



Article

Tribological Properties of Alkyldiphenylethers in Boundary Lubrication

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Abstract: Lubricants that are used in miniaturized moving mechanical components generally serve under severe conditions, such as high temperature, high speed, and high load. Although alkyldiphenylethers (ADEs) are used as base oils for high-temperature greases, their tribological properties remain unclear. This study investigated the influence of the alkyl chains on the tribological properties of ADEs. Longer and more attached alkyl chains decreased the friction coefficient of ADEs under both reciprocating and continuous sliding conditions. Wear was found to be independent of the alkyl chain under reciprocating sliding conditions due to abrasion being caused by debris that was not readily removed. ADEs showed good anti-wear properties when used as either a lubricant or an additive under continuous sliding conditions. Much smoother surfaces in the friction track were observed in comparison to poly- α -olefin. Fourier-Transform infrared spectroscopy analysis suggested that the ether groups were attracted to the worn steel surface, phenyl groups became consequently perpendicular, and the attached alkyl chains repelled other substances and prevented further wear of the surface. Moreover, ADEs showed a high adaptability with traditional additives.

Keywords: alkyldiphenylether; alkyl chain length; boundary lubrication; reciprocating sliding contact; continuous sliding contact; lubricant additive

1. Introduction

As machines have been increasingly downsized in recent years, demand for smaller, lighter, higher-speed, and maintenance-free moving mechanical components has grown [1–3]. Lubricants used in such components generally serve under severe conditions, such as high temperature, high speed, and high load. It is known that temperature rise, thermal decomposition, and oxidative deterioration in lubricants occur due to the breakage of the oil film under operating conditions of high temperature and high speed. As a result, accelerated evaporation of the base oil leads to sludge generation, bearing damage, and reduction in machine life. Moreover, it is known that lubricant oils tribochemically decompose, due to the high activity of a nascent steel surface: hydrogen and gaseous hydrocarbons are generated [4]. Hydrogen embrittlement occurs once generated hydrogen diffuses into the substrate steel. Furthermore, lubricant decomposition leads to poor lubrication.

Therefore lubricants with thermal and chemical stabilities are required. Polyphenyl ethers, silicone oil, perfluoropolyether (PFPE), and ionic liquids have been developed as high-temperature base oils [5–8]; however, either the high price of polyphenyl ethers, PFPE, and ionic liquids, or the poor lubricity of silicone oil limits their application. Moreover, PFPE is known to become highly chemically active when in contact with a Lewis acid, such as alumina [9].

Alkyldiphenylethers (ADEs) are used as base oils in high-temperature greases due to their high thermal and oxidation stabilities. ADEs are also used as pump oils because of their low evaporation and small change in viscosity, even at high temperature. On an AISI 52100 steel surface, the tribochemical

decomposition of ADEs was found to be negligible, even after a long period of friction, and no hydrogen was generated during friction [10]. It was found that hydrogen embrittlement that was caused by hydrogen ingress greatly decreased when ADE was used as the base oil in bearing grease [11]. However, to date, the tribological properties of ADEs have rarely been discussed.

The aim of this study was to investigate the tribological properties of ADEs in boundary lubrication, especially the effect of alkyl chain length and the number of attached alkyl chains. We demonstrated the lubricity of ADEs under reciprocating and continuous sliding conditions. The adaptability of traditional additives in ADEs is discussed and the mechanisms are explored.

2. Materials and Methods

2.1. Lubricants and Additives

Alkyldiphenylethers (MORESCO Corporation, Kobe, Japan) with different alkyl chain lengths and numbers of attached alkyl chains were used. Figure 1 shows the chemical structure of the ADEs and Table 1 lists the alkyl chains. The phenyl group provides chemical stability, while the alkyl chain length and the number of attached chains control the flexibility. All of the alkyl chains were straight. Table 2 lists the typical physical properties of the ADEs. Poly- α -olefin (PAO30, provided by MORESCO Corporation, Kobe, Japan) was used as a reference.

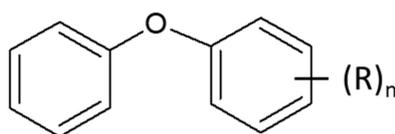


Figure 1. Chemical structure of alkyldiphenylethers.

