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Perfluorotetradecanoic Acid as an Additive for Friction Reduction in Full-Film EHD Contacts: The Role of Functional Group, Base Oil Polarity, Additive Concentration and Contact Pressure

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Abstract: Perfluorinated tetradecanoic acid was added as an additive to a base oil and tested under full film elastohydrodynamic (EHD) contact conditions between a steel ball and a steel disc. By varying key performance parameters, we aimed to assess the feasibility and limitations of perfluorinated carboxylic acids in reducing friction in lubricated contacts. The results demonstrate that the tested perfluorinated additive is effective in reducing friction when blended with a non-polar synthetic poly-alpha-olefin oil. However, no significant friction reduction was observed when the same additive was used in a slightly polar synthetic ester. The carboxylic acid functional group plays a crucial role in the observed friction-reducing effect. Adjusting the additive concentration further plays an important role in reducing friction. A concentration of at least 0.35 wt.% is required to achieve a notable friction reduction of approximately 10%. Increasing the concentration beyond this threshold continues to improve the friction-reducing effect. Conversely, increasing the contact pressure has a detrimental impact on friction reduction. The greatest reduction in friction—over 20% compared to the base oil—was achieved at the lowest contact pressure tested (0.69 GPa).

Keywords: additives; EHD contact; base oil; concentration; pressure

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1. Introduction

Additives are an essential component of every lubricant, as they significantly enhance lubricant performance, especially under demanding conditions where the base oil alone, due to its inherent physical properties, cannot effectively separate the contacting surfaces. In such scenarios, friction- and wear-reducing additives play a crucial role. Traditionally, it is believed that these additives function primarily under severe conditions by forming boundary films on the surface, thereby modifying friction and reducing wear. However, recent studies have shown that these additives can also influence friction in the regime of a full lubricating film, where the contact surfaces are completely separated under elastohydrodynamic (EHD) conditions [1]. Notably, it has been demonstrated that significant reductions in friction can be achieved through the appropriate selection of such additives [1].

The primary mechanism proposed for friction reduction in the full-film lubrication regime is interfacial slip between the surface and the lubricant. This effect is attributed to the modified steel surface becoming more slippery due to the adsorption of specific

additives. These additives considerably lower the surface energy, thus weakening the interactions between the lubricant and the solid surface [1]. The correlation between a low surface energy, reduced wettability, and lower friction has been confirmed in numerous studies involving a variety of materials [2–5].

A consistent finding among these studies is that the largest reductions in friction are achieved when the surface exhibits an extremely low surface energy, comparable to that of polytetrafluoroethylene (PTFE or Teflon)—a material well-known for its non-stick properties. Several investigations have shown that achieving such low surface energy requires the presence of fluorine in the surface materials [6].

Perfluorinated carboxylic acids have bene used in several studies and have been shown to provide a low surface energy surface with poor wetting [7–9], and in addition, they also reduce the coefficient of friction in boundary lubrication [7,10] as well as in full-film low-pressure lubrication conditions [8,9,11].

In our recent study, we have shown that these perfluorinated carboxylic acids can also reduce friction in full-film EHD contacts and that longer chain lengths of more than 12C atoms are needed to achieve significant friction reduction [12]. In that study, it was also shown that the reduction in friction is attributed to the formation of low-surface-energy slippery layers of the perfluorinated carboxylic acids used as additives.

Based on these established findings, this study extends our previous study, whose focus was on different chain lengths of perfluorinated carboxylic acids and corresponding mechanism for friction reduction [12]. This study thus elaborates upon the use of perfluorinated carboxylic acids as additives to investigate the limitations of this approach and how variation in testing parameters can be used to maximize the friction-reducing effect. To prove this, we varied several other key performance parameters, namely the functional group, base oil polarity, additive concentration, and contact pressure.

