM, Zeiss FESEM Ultra Plus) and transmission electron microscopy (TEM, JEOL
124 1011) techniques were used to characterize the shape and size of the Al₂O₃ NPs. SEM and TEM
125 images (Fig. 1) show that these NPs present a nearly spherical shape.

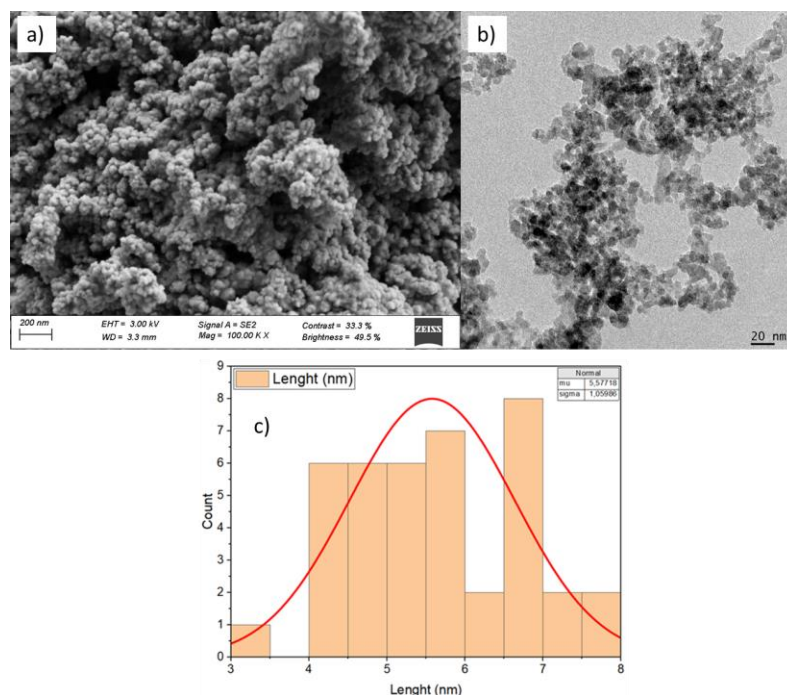


Fig. 1 SEM (a) and TEM (b) images and NPs size distribution (c) of Al₂O₃ NPs.

Regarding the nanoparticle sizes, Fig. 1c shows the size distribution of Al₂O₃ NPs (obtained with ImageJ), observing that the average sizes for the NPs are around 5.5 nm, which agrees with the information provided by the manufacturer (5 nm).

Moreover, the Al₂O₃ NPs were also characterized by infrared spectroscopy (FTIR) to identify the chemical bonds as well as functional groups of the nanoparticles (Fig. 2a). In this spectrum, the absorption peaks at 3457 and 1643 cm⁻¹ can be assigned to the stretching and bending vibrations of adsorbed water molecules and the stretching of the framework Al–OH group with the defective sites [28]. This is coherent with the fact of the chemical composition of the transition alumina is that of aluminium oxide, with a residual content of hydroxyl groups depending on the dehydroxylation of the aluminium hydroxides [27,29]. Furthermore, the observed peaks at 813, 755, and 512 cm⁻¹ for the vibrations of Al–O–Al in Fig. 2 proves the γ -form of Al₂O₃ NPs [28,29].