Table 1. Alkyl chains in studied alkyldiphenylethers.

R	Denotation for ADEs	
	n = 1	n > 2
C ₁₂ H ₂₅ & C ₁₄ H ₂₉	C12	2C12
C ₁₈ H ₃₇	C18	-
C ₂₀ H ₄₁	C20	-

Table 2. Physical properties of alkyldiphenylethers.

Physical Properties	C12	C18	C20	2C12
Density (15 °C g·cm ⁻³)	0.945	0.928	0.905	0.895
Flash point (°C)	214	250	206	286
40 °C viscosity (mm ² ·s ⁻¹)	15.8	26.1	37.5	102.0
100 °C viscosity (mm ² ·s ⁻¹)	3.4	4.9	5.8	12.6
Viscosity index	74	110	95	118
Pour point (°C)	-55	-50	-42.5	-40

We used oleic acid (FUJIFILM Wako Pure Corporation, Osaka, Japan), stearic acid (FUJIFILM Wako Pure Corporation, Osaka, Japan), zinc dialkyldithiophosphate (ZnDTP, provided by MORESCO Corporation, Kobe, Japan), and molybdenum dithiocarbamate (MoDTC, provided by MORESCO Corporation, Kobe, Japan) as additives when discussing the effect of additives on tribological properties of ADEs. Table 3 shows the concentrations of the additives in the lubricants.

2.2. Tribological Tests

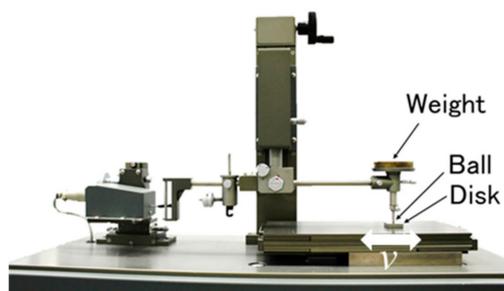
All of the tribological tests were carried out under ambient conditions (23 ± 2 °C, relative humidity of 45–55%) and repeated at least three times for each test condition.

Table 3. Additive concentrations.

Lubricants	Additive Concentration, wt %
C12 containing MoDTC/ZnDTP	0.1/1
C12 containing oleic acid	1
C12 containing stearic acid	0.1

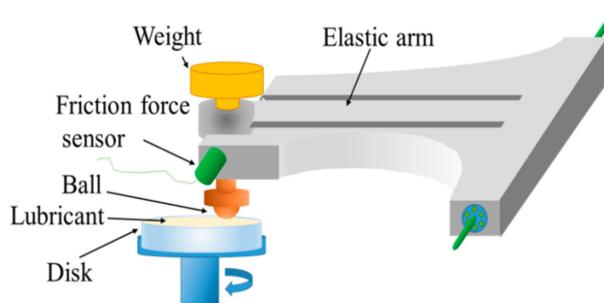
2.2.1. Friction Tests under Reciprocating Sliding Condition

A reciprocating ball-on-disk friction tester (Heidon-14DR, SHINTO Scientific Co., Ltd., Tokyo, Japan), as illustrated in Figure 2, was used to measure the friction coefficients of the ADEs under reciprocating sliding conditions. The ball was made of AISI 52100 steel and it was 6.35 mm in diameter. An AISI 52100 steel disk was polished with a surface roughness of Ra 0.015. The load was 10 N and the speed was $8.0 \text{ mm}\cdot\text{s}^{-1}$. The sliding stroke was set to 4 mm and the test duration was 1 h.

**Figure 2.** Schematic of reciprocating friction tester.

2.2.2. Friction Test under Continuous Sliding Condition

Friction tests under continuous sliding conditions were conducted on a ball-on-disk tribometer (Tribometer Pin-disc Machine, CSEM, Newchatel, Switzerland), as illustrated in Figure 3. The same ball and disk were used, as mentioned in Section 2.2.1. The applied load was controlled at 10 N and the rotating speeds were 8.0 and $37.7 \text{ mm}\cdot\text{s}^{-1}$. The test duration was 1 h.