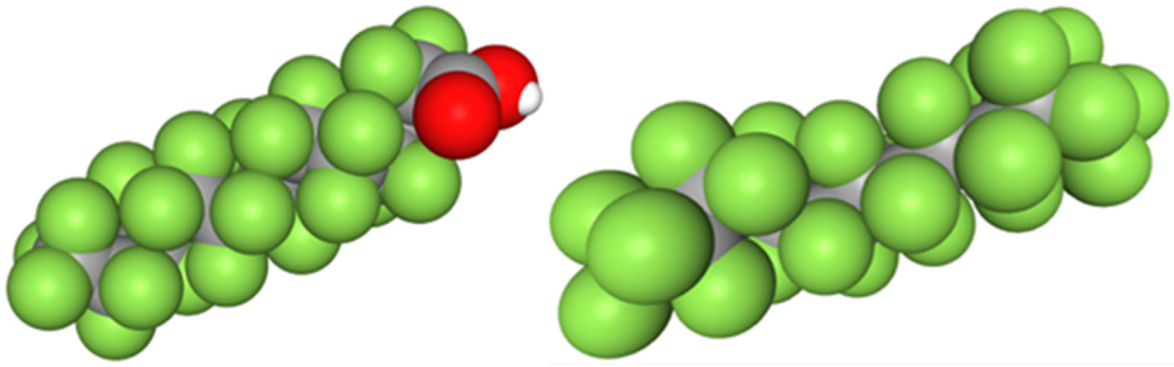
2. Materials and Methods

2.1. Specimens and Lubricants

In this study, we used standard steel specimens made of AISI 52100 (DIN 100Cr6) bearing steel—specifically, an MTM ball with a diameter of 19.05 mm and a surface roughness of R_a = (0.020 ± 0.005) µm and R_q = (0.025 ± 0.005) µm, and an MTM disc with a diameter of 46 mm, thickness of 6 mm and a surface roughness of R_a = (0.008 ± 0.002) µm and R_q = (0.012 ± 0.002) µm. The mechanical properties of both specimens, which are essential for determining the lubrication regimes, are as follows: Young's modulus E = 210 GPa and Poisson's ratio ν = 0.3. The MTM samples were provided by PCS Instruments, London, UK.

The primary lubricant used was a synthetic poly-alpha-olefin oil (denoted as PAO), with a dynamic viscosity η_0 of 46.6 mPas at 25 °C, a density ρ of 814 kg/m^3, and a pressure-viscosity coefficient α of 15.3 GPa^{-1}. In the selected tests, a synthetic monopentaerythritol tetraester (further denoted as the ester), was also used. This ester has a dynamic viscosity η_0 of 43.2 mPas at 25 °C, a density ρ of 984 kg/m^3, and a pressure–viscosity coefficient α of 20.0 GPa^{-1}.

As the main additive, we used perfluorinated tetradecanoic acid (denoted as CF13-COOH), as shown in Figure 1a. The additive was supplied by Gute Chemie abcr GmbH, Karlsruhe, Germany. Unless explicitly stated otherwise, this additive was mixed into both base oils (PAO and Ester) at a concentration of 1.00 wt.%. Additionally, we varied the concentration of CF13-COOH in PAO oil from 0.10 wt.% to 2.00 wt.% to study its effect on friction reduction.



(**a**) (**b**)

Figure 1. Additives used: (**a**) unbranched perfluorinated tetradecanoic acid (CF13-COOH) and (**b**) perfluorinated n-tetradecane (CF14). Colours: grey (carbon), green (fluorine), red (oxygen), and white (hydrogen).

For comparison, we also tested perfluorinated n-tetradecane ($C_{14}F_{30}$, further denoted as CF14) (Figure 1b), which is structurally similar to CF13-COOH, and also has 14-carbon-atoms-long chain, but lacks a terminal carboxylic acid functional group. This additive was also blended into the PAO base oil at 1.00 wt.%. Both additives used in the study are shown in Figure 1. As seen, the only difference between them is the presence or absence of the acid functional group.

The base oil–additive mixtures were stirred with a magnetic mixer at 100 °C for 2 h to completely dissolve them in the base oil. They were then cooled to 25 °C and stirred for an additional 2 h before their use in the tests to ensure homogeneity, because at the testing temperature the additive would not fully dissolve in the oils. The viscosity of the oil–additive mixtures increased slightly compared to base oils. This effect was most pronounced for the highest additive concentrations, where the dynamic viscosity reached up to 48.2 mPas for PAO oil and up to 44.5 mPas for the ester.

2.2. Tribological Tests

Tribological tests were performed using an MTM test rig (PCS Instruments, London, UK) with an MTM steel ball–MTM steel disc contact pair. Prior to testing, both contact surfaces were thoroughly cleaned with 98% ethanol and dried using hot air. A total volume of 10 mL of either the base oil or the additive-containing mixture was introduced into the contact area before the start of each test.

All tribological tests were conducted under constant operating conditions, with the following constant parameters: entrainment speed U_e = 2500 mm/s, slide-to-roll ratio SRR = 50%, temperature T = 25 ± 2 °C, and test duration 15 min. The only variable parameter was the normal load W, for which three different values were applied:

- 12 N, corresponding to a Hertzian contact pressure of 0.69 GPa;
- 35 N, corresponding to 0.98 GPa;
- And 75 N, corresponding to 1.27 GPa.

During each test, the friction coefficient was continuously recorded in the contact zone. To determine the lubrication regime, the Tallian parameter λ [13] was calculated using Equation (1), based on the minimum elastohydrodynamic (EHD) film thickness h_0, which was determined using the Hamrock–Dowson equation [14], as shown in Equation (2). The

E' and R' in Equation (2) refers to the reduced Young's modulus and a reduced radius of curvature, respectively. The k refers to the ellipticity parameter.

$$\lambda = \frac{h_0}{\sqrt{{R_{q,ball}}^2 + {R_{q,disc}}^2}} \quad (1)$$

$$\frac{h_0}{R'} = 3.63\left(\frac{U_e\eta_0}{E'R'}\right)^{0.68}(\alpha E')^{0.49}\left(\frac{W}{E'R'^2}\right)^{-0.073}\left(1 - e^{-0.68k}\right) \quad (2)$$

The minimum film thickness calculated for the PAO oil was 189 nm and for the ester it was 204 nm, resulting in a Tallian parameter λ of 6.8 for PAO oil and 7.4 for the ester, which confirms that the contact was completely separated by a lubricating film, because if λ > 4, the contact is considered to be in a full-film regime. The minimum film thickness for the oil–additive mixtures with the highest additive concentration was slightly higher than that for the base oil due to the slightly higher dynamic viscosity, namely 193 nm for the PAO oil and 208 nm for the ester.