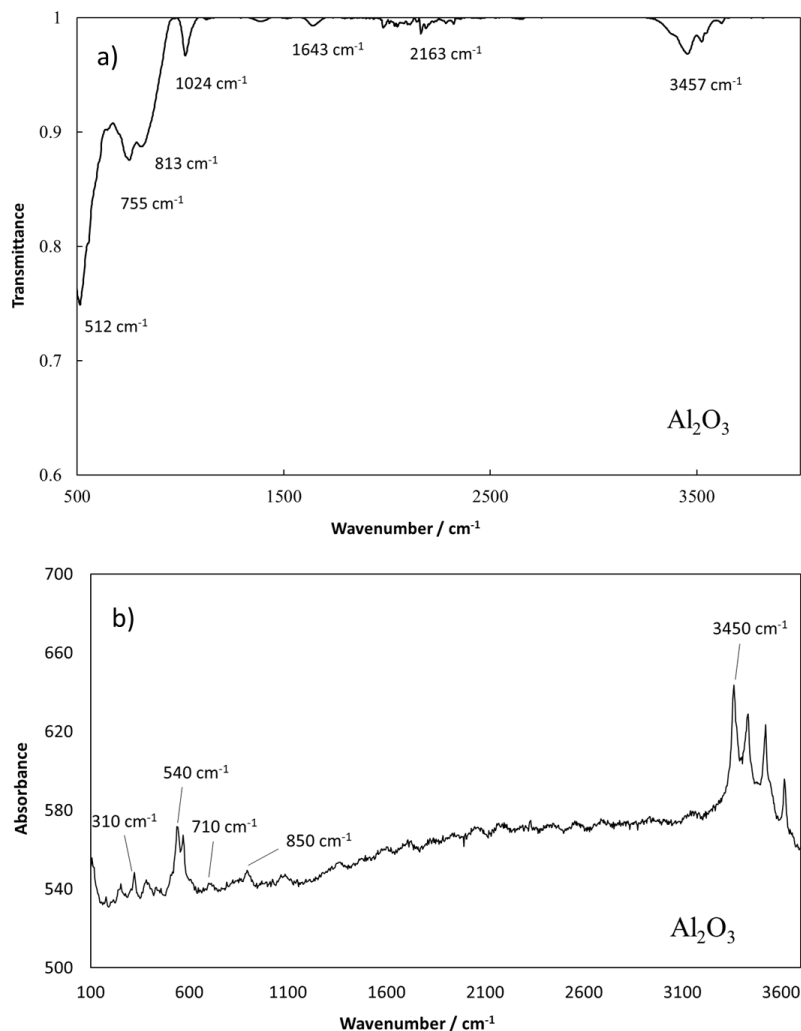


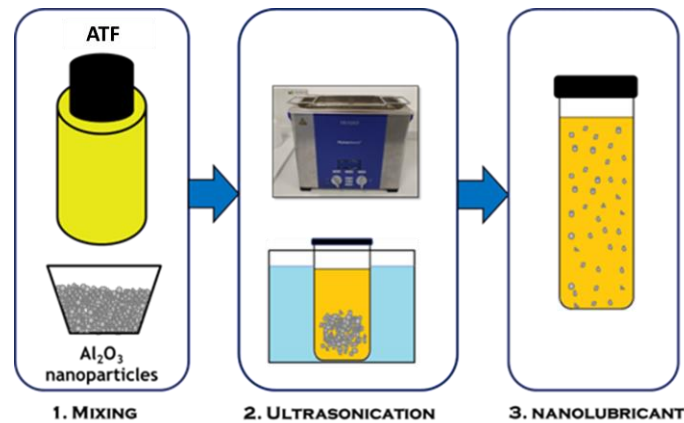
Fig. 2 FTIR (a) and Raman (b) spectra of Al₂O₃ NPs.

Regarding the Raman characterization of Al₂O₃ NPs, the spectrum (Fig. 2b) displays several bands in the region of 100–3600 cm⁻¹ with an absorbance maximum at 3450 cm⁻¹ attributed to stretching vibrations in absorbed water molecules [30]. Fig. 2b also shows that the bands have different intensities and non-elementary shapes. The sharp peaks in the 200–850 cm⁻¹ region with significant bands at 310, 415, 540, 710 and 850 cm⁻¹ correspond to vibrations of the Al–O bond in the tetrahedral structure of AlO₄ [30,31]. The presence of this structure in bulk γ -Al₂O₃ though Raman spectra was identified previously [30,31].

152 2.2. Nanodispersions formulation

153 Nanolubricants were designed by means of the traditional two-step method (Fig. 4). Dry
154 Al_2O_3 NPs were blended with ATF using a Sartorius balance (readability of 0.01 mg) to determine the
155 nanolubricant mass concentrations. Subsequently, the blend homogenization was carried out
156 through an ultrasonic bath. The sonication duration is four hours, with temperature control
157 throughout the procedure. The bath water is replaced about every hour when the temperature
158 reaches around 45 °C. Using the previous method, different masses of Al_2O_3 NPs were added in the
159 ATF to obtain the follow nanodispersions: ATF + 0.05 wt% Al_2O_3 , ATF + 0.10 wt% Al_2O_3 , ATF + 0.15
160 wt% Al_2O_3 and ATF + 0.20 wt% Al_2O_3 .

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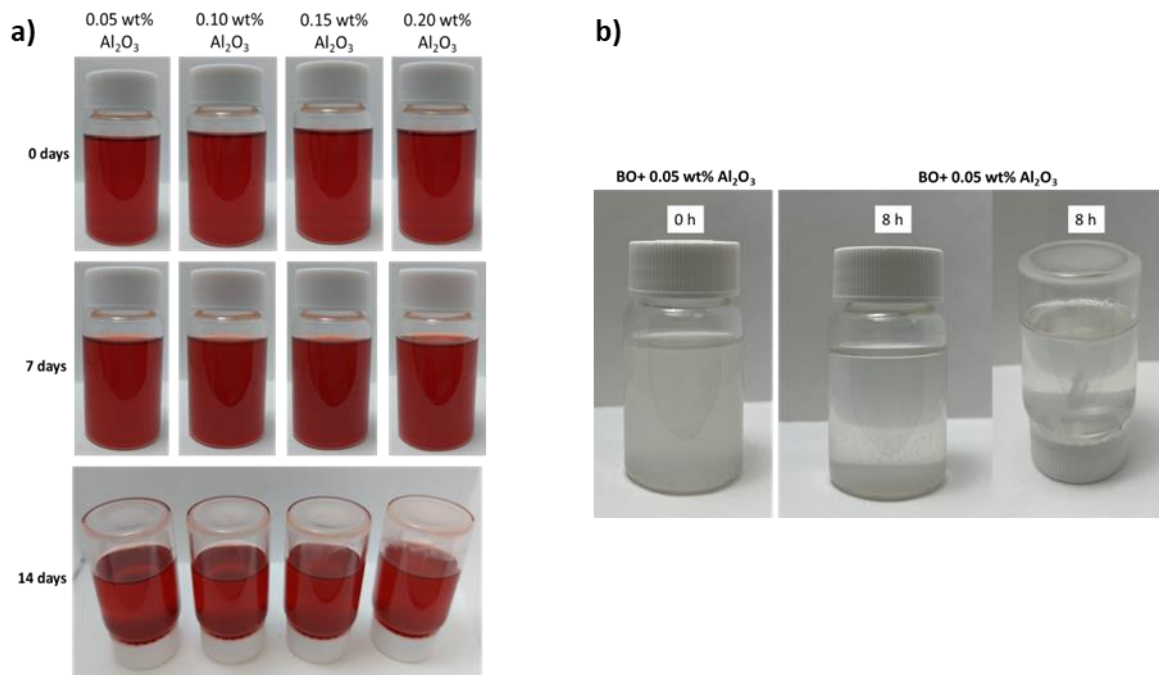