**Figure 3.** Schematic of ball-on-disk tribometer.

2.3. Characterization

Following the friction tests, the test pieces were rinsed while using hexane to remove excess lubricant. The wear track on the disk was observed while using an optical microscope and the surface morphology of the wear scar was analyzed using a surface profilometer (Surfcorder SE-3400, Kosaka Laboratory Ltd., Tokyo, Japan), unless otherwise noted.

Fourier-Transform infrared (FTIR) spectrometry was used to analyze the molecular conformation before and after friction. The test conditions were as follows: IR instrument, LUMOS (Bruker, Ettlingen, Germany); mode, reflection; aperture size for IR sampling, $100 \mu\text{m} \times 100 \mu\text{m}$; resolution wavenumber, 4 cm^{-1} ; number of scans, 64.

The acid concentration that was adsorbed on a steel surface was measured while using the FTIR spectrometer in attenuated total reflectance (ATR) mode. FTIR in reflection mode generally provides the bulk information, while that in ATR mode could provide the information of adsorption, since the crystal is directly pressed onto sample surface. The relative intensity of C=O in carboxyl ring versus C–H stretching vibration ($I\nu_{(C=O)}/I\nu_{(C-H)}$) can be used to represent the concentration of an acid in the lubricants. We measured $I\nu_{(C=O)}/I\nu_{(C-H)}$ in ATR mode and calculated the acid concentration adsorbed on AISI 52100 steel surface by using the correlation between $I\nu_{(C=O)}/I\nu_{(C-H)}$ and the acid concentration in bulk lubricants, which was measured in reflection mode.

X-ray photoelectron spectroscopy (XPS) analysis was carried out using a PHI X-tool scanning X-ray microprobe (ULVAC-PHI, Chiagasaki, Japan) to determine the chemical component of the tribofilm when discussing the effect of additives on tribological properties of ADEs. The photoelectrons were excited by monochromated Al-K α at 12.5 W and 15 kV. The analyzed area was 100 $\mu\text{m} \times 100 \mu\text{m}$. The take-off angle was 45°. The binding energies of the spectra were calibrated by setting a maximum C1s peak at 284.5 eV.

3. Results and Discussion

3.1. Tribological Properties of Alkyldiphenylethers under Reciprocating Sliding Condition

Typical friction curves, as measured using the reciprocating friction tester, are shown in Figure 4. There was no obvious difference in friction coefficient during the first few minutes; however, that of C12 gradually increased with friction time, which was similar behavior to that of PAO30. The friction coefficients of C18, C20, and 2C12 stabilized within a few minutes. These results suggest that ADEs with longer alkyl chains or more attached alkyl chains preferred operating under more stable friction conditions.

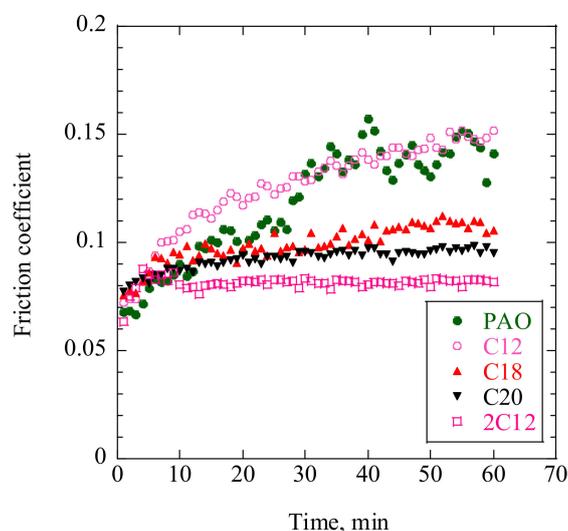


Figure 4. Variation of friction coefficient with friction time under reciprocating sliding condition.