Tribological tests were carried out in four sequential steps, as illustrated in Figure 2.

Step of study	Lubricant	Test conditions
1. Step: Role of additive polar group	▪ Base synthetic PAO + 1 wt.% CF13-COOH ▪ Base synthetic PAO + 1 wt.% CF14	U_e = 2500 mm/s, *SRR* = 50 % *T* = 25±2 °C *W* = 35 N (0.98 GPa)
2. Step: Role of base oil polarity	▪ Base synthetic PAO + 1 wt.% CF13-COOH ▪ Base synthetic Ester + 1 wt.% CF13-COOH	U_e = 2500 mm/s, *SRR* = 50 % *T* = 25±2 °C *W* = 35 N (0.98 GPa)
3. Step: Role of additive concentration	Base synthetic PAO with wt. % concentrations of CF13-COOH: 0.10; 0.20; 0.35; 0.50; 0.75; 1.00; 2.00	U_e = 2500 mm/s, *SRR* = 50 % *T* = 25±2 °C *W* = 35 N (0.98 GPa)
4. Step: Role of contact pressure	▪ Base synthetic PAO + 1 wt.% CF13-COOH	U_e = 2500 mm/s, *SRR* = 50 % *T* = 25±2 °C *W* = 12 N (0.69 GPa) *W* = 35 N (0.98 GPa) *W* = 75 N (1.27 GPa)

Figure 2. Test matrix with a variety of lubricants and test parameters.

- Step 1: We investigated the effect of the carboxylic acid functional group on friction reduction. In this phase, both additives—CF13-COOH (with the acid group) and CF14 (without the acid group)—were tested under identical conditions, with a constant additive concentration and testing parameters.
- Step 2: We examined the influence of the base oil polarity by testing two different base oils, both containing 1 wt.% of the CF13-COOH additive. All other testing conditions and the additive concentration were kept constant.
- Step 3: We studied the effect of additive concentration by varying the amount of CF13-COOH in the PAO base oil. The testing parameters remained unchanged throughout this step.

- Step 4: We explored the influence of contact pressure by varying the normal load applied, thereby generating different Hertzian contact pressures, while keeping all other test conditions constant. The CF13-COOH additive mixed in 1 wt.% in PAO oil was used in this step.

Each test combination shown in Figure 2 was repeated at least twice. In the Section 3 (Results) the representative evolution of the coefficient of friction is shown, while in the discussion, in which the results are summarized, the standard deviations are reported to illustrate the significance of the differences in the coefficient of friction between the influencing parameters investigated.

3. Results

The results are presented in the following four sub-sections corresponding to the four experimental test matrix steps shown in Figure 2.

3.1. Role of Additive Polar Group

The results of Step 1, where both additives (CF13-COOH and CF14) were tested, are shown in Figure 3. It can be observed that both the base PAO oil and the mixture with the CF14 additive exhibit a similar friction coefficient profile throughout the 15 min test, with the final coefficient of friction stabilizing at approximately 0.037.