162

163 **Fig. 3.** Scheme of two-step preparation method.

164 After the preparation of nanolubricants, their stability times were evaluated through the
165 visual control, taking photos of the unaltered nanodispersions each day until the precipitation of
166 the Al_2O_3 NPs. As can be observed in Fig. 4, NPs start to sediment 14 days after the nanolubricant
167 preparation, especially in the case one with the highest concentration, 0.20 wt% Al_2O_3 NPs. This
168 time is much longer than the time needed to carry out the tribological tests: friction and wear
169 analysis. For comparison, we have included in Fig. 4 photos of the nanodispersion containing the

170 base oil of the ATF (API G-III) and 0.05 wt% Al_2O_3 NPs. As can be observed, sedimentation of the
171 Al_2O_3 NPs is evident eight hours after its preparation.



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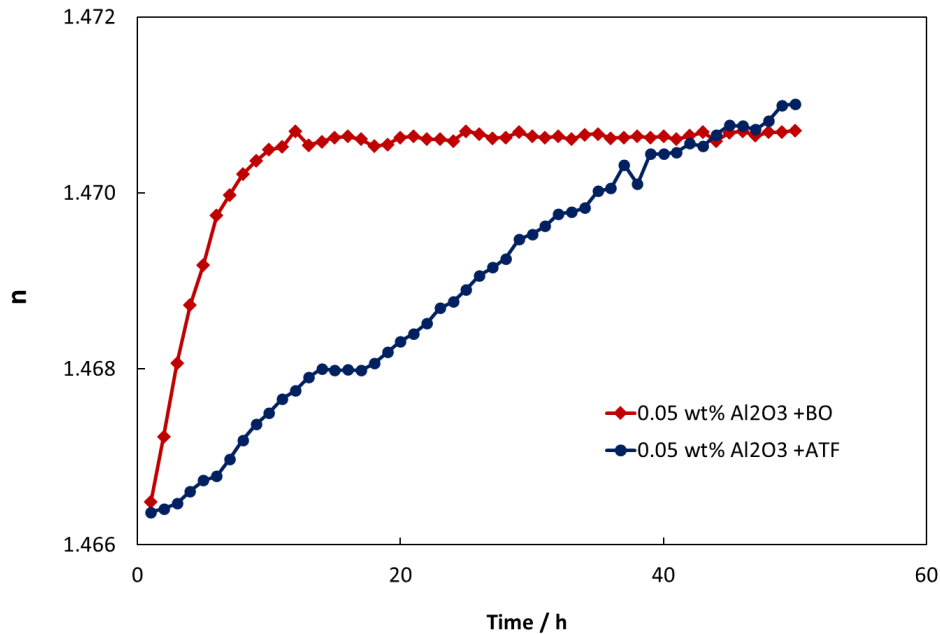
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174 **Fig. 4.** Visual observation of a) ATF nanolubricants containing Al_2O_3 NPs, b) base oil (API G-III) of
175 ATF with 0.05 wt% Al_2O_3 .

176

177 Furthermore, to complete the stability analysis of the nanolubricants, the refractive index
178 for the 0.05 wt% Al_2O_3 nanolubricant (based on ATF) at 298.15 K was the measured together with
179 that for the dispersion of Base oil API G-III + 0.05 wt% Al_2O_3 to know if this additive package of the
180 ATF plays a role in achieving a good stability. For this aim, a Mettler Toledo RA-510M refractometer
181 was used. Fig. 5 displays the evolution of the refractive index for both Al_2O_3 nanolubricants with an
182 NPs concentration of 0.05 wt%. After the initial ten hours, the refractive index of the Base oil + 0.05
183 wt% Al_2O_3 nanolubricant reaches a constant value, i.e., the NPs are fully sedimented. However, the
184 NPs sedimentation in the ATF + 0.05 wt% Al_2O_3 nanolubricant occurs gradually. Specifically, for the
185 first nanolubricant, refractive index increased by 0.27% after 10 hours, while for the ATF

186 nanolubricant, the refractive index only increased by 0.07%. Therefore, it is suggested that the
187 additive package in ATF lubricants has a positive effect on the stability of Al₂O₃ nanolubricants.
188



189

190 **Fig. 5** Refractive index evolution for Al₂O₃ nanolubricants.