Figure 5a summarizes the averaged friction coefficients at steady state, as determined from the friction coefficient curves illustrated in Figure 4. The C12 ADE with a short alkyl chain showed the highest friction coefficient of 0.142. When the alkyl chain length increased to C18, the friction coefficient decreased to 0.105. When the alkyl chain length further increased to C20, a much lower friction coefficient of 0.095 was observed, which was 33% lower than that of C12. These results showed that the friction coefficients of ADEs strongly depended on the alkyl chain length. Moreover, 2C12, which featured more than two alkyl chains, showed the lowest friction coefficient of 0.081. The friction coefficient decreased by 43% on increasing the number of attached alkyl chains, even if the alkyl chain length was short.

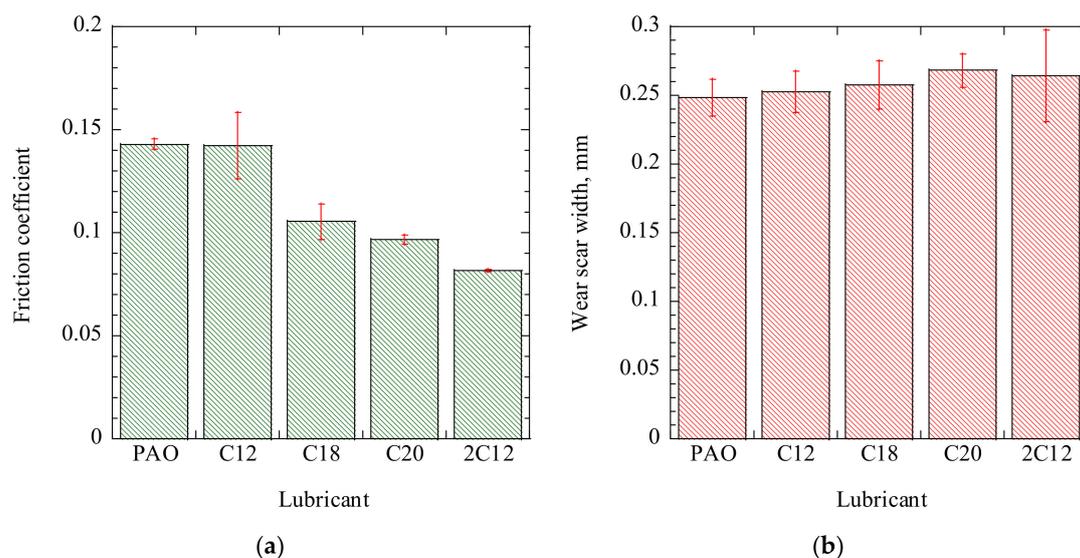


Figure 5. Tribological properties under reciprocating sliding condition: (a) averaged friction coefficient and (b) wear scar width.

Figure 5b shows the wear scar width on the disks that were lubricated with the sample oils. Although the friction coefficients of ADEs depended on alkyl chain length, the wear scar width seemed to be independent of alkyl chain length and number of attached chains. The wear scar width was almost the same when PAO30 was used, so we think that reasons other than the molecular chemical structures induced this phenomenon. Figure 6 shows a typical photograph of the friction track on the disk under reciprocating sliding condition. We observed much debris, both inside and outside of the friction track. In this case, the speed decreased to zero at the end of a specimen traverse and then accelerated to a similar speed in the opposite direction, because the motion was reciprocating. Therefore, the debris could not be quickly removed from the friction area. Moreover, the short sliding stroke made the debris easily accumulate in friction track. Therefore, abrasion occurred [12]. As a result, we observed severe wear on the disks for all of the sample oils.

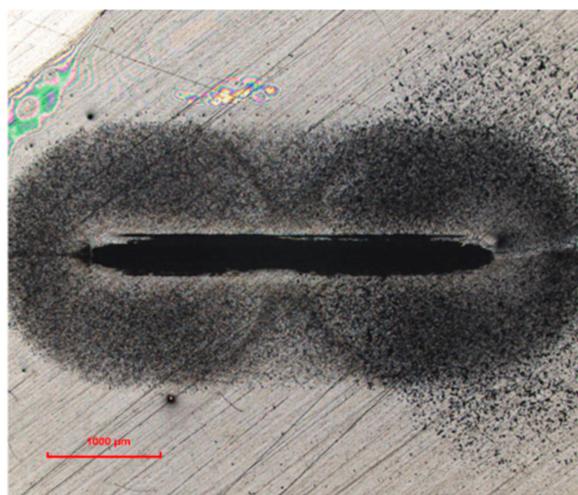


Figure 6. A typical photograph of friction track on the disk under reciprocating sliding condition (lubricant. C12).