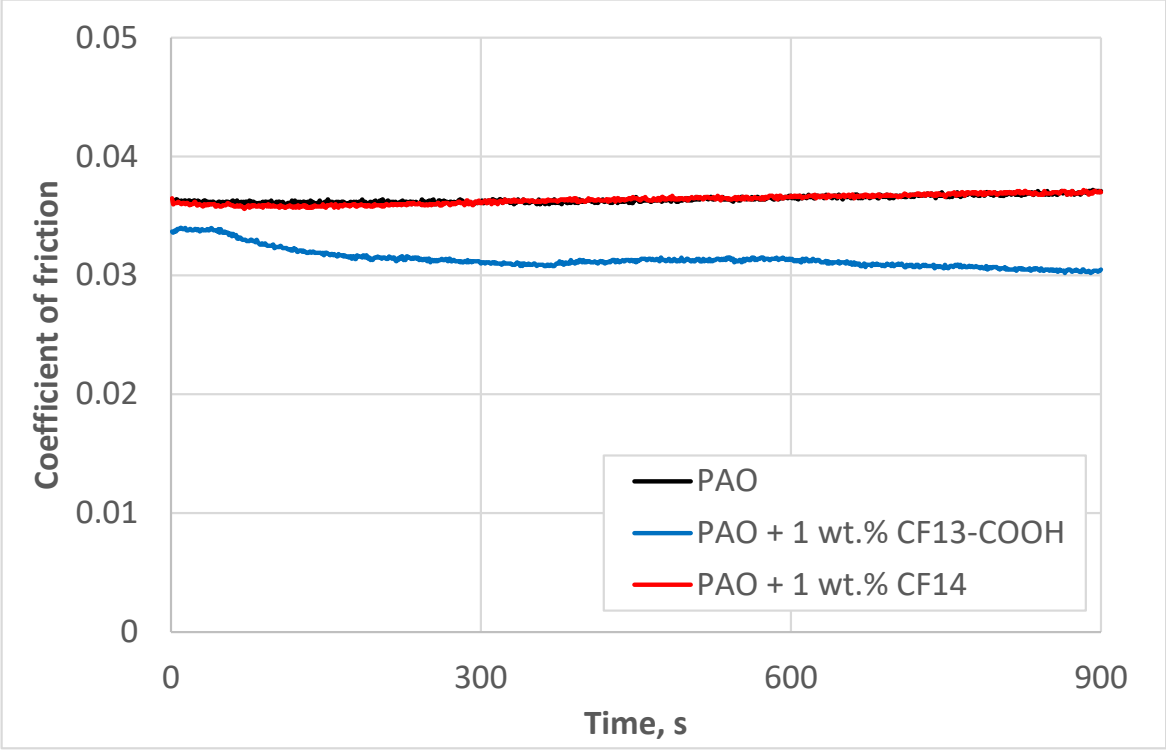


Figure 3. Coefficient of friction evolution for the base synthetic PAO oil and two mixtures of this oil, one with 1 wt.% of CF13-COOH (with functional polar group) and the other with a CF14 (without functional additive group) additive.

In contrast, the mixture with the CF13-COOH additive shows a significantly lower friction coefficient right from the beginning of the test. During the first 200 s, the coefficient of friction drops rapidly, followed by slight fluctuations, eventually stabilizing at around 0.030 after 15 min (900 s).

3.2. Role of Base Oil Polarity

The results from Step 2, where we investigated the effect of the base oil polarity, to which CF13-COOH additive was added, on the friction coefficient, are shown in Figure 4. These two oils have a similar dynamic viscosity at the testing temperature of 25 °C, but the ester is significantly more polar due to the presence of ester groups, unlike the fully non-polar PAO.

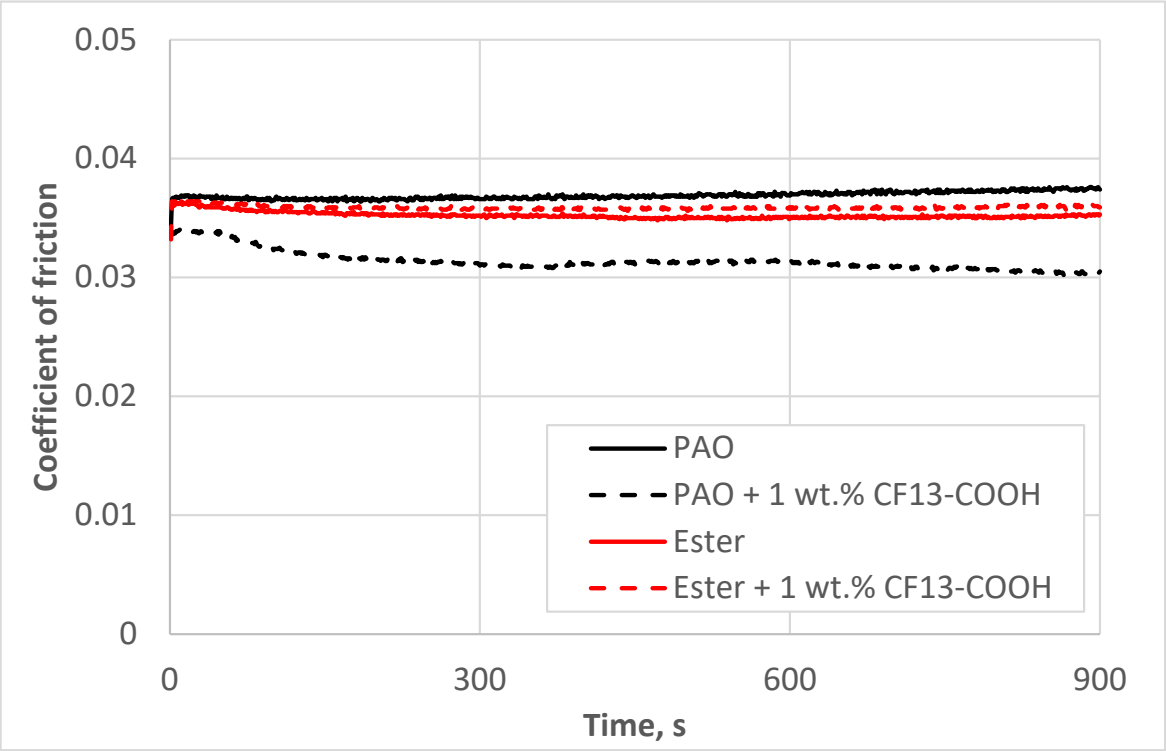


Figure 4. Coefficient of friction evolution for base synthetic PAO oil and base synthetic ester and mixtures of these two oils with 1 wt.% of CF13-COOH additive.