191

192

2.3. Friction tests and wear evaluation

193

194 Tribological studies with ATF and nanolubricants were performed using a T-PTD200 tribology cell

195 coupled to a rheometer MCR 302, Anton Paar (Graz, Austria), in a ball-on-three pins geometry. The

196 steel ball is fixed in a rotating tube that is driven by the rheometer. The pins form an angle of 135°

197 with respect to the tube and are located inside a thermostated container. In each tribological test,

198 the ball rotates on the three pins under a force applied by the rheometer. This axial force is

199 transferred, giving rise to three normal forces perpendicular to each pin surface. More details can

200 be found in [32-34]. For this work, cylindrical pins of 6x6 mm and 12.7 mm diameter balls both made

201 of hardened 100Cr6 steel and with a hardness of 62-66 Rockwell C, were used. The tests were

performed at 213 rpm rotational speed (0.1 m s⁻¹ at the contact points) and 20 N axial load, equally

202 distributed on the three pins (9.43 N on each pin) implying a maximum Hertzian pressure of 1.1 GPa,
203 a duration of 3400 s and a temperature of 393.15 K. As the performance of electric motors improves,
204 the power density increases, with a consequent increase in heat generation in the coil, and the
205 resulting increase in temperature. In addition, to avoid demagnetization, the temperature must be
206 below 423.15 K [35], thus effective motor cooling is crucial. As a result, to meet the cooling
207 requirements, different approaches have been proposed. Among them, for high-power motors,
208 liquid cooling is one of the most promising cooling techniques. Thus, the temperature was chosen
209 393.15 K according with previous results on the literature [36], in which the maximum motor
210 temperature is close. More information regarding this tribological device can be found in a previous
211 work [34].

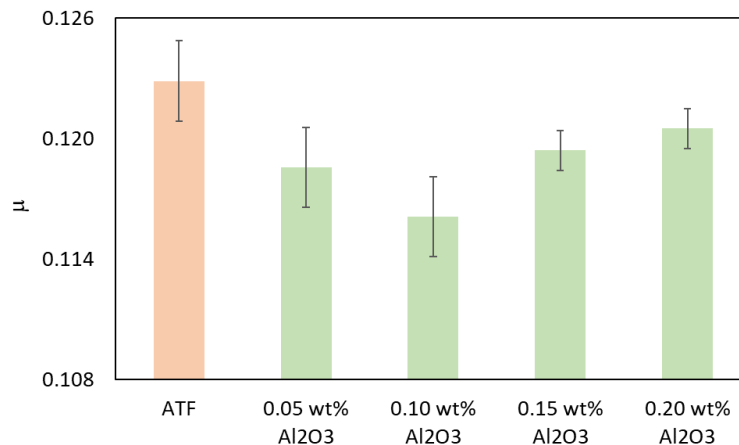
212 Once the friction experiments have been carried out with each of the formulated Al_2O_3
213 nanolubricants and the ATF (at least 3 replicates of each one), the wear caused on each of the pins
214 is evaluated to establish which of the nanolubricants has the best anti-wear performance. For this
215 purpose, a 3D profiler (Sensofar S neox) was utilized to quantify the generated wear using different
216 wear parameters: wear scar diameter (WSD), wear track depth (WTD) and worn area. The S neox
217 3D profiler was calibrated and verified by the manufacturer, following the ISO 25178 standard.
218 These wear parameters were evaluated on the three pins tested with each nanolubricant to obtain
219 suitable average values. The 3D profiler was also used to determine the wear track roughness (R_a
220 and R_q) of the worn pins to illustrate the antiwear ability of each nanolubricant. For this task,
221 ISO4287 standard was used, making use of a Gaussian filter (cut-off: 0.08 mm wavelength). In
222 addition, SEM-EDX microscopy was also used to observe the worn surfaces of pins tested with all
223 the formulated nanolubricants and the ATF and to obtain information on chemical composition of
224 the worn track. Finally, a confocal Raman microscope from WITec was used to examine the worn

225 surfaces of pins and reveal information about the NPs in the worn track and the possible tribological
226 mechanisms that may have taken place.