3.2. Tribological Properties of Alkyldiphenylethers under Continuous Sliding Condition

The friction coefficients of the ADEs were measured under continuous sliding conditions while using the ball-on-disk tribometer illustrated in Figure 3. Figure 7a,b, respectively, show the friction

coefficient of ADEs and wear scar width on the disks, which were tested at the same load and speed as those of the reciprocating sliding conditions. In this case, we also confirmed that the friction coefficient of the ADEs decreased with the number of attached alkyl chains. Although the averaged friction coefficient of C20 was higher than that of C18, both of them were lower than C12. Moreover, the wear scar width on the disk was 0.214 mm when lubricated with C12 in this case, but it decreased to 0.198 mm when C18 was used. The wear scar width further decreased to 0.155 mm when the longest alkyl chain, C20, was used. These results demonstrate that the increase in the alkyl chain length effectively improved the anti-wear properties. The wear decreased by 28% in the wear scar width when C20 was used. In contrast, the wear scar width was 0.210 mm when 2C12 was used, which was almost the same as that of C12. This indicated that the number of attached alkyl chains displayed less effect on the anti-wear properties than the alkyl chain length.

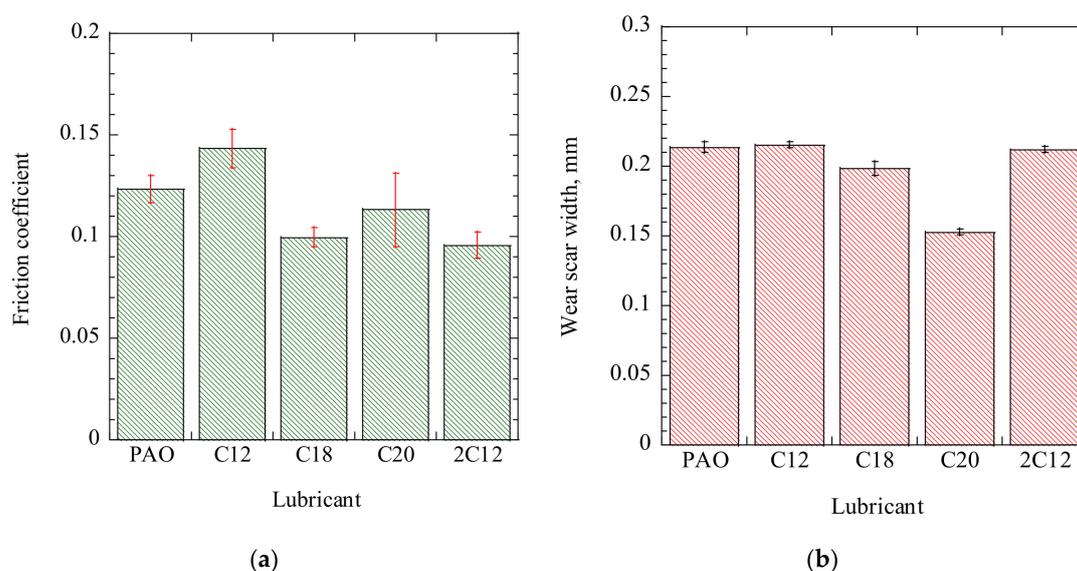


Figure 7. Tribological properties of alkyldiphenylethers under continuous sliding condition: (a) averaged friction coefficient and (b) wear scar width. (Speed: $8.0 \text{ mm}\cdot\text{s}^{-1}$)