It can be seen that the two base oils provide different friction coefficient values, even though they have similar dynamic viscosities at 25 °C (the temperature at which the tests were conducted). Specifically, the non-polar synthetic PAO oil results in a higher friction coefficient (around 0.037) compared to the more polar synthetic ester, which has a friction coefficient around 0.035. The base PAO oil also shows a slight increase in the friction coefficient over the course of the test, while the synthetic ester behaves oppositely. This difference can be attributed to the fact that low polarity is one of the key properties of the base oil that contributes to higher friction, as demonstrated in a previous study [15]. It is also known that base oils with less rigid bonding (higher molecular flexibility) result in lower friction due to their higher shear-thinning tendency [16]. In this case, the higher shear-thinning of the ester prevails over its slightly higher pressure-viscosity coefficient compared to the base PAO oil.

When the CF13-COOH additive was added to both base oils, we observed different effects. In the case of the synthetic PAO oil, the friction coefficient was lower from the start and continued to decrease throughout the test, eventually reaching a value around 0.030. On the other hand, the friction coefficient in the synthetic ester with the CF13-COOH additive initially showed a similar trend to the base oil, as did the overall friction profile (Figure 4). However, the final friction coefficient for the mixture with the CF14 additive in this case is even slightly higher than that of the base oil.

3.3. Role of Additive Concentration

The results of the tests from Step 3, where we examined how the concentration of the CF13-COOH additive affects the coefficient of friction, are shown in Figure 5. The two mixtures with the smallest tested concentrations of CF13-COOH additive (0.10 wt.% and 0.20 wt.%) exhibit a similar friction coefficient profile compared to that of the base PAO oil (Figure 5), where the coefficient of friction only slightly increased throughout the test. By the end of the test, these two mixtures had a slightly lower coefficient of friction compared to the base oil, but there were no significant differences between them. A substantial decrease in the coefficient of friction is observed when the concentration of the CF13-COOH additive is raised to 0.35 wt.%. At this concentration, the coefficient of friction is significantly lower at the beginning of the test than that of the base PAO oil and decreases rapidly during the first 300 s, followed by a moderate increase. Nevertheless, the final coefficient of friction (0.033) remains significantly lower than the base oil's value of approximately 0.037. With further increases in the CF13-COOH additive concentration to 0.50 wt.% and 0.75 wt.%, these two mixtures, at least initially, do not provide as low a friction coefficient as the mixture with 0.35 wt.% CF13-COOH. However, in these cases friction remains more stable and does not show a marked increase towards the end of the tribological test. The lowest coefficient of friction is achieved with the largest two concentrations of CF13-COOH additive (1.00 wt.% and 2.00 wt.%). For these two mixtures, the coefficient of friction is very low at the start of the test, followed by a relatively steep decrease in the first 200 s to 300 s. Even in the later part of the test, these two mixtures with CF13-COOH additive do not show as significant an increase in friction compared to the mixtures with lower concentrations of the CF13-COOH additive (Figure 5).