227 3. Results and analysis

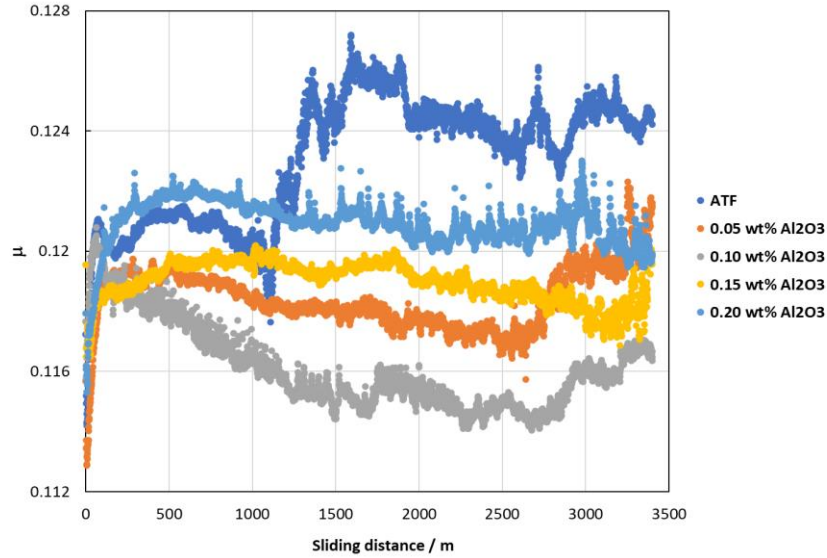
228 3.1. Friction and wear results

229 Fig. 6 and Table 1 present the mean friction coefficients (μ) for the ATF and base oil and its
230 nanolubricants containing Al_2O_3 NPs at a temperature of 120 °C. It is clearly observed that the four
231 nanolubricants have friction coefficients slightly lower than that of the ATF. In particular, the lowest
232 μ value is obtained with the nanolubricant ATF + 0.10 wt% Al_2O_3 NPs, i.e., the optimum
233 concentration of the Al_2O_3 NPs is 0.10 wt%, being the friction reduction 6 %. Additionally, Fig. 7
234 which displays the evolution of friction coefficient values during time, also shows that the
235 nanolubricant ATF + 0.10 wt% Al_2O_3 NPs present the lowest friction results.



236

237 **Fig. 6** Comparison between the average friction coefficients (μ) found with ATF and its
238 nanolubricants containing Al_2O_3 NPs.



239

240 **Fig. 7** Evolution of friction coefficient values for ATF and its nanolubricants containing Al_2O_3 NPs
 241 with the sliding distance at 120 °C.

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243

244 **Table 1**

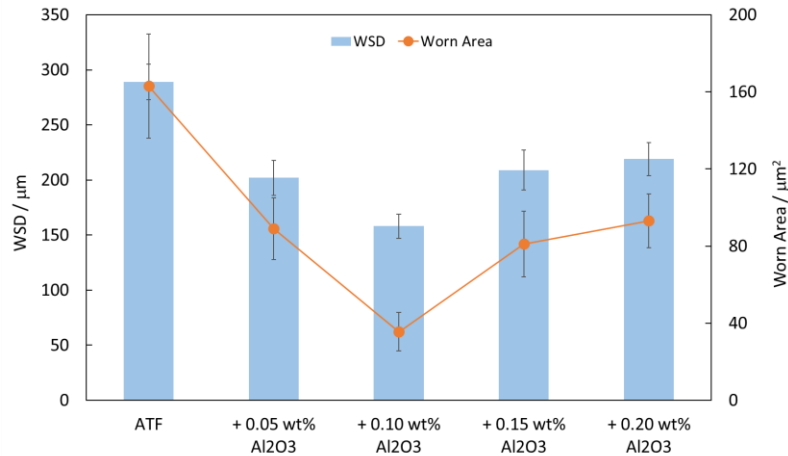
245 Average friction coefficients, μ , and average wear parameters with their standard deviations for
 246 the tested ATF lubricants.

Lubricant	μ	σ	WSD/ μm	$\sigma/\mu\text{m}$	WTD/ μm	$\sigma/\mu\text{m}$	Area/ μm^2	$\sigma/\mu\text{m}^2$
ATF	0.123	0.002	289	16	0.82	0.09	163	27
+ 0.05 wt% Al_2O_3 NPs	0.119	0.002	202	16	0.49	0.07	89.1	16
+ 0.10 wt% Al_2O_3 NPs	0.116	0.002	158	11	0.35	0.04	35.5	10
+ 0.15 wt% Al_2O_3 NPs	0.120	0.001	209	18	0.49	0.08	81.1	17
+ 0.20 wt% Al_2O_3 NPs	0.121	0.001	219	15	0.35	0.14	93.1	14

247

248 As indicated previously, the wear generated during the tribological experiments was
 249 analyzed by means of the wear track parameters: diameter, depth and worn area. The values of
 250 WSD, WTD and worn area observed on the worn pins lubricated with the tested ATF dispersions are
 251 summarized in Table 1. All the wear parameters analyzed (Fig. 8 and Table 1) indicate that using the
 252 nanolubricants with Al_2O_3 NPs, the wear created during friction tests is much lower than that
 253 obtained with ATF. As with antifriction performance, it should be emphasized that the mass