As shown in both Figures 5 and 7, C12 showed almost the same friction coefficient and wear as the reference lubricant, PAO30; however, the friction coefficient of PAO30 decreased from 0.143 to 0.125 when the continuous sliding friction tests were carried out at a higher speed of $37.7 \text{ mm}\cdot\text{s}^{-1}$, while the wear scar width on the disk increased from 0.212 to 0.268 mm, as shown in Figure 8. For C12, the wear scar width decreased to 0.200 mm, although the friction coefficient was almost unchanged. In other words, C12 featured much lower wear on the disk when compared with PAO30. These results suggest that PAO and ADEs exhibited different friction mechanisms. Figure 9 provides photographs and surface profiles of the wear scars lubricated with PAO30 and C12 to improve understanding of the mechanisms. A much rougher surface can be observed when PAO30 was used, with many grooves and deep furrows distributed in the friction track. In contrast, a relatively smooth surface was generated in the friction track when C12 was used. Moreover, the contact surface in the friction track became very smooth when only 1 wt % C12 was added into PAO30, as shown in Figure 8. These data indicate that ADE effectively protected the surface from severe wear.

The question remained as to why ADEs showed these anti-wear properties. It was postulated that this might relate to their adsorption on the steel surface. Figure 10 shows the FTIR spectra of C12. The peaks at $1350\text{--}1500 \text{ cm}^{-1}$ correspond to the deformation mode of the C–H vibration. The peak at 1594 cm^{-1} is assigned to the aromatic C=C stretching vibration (ν), while that at 750 cm^{-1} is attributed to the aromatic C–H out-of-plane deformation vibration (ω). As illustrated in Figure 11, the observed IR absorption will be the largest for vibrations with in-plane dipole derivative vectors more parallel to the surface normal [13,14], while the lowest for vibration with out-of-plane dipole derivative vectors.

The relative IR intensities of vibrations whose dipole derivatives are oriented along different molecular axes can be determine the orientation of the molecules. In this case, the observed IR absorption will be largest for aromatic C=C stretching vibrations and simultaneously smallest for the aromatic C-H out-of-plane deformation vibration when the aromatic ring is perpendicular to an infrared beam; therefore, the relative intensity of the aromatic C=C stretching vibration as compared with that of the aromatic C-H out-of-plane deformation vibration ($I\nu_{(C=C)}/I\nu_{(C-H)}$) can be used to represent the degree of orientation of the aromatic ring, i.e., the phenyl groups in ADEs. Based on the FTIR spectra, we found that I

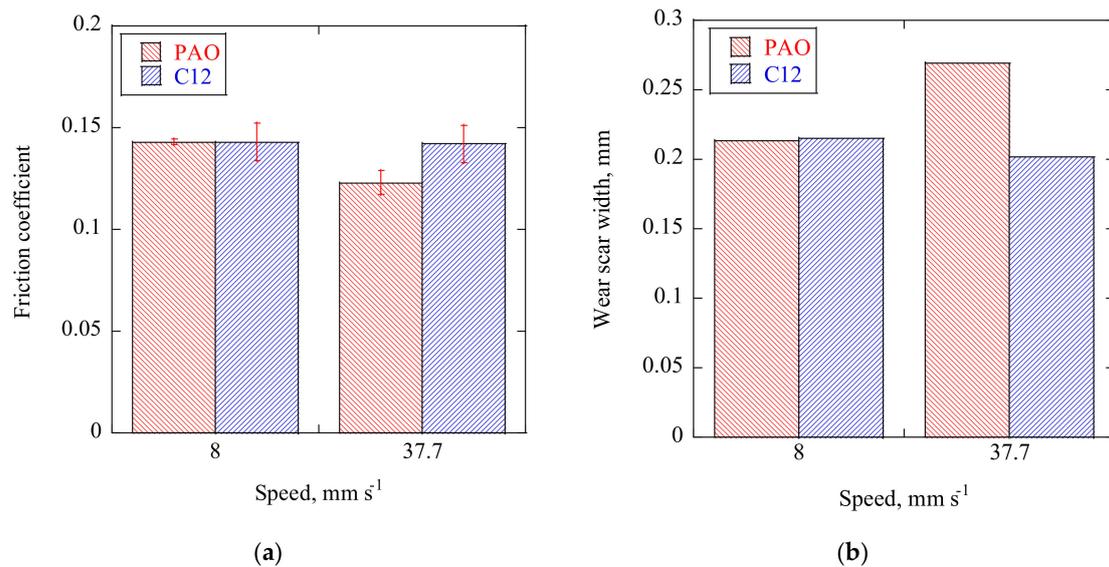


Figure 8. Effect of sliding speed on tribological properties of poly- α -olefin (PAO) and C12 alkyldiphenylether under continuous sliding condition: (a) averaged friction coefficient and (b) wear scar width. (Speed: 37.7 mm·s⁻¹).