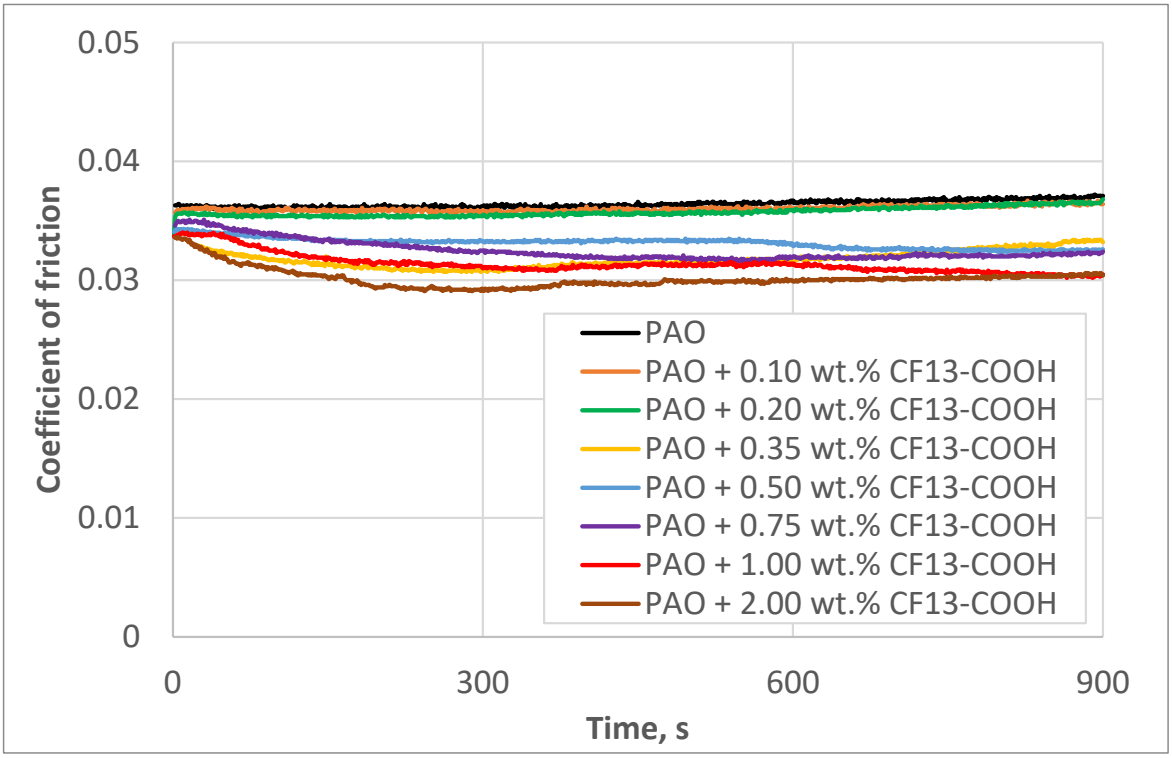


Figure 5. Coefficient of friction evolution for base synthetic PAO oil and mixtures of this oil with different concentrations (from 0.10 wt.% to 2.00 wt.%) of CF13-COOH additive.

3.4. Role of Contact Pressure

The results of the final step, Step 4, are shown in Figure 6. In this step, we tested the effect of contact pressure on the friction coefficient for a 1 wt.% CF13-COOH additive in base PAO oil. First, observing the friction coefficient trends shown in Figure 6, we can see that for the base PAO oil, as the contact pressure increases, the friction coefficient also increases (see the solid lines in Figure 6). At a pressure of 0.69 GPa, the friction coefficient is around 0.028 at the end of the test; at 0.98 GPa, it is around 0.037; and at 1.27 GPa, it is around 0.041. This is expected, as dynamic viscosity increases exponentially with pressure, inducing significantly higher viscous forces when in contact and thereby increasing friction. As the *SRR* was set to 50%, a higher pressure also induces higher shear. When 1 wt.% CF13-COOH additive was added to the PAO oil, the friction coefficient decreased for all three pressures, with the trends showing that the reduction was not the same across all pressures. Namely, the highest coefficient of friction reduction when CF13-COOH was added was observed at the lowest pressure (of 0.69 GPa), while at the highest pressure (of 1.27 GPa) this reduction was the lowest.

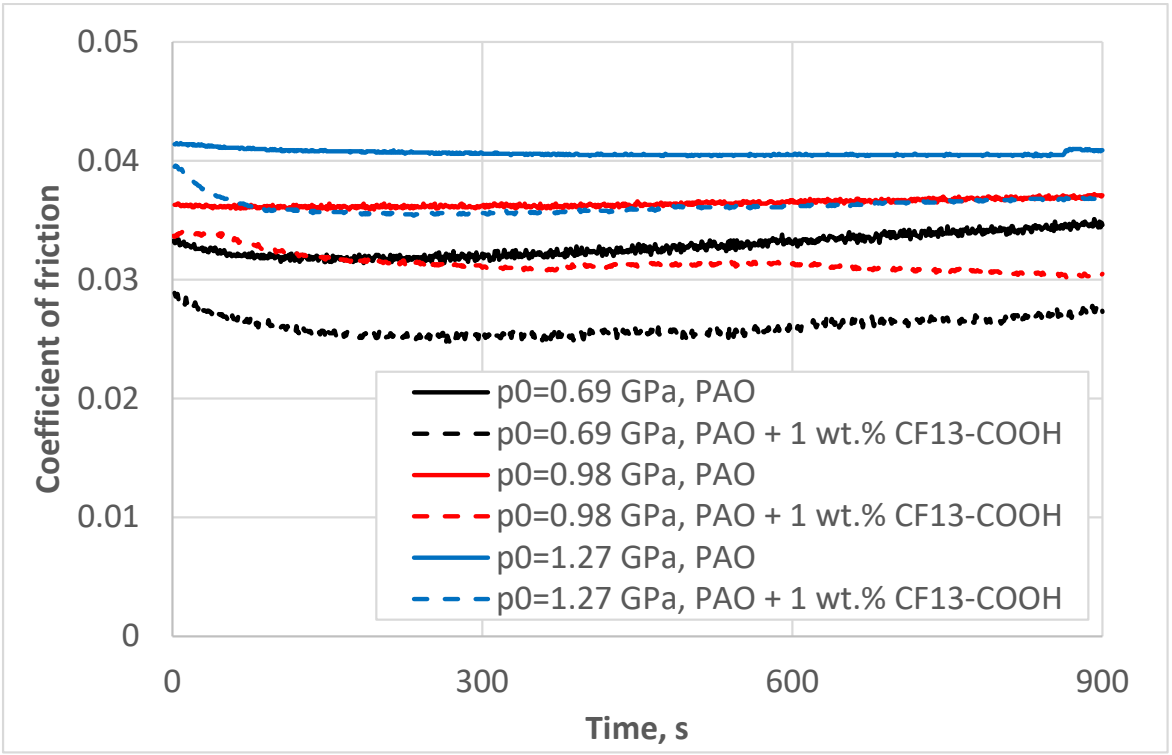


Figure 6. Coefficient of friction evolution for base synthetic PAO oil and mixture of this oil with 1 wt.% of CF13-COOH additive tested at three different contact pressures (0.69 GPa, 0.98 GPa and 1.27 GPa).