254 concentration of additives significantly affects antiwear performance. The wear results obtained
255 have a similar trend with the values obtained for the friction coefficient (Fig. 6) Thus, the largest
256 wear reductions in diameter, depth and area were also found with the nanolubricant ATF+ 0.10 wt%
257 Al₂O₃ NPs, with reductions of 45, 57 and 78%, respectively. This phenomenon may be due to at very
258 low concentrations (0.05 wt%) the content of NPs is not sufficient and when the concentration of
259 additives is higher (0.15 and 0.20 wt%), NPs are more likely to agglomerate during friction tests,
260 making it difficult for the NPs to enter the tribological contact region, causing poorer lubrication
261 performance. Optimal concentrations for the tribological properties of many nanodispersions have
262 been found previously [24,37-40], including some containing Al₂O₃ NPs [24,37]. For another
263 spherical nanomaterials (ZrO₂/SiO₂ composite NPs) Zheng et al. [41] explain this phenomenon
264 indicating that when less nanoparticles than the corresponding to the optimal concentration were
265 added the friction zone could not be completely filled. Therefore, the ball bearing effect cannot play,
266 and the anti-friction effect was not obvious. When more NPs were added the NPs were sintered into
267 blocks by friction. Therefore, larger nanoparticles (aggregates) as impurities scratched the friction
268 surface and the friction coefficient increased. Therefore, only when the added amounts of
269 nanoparticles were in the optimal concentration range, the friction-reducing effect is better.
270 Furthermore, for non-spherical NPs as reduced graphene oxide nanosheets, this phenomenon also
271 occurs, which is explained by the existence of a point of saturation for the filling of the nanosheets
272 into the friction zone [38].

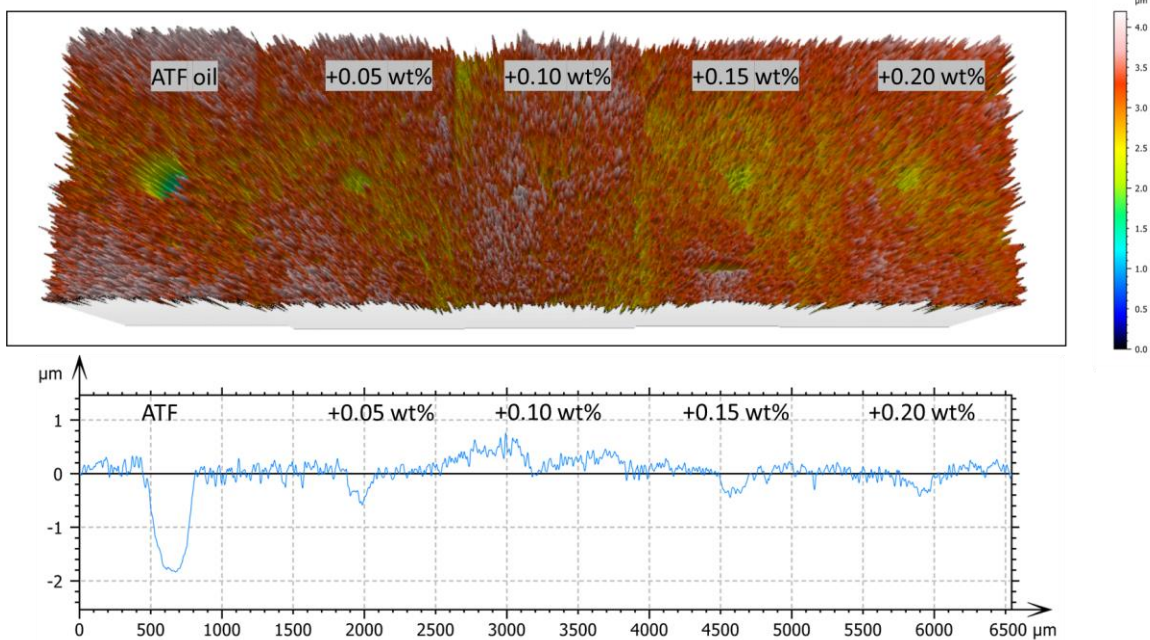


273

274 **Fig. 8** Mean WSD and worn areas achieved with ATF and with its nanolubricants with Al_2O_3 NPs.

275 A comparison of the cross-sectional profiles and a 3D mapping of worn surfaces lubricated
 276 with ATF and its nanolubricants with Al_2O_3 NPs is presented in Fig. 9, in where the significant wear
 277 reductions can be observed when nanolubricants are used instead ATF. Therefore, with low
 278 concentrations of Al_2O_3 nanoadditives considerable antiwear improvements can be observed. These
 279 results are very important because the ATF is a formulated oil with an additive package (antifriction,
 280 antiwear, among others) and therefore it is very difficult to improve its lubrication properties as it
 281 was done in this work.

282



283

284 **Fig. 9** 3D Surface topography and cross-sectional profiles comparison of worn surfaces lubricated
 285 with ATF and with its nanolubricants containing Al₂O₃ NPs.
 286

287 *3.2. Tribological mechanisms discussion*

288 To understand the tribological mechanism that could describe the better antifriction-
 289 antiwear behavior of nanolubricants compared to ATF, roughness analysis, Raman mapping, SEM
 290 microscopy and EDX analysis were carried out on the worn surfaces lubricated with the ATF and the
 291 nanolubricants with Al₂O₃ NPs.

292 Measurements of roughness (Ra and Rq) on worn pins reveal that the worn surfaces
 293 lubricated with nanolubricants containing Al₂O₃ NPs present lower roughness compared to those
 294 lubricated with ATF (Table 2). Thus, a Ra value of 18.8 nm was reached for the worn pins lubricated
 295 with ATF whereas for the those tested with the nanolubricant ATF + 0.10 wt% Al₂O₃ NPs the lowest
 296 Ra value (11.9 nm) was reached implying a roughness reduction of 37%. These roughness results
 297 showed that due to the presence of the NPs in the contact, a more regular surface is found after
 298 tribological tests.