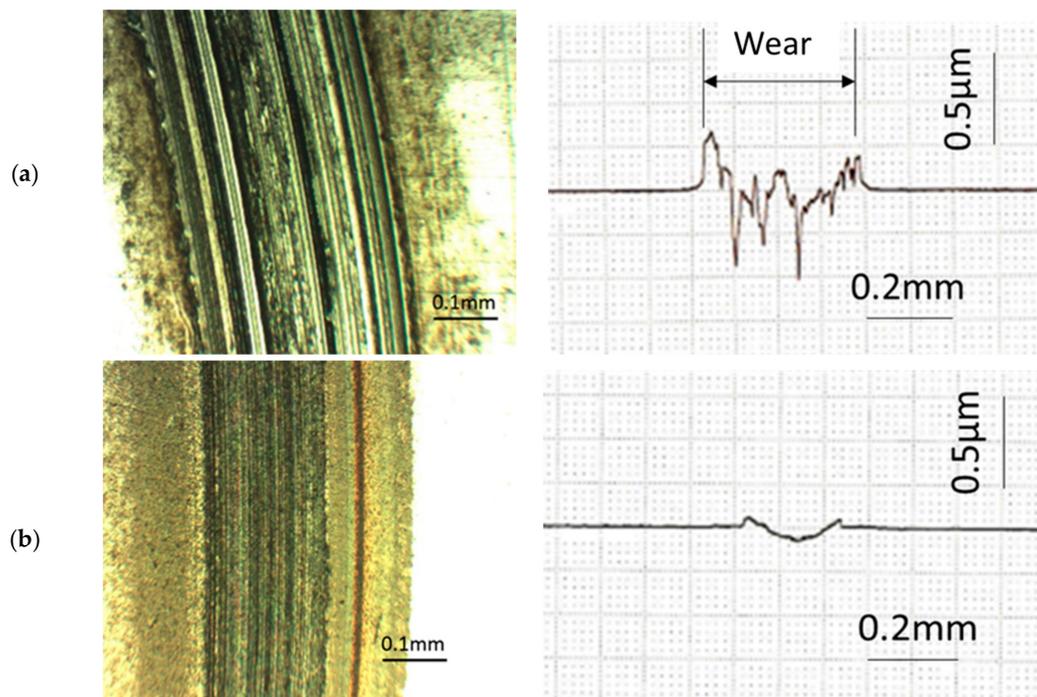


Figure 9. Cont.

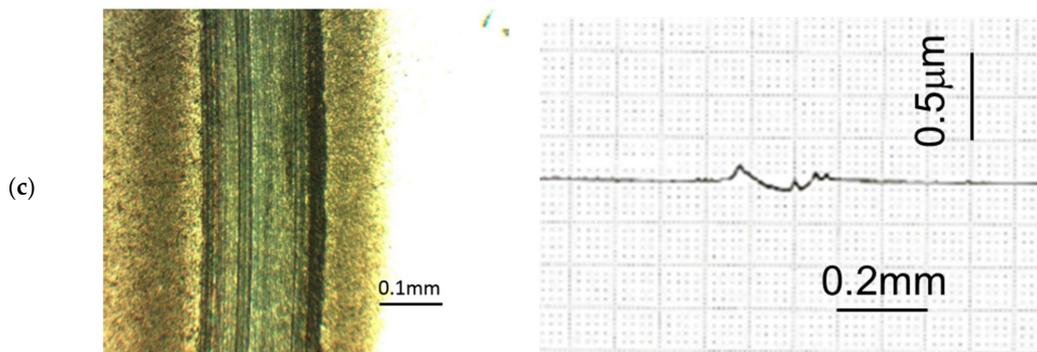


Figure 9. Optical micrographs and surface profiles of wear scars on disks lubricated with (a) PAO30, (b) C12, and (c) C12/PAO30 (1 wt %). (Speed: $37.7 \text{ mm}\cdot\text{s}^{-1}$).