4. Discussion

To show the effect of perfluorotetradecanoic acid as an additive for friction reduction in full-film EHD contacts, the changes in the coefficient of friction compared to the base oil at the end of the tests were calculated from the results in Figures 3–6. These effects have been studied with respect to the influential parameters tested (additive polar group, base oil polarity, additive concentration and contact pressure) and are shown in Figure 7.

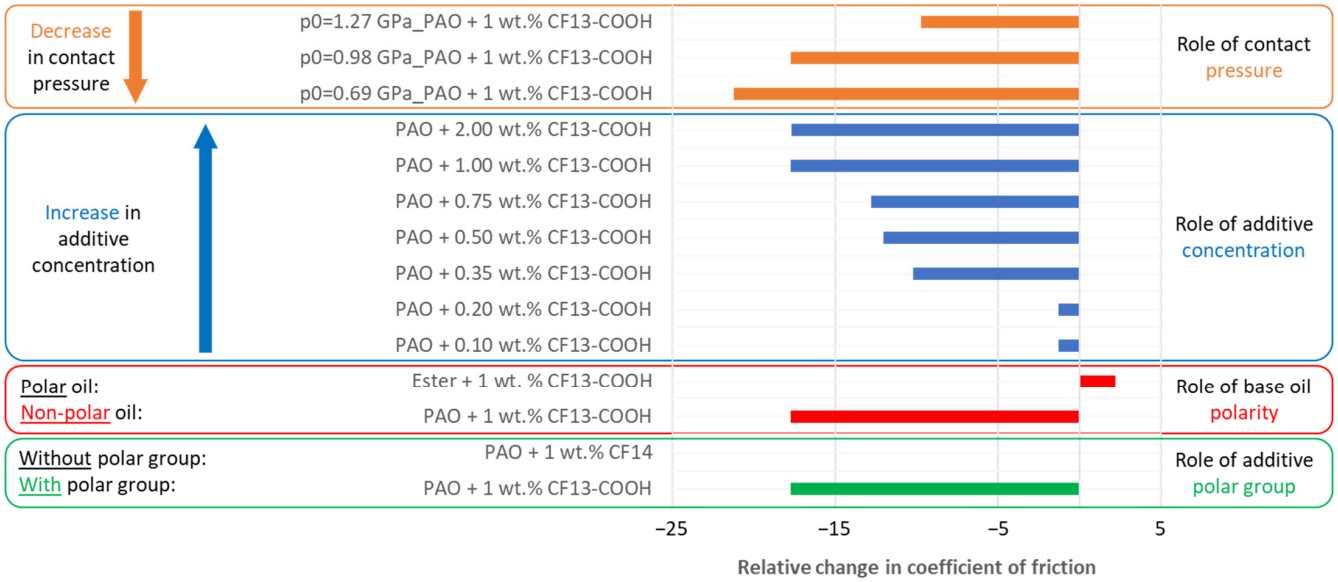


Figure 7. Relative changes in coefficient of friction when perfluorotetradecanoic acid additive was added into base oil for all influential parameters studied.

4.1. Role of Additive Polar Group

As shown in Figure 7, the additive without the polar functional group (CF14 additive) does not reduce friction, while the CF13-COOH with a polar carboxylic functional group achieves a nearly 18% reduction (Figure 7). Based on these results, it is evident that the carboxylic acid functional group is essential for achieving friction reduction and that long chain perfluorinated hydrocarbons do not spontaneously deposit onto a steel surface. This can be explained by the need for an active, polar part of the additive that can adsorb onto the surface—an effect that has been demonstrated in previous studies involving similar types of additives on different substrate materials [17].

4.2. Role of Base Oil Polarity

When we look at the relative friction reduction compared to the base oil, shown in Figure 7, it is evident that a reduction of around 18% in the friction coefficient is achieved only when the CF13-COOH additive is added to synthetic PAO oil. Conversely, the addition of CF13-COOH to the synthetic ester has a detrimental effect on friction reduction: the friction coefficient even increases by about 4%. These findings suggest that in our experiments the CF13-COOH additive only reduces the friction coefficient when added to synthetic PAO oil.