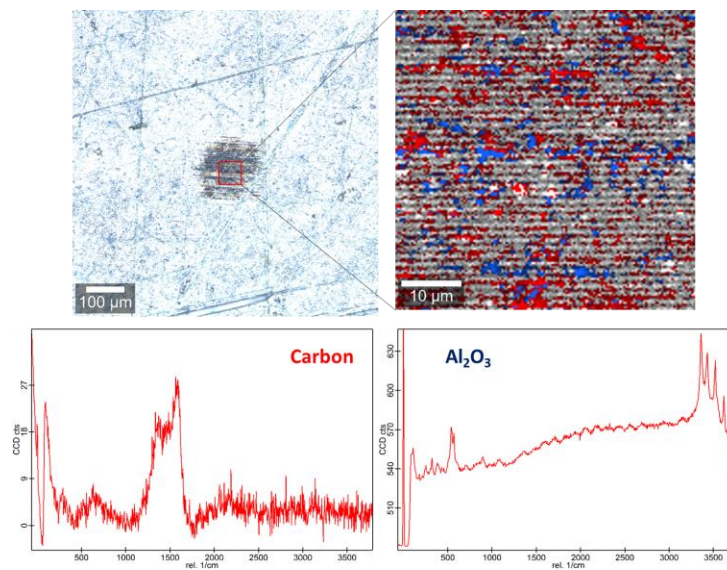
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300 **Table 2**
 301 Mean roughness values, Ra and Rq with their uncertainties, σ , in worn pins tested with ATF
 302 lubricants.
 303

Lubricant	Ra/nm	σ	Rq/ nm	σ
ATF	18.8	1.8	21.3	2.2
+ 0.05 wt% Al ₂ O ₃ NPs	16.5	1.5	17.8	1.4
+ 0.10 wt% Al ₂ O ₃ NPs	11.9	1.1	12.7	1.3
+ 0.15 wt% Al ₂ O ₃ NPs	13.2	1.3	15.0	1.2
+ 0.20 wt% Al ₂ O ₃ NPs	16.4	1.3	18.2	1.5

304

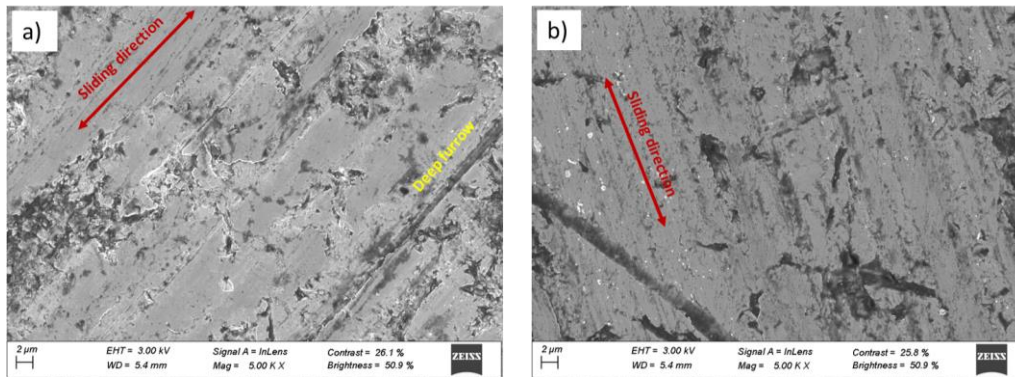
305 Elemental mapping and Raman images of the worn surfaces lubricated with the optimal
 306 nanolubricant ATF + 0.10 wt% Al₂O₃ NPs were recorded with the confocal Raman microscope to gain
 307 insight into the role of NPs in decreasing the worn surface on pins. The Raman spectrum of the Al₂O₃
 308 NPs (Fig. 3) match those obtained by analyzing the worn tracks lubricated with the optimum
 309 nanolubricant. Thus, Fig. 10 displays a significant presence of ATF (red color) and significant spots
 310 due to Al₂O₃ NPs (blue color) in the mapping of the worn surface lubricated with the optimum
 311 nanolubricant. The average size of the Al₂O₃ NPs is very small (5 nm), so they can easily come into
 312 the sliding contact.