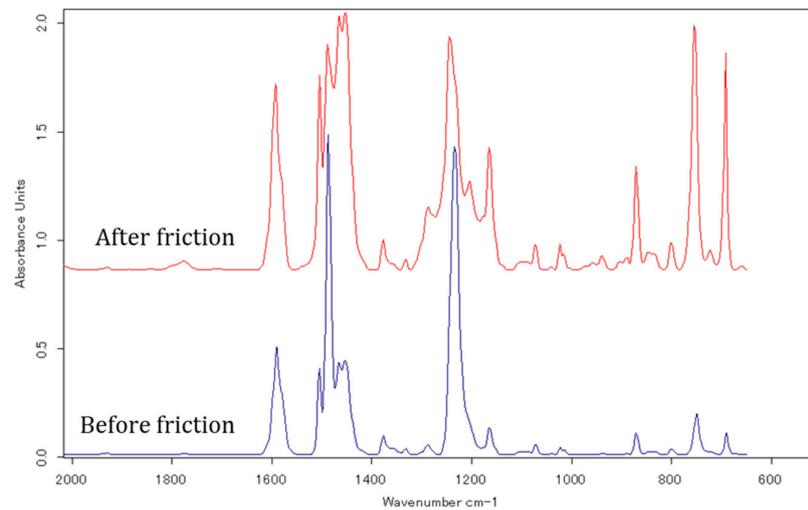


Figure 10. Fourier-Transform infrared spectra of C12 before and after friction.

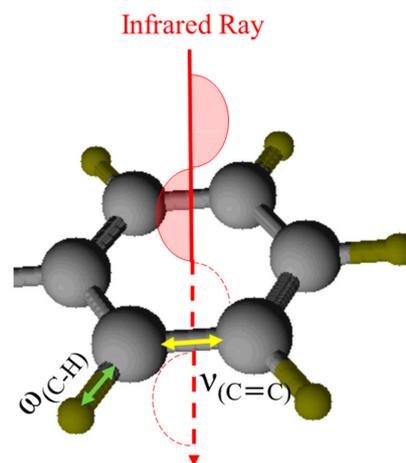


Figure 11. Relationship between incident light and the aromatic ring in the molecular structures.

$\nu_{\text{(C=C)}}/\omega_{\text{(C-H)}}$ was 4.0 before friction and decreased to 1.1 after friction. In other words, the phenyl group tended to adsorb parallel to the metal surface before friction, but perpendicularly to the worn metal surface. When the phenyl group adsorbed parallel to the metal surface, the attached alkyl chains simultaneously lay on surface due to the molecular conformation, leading to higher surface coverage and therefore protected the surface from wear. Once the surface was worn, the ether groups were attracted to the metal oxide surface, the phenyl groups became perpendicular, and the attached alkyl chains repelled other substances and prevented further wear of the surface. At the same time,

weak bonds between the alkyl chains in adjacent molecules enhanced the durability of the lubricant film and reduced the friction, especially for longer alkyl chains.

3.3. Effect of Additives on Tribological Properties of Alkyldiphenylethers

We found that ADEs showed a lower friction coefficient and less wear, so we further sought means to promote their tribological properties. We hypothesized that the addition of traditional additives, such as ZnDTP, MoDTC, oleic acid, and stearic acid, would still show friction reduction or this anti-wear effect.

Here, we focused on C12. We measured the friction coefficients of the sample oil with these additives while using the ball-on-disk tribometer in a continuous sliding motion, as shown in Figure 3. As shown in Figure 12, additive-free C12 showed a high friction coefficient with an average value of 0.153. Fluctuations in the friction coefficient with friction time were also observed. When ZnDTP or MoDTC were used as the additive, the friction coefficient significantly decreased and then stabilized. The average friction coefficient of C12 containing ZnDTP/MoDTC was 0.112, which was 27% lower than that of additive-free C12. Oleic acid also decreased the friction coefficient by 27%. Moreover, the friction coefficient seemed to be more stable during