This difference in performance can be explained by the difference in polarity between the synthetic PAO oil and the synthetic ester. The PAO oil, due to its chemical structure based on long alkyl chains without double bonds or polar functional groups, is completely non-polar. When a predominantly non-polar additive with a polar carboxyl functional group is added to such an oil, the additive can easily adsorb onto the steel surface, as there is no other chemical compound in the non-polar oil that would compete for adsorption. In contrast, the synthetic Ester has a polar ester group, which also seeks to bind to the steel surface and can prevent the CF13-COOH additive from adsorbing to the steel surface, thereby hindering its ability to reduce friction. In addition, the polarity of the base oil affects also the solubility of the additives and the potential hydrogen bonding between the polar molecules in the base oil and polar head of the CF13-COOH additive. The fact that oil polarity reduces the adsorption ability of polar molecules has already been shown in other studies [18].

4.3. Role of Additive Concentration

Relative changes in the friction coefficient compared to the base oil, shown in Figure 7, demonstrate that mixtures with the lowest concentrations of the additive (0.10 wt.% and 0.20 wt.%) only slightly reduce the coefficient of friction by about 1%, which is comparable to the measurement uncertainty. A substantial reduction in friction is observed in the mixture with 0.35 wt.% CF13-COOH additive, which already lowers friction by about 10% relative to the base oil. Thus, 0.35 wt.% CF13-COOH additive can be considered as the critical concentration required to effectively reduce friction. Further increases in the concentration of CF13-COOH additive lead to additional reductions in the coefficient of friction, with reductions of 12–13% for 0.50 wt.% and 0.75 wt.%, and an 18% reduction at both of the highest concentrations (1.00 wt.% and 2.00 wt.%). These results indicate that the concentration of the CF13-COOH additive has a significant impact on the reduction in the coefficient of friction relative to the base oil, and that there is a critical concentration (around 0.35 wt.%) at which the friction-reducing effect becomes noticeable. The effect of additive concentration depends much on the additive-base oil system and in turn on the adsorption ability. There have been many studies on the effect of additive concentration on the friction, but for the boundary lubrication regime [17,19] where the additives plays a key role. However, in this study, the critical concentration of CF13-COOH additive is needed to enhance the formation of low surface-energy slippery layer, as shown in detail in our recent study, where the mechanism of the friction reduction is elaborated in detail [12]. In addition, several other studies support the slip including mechanism for friction reduction mechanism in both low pressure [20–22] and high-pressure contacts [4,5,23–25].

As can be seen from Figure 5, at the end of the test there is a clear correlation between the decrease in the coefficient of friction and the increase in the additive concentration. This is attributed to the adsorption mechanism of the CF13-COOH additive. As reported in our complementary study, the CF13-COOH additive immediately forms a boundary layer [12], which also explains the rapid drop in the coefficient of friction at the beginning of the test (see Figure 5). This drop in friction requires the critical concentration of the additive (above 0.20 wt.%), while the concentration above this threshold does not play a role in this initial phase of the test. The role of the concentration is determined by the duration of the test. Indeed, the increase in the coefficient of friction during the test is due to the partial removal of the additive layers, as reported in [12], and a higher additive concentration can maintain the replenishment of the additive layers and thus cause a higher friction reduction.

4.4. Role of Contact Pressure

As shown in Figure 7 the largest reduction in the coefficient of friction relative to the base oil was achieved at the lowest pressure (0.69 GPa) by around 22%, slightly lower at the pressure of 0.98 GPa (by around 18%) and the lowest at the highest pressure (1.27 GPa) by just under 10%. Higher contact pressure thus negatively affects the extent of the friction coefficient reduction, which means that CF13-COOH additive is significantly more effective at reducing friction at low than at high pressures. This can be attributed to the fact that the CF13-COOH additive is, due to performing tests at ambient temperatures at 25 °C, weakly physiosorbed to the steel surface and more prone to removal at higher loads and thus higher lubricant shear.

5. Conclusions

Perfluorinated tetradecanoic acid (CF13-COOH) was used as an additive in oil and tested for its ability to reduce the coefficient of friction. We found the following:

1. A polar carboxyl functional group is indispensable in the additive to reduce the coefficient of friction relative to the base oil.

2. Perfluorinated tetradecanoic acid reduces friction in non-polar synthetic PAO oil, while in a more polar synthetic ester, it is unable to reduce friction compared to the base oil.
3. Increasing the concentration of perfluorinated tetradecanoic acid also increases the reduction in friction. The minimum concentration required, at which a perceptible reduction in friction is obtained (by about 10%) is 0.35 wt.%; however, the highest reduction in friction (by about 18%) is obtained at both largest concentrations of 1.00 and 2.00 wt.%.
4. Perfluorinated tetradecanoic acid is best at reducing friction at lower pressures, with a reduction by about 22% at the lowest pressure tested (0.69 GPa) and just below 10% at the highest pressure (1.27 GPa).

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Data Availability Statement: The data presented in this study are available on request from the corresponding author.

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

Abbreviations

The following abbreviations are used in this manuscript:

EHD	Elasto-hydrodynamic
PAO	Poly-alpha-olefin
PTFE	Polytetrafluoroethylene

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