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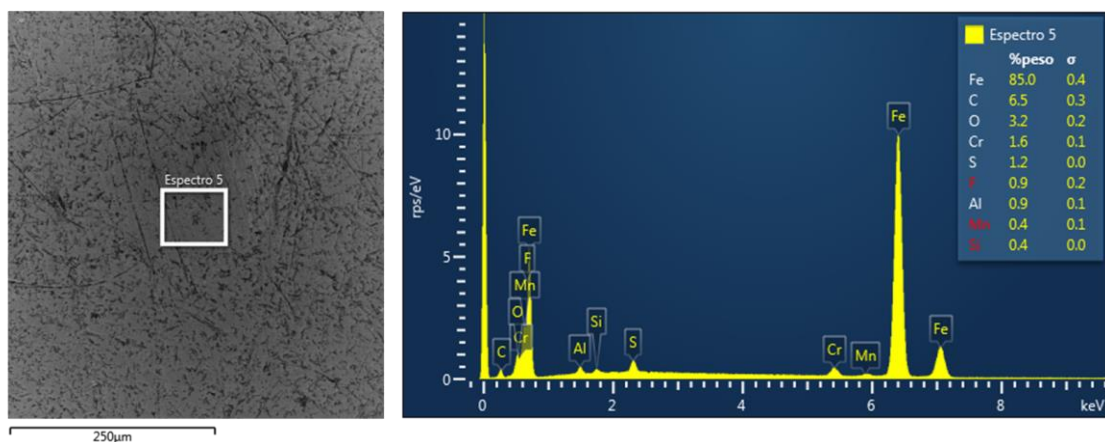
314 **Fig. 10** Raman spectra and elemental mapping of the worn surface tested with the optimal Al₂O₃
 315 nanolubricant.

316
317 Regarding the SEM analysis, Fig. 11 shows the worn tracks of the tested pins lubricated with the ATF
318 (Fig. 11a) or with the optimal nanolubricant ATF + 0.10 wt% Al₂O₃ NPs (Fig. 11b) at 5000x. It can be
319 seen that for the worn track lubricated with the optimal nanolubricant the surface is smoother,
320 specifically in Fig. 11a there is a deep elongated groove along the sliding direction, which can be
321 formed as a product of abrasive wear. This agrees with the roughness measurements (Table 2).



322
323 **Fig. 11** SEM micrographs of worn surfaces of the tested pins lubricated with a) ATF and b) ATF +
324 0.10 wt% Al₂O₃ NPs.

325
326 The presence of Al₂O₃ NPs on the worn surface of a tested pin lubricated with the optimal
327 nanolubricant was analyzed with EDX, as shown in Fig. 12. The signals detected indicate that Al₂O₃
328 NPs are deposited and filled into the plow on the worn surface during the tribological tests, with an
329 aluminum (Al) content of 0.9 wt%. Therefore, this fact suggests mending effect where the surface
330 scratch is smoother, and the plow furrow is shallower, reducing both the friction coefficient and
331 wear.



332

333 **Fig. 12** EDX spectrum of the worn surface of a tested pin lubricated with the optimal
 334 nanolubricant.
 335

336 Finally, due to the high temperature produced by friction and taking into account that tribological
 337 tests are performed at 120 °C, the Al₂O₃ NPs can be chemically adsorbed on the worn surface
 338 through hydroxyl groups [42,43] and tribo-sinter to the worn surface, reducing the metal to metal
 339 contact [4,44]. Furthermore, Al₂O₃ NPs are mainly spherical (Fig. 1); consequently, the NPs can play
 340 the role of ball bearings (known as rolling effect) that avoid the direct contact of the components of
 341 the tribological pair, and the sliding friction is shifted to rolling friction improving the extreme
 342 pressure behavior and the carrying capability of the nanolubricant [37]. Based on the obtained
 343 results, Al₂O₃ NPs used as additives of ATF play an improved anti-friction and anti-wear effect.

344 Thus, considering all the characterization for describe the possible tribological mechanisms
 345 (Roughness, Raman Mapping and SEM-EDX) it can be summarized that these mechanisms can be
 346 mending, the tribo-sintering and the rolling effect due to the presence of Al₂O₃ NPs. Therefore, the
 347 important improvement achieved mainly in the reduction of wear with respect to the base oil is
 348 supported by the appearance of these three important tribological mechanisms.

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351 **4. Conclusions**

352 Four low viscosity nanolubricants, which are based on a commercial ATF and containing up to 0.20
353 wt% of Al₂O₃ NPs, were formulated, achieving temporal stabilities of two weeks. The addition of
354 these NPs enhances the tribological properties of the commercial ATF. Specifically, the best
355 enhancements were found with the ATF + 0.10 wt% of Al₂O₃ nanolubricant: the reductions of the
356 diameter, depth, and transversal area of the worn scar compared to the commercial ATF are 45, 57
357 and 78%, respectively. For this reason, 0.10 wt% of Al₂O₃ NPs is considered the optimum
358 concentration. The lubrication mechanism can be primarily explained through the mending and the
359 tribo-sintering effects, as revealed by Raman, roughness and SEM-EDX studies of worn surfaces,
360 apart from the rolling effect resulting from the NPs' spherical shape. It is not obvious to evidence
361 this last effect. This result is highly significant because a) the ATF oil is specifically formulated with
362 additive packages for its use as an automatic transmission fluid, and b) it contains optimized anti-
363 wear and anti-friction additives, which have positive synergies with nanomaterials.

364

365 **CRedit authorship contribution statement**

366 **José M. Liñeira del Río:** Writing - review & editing, Writing - original draft, Methodology,
367 Investigation, Conceptualization. **Enriqueta R. López:** Writing - review & editing, validation,
368 supervision, formal analysis **Josefa Fernández:** Writing - review & editing, Validation, Supervision,
369 Project administration, Funding acquisition, Conceptualization.

370

371 **Declaration of competing interest**

372 None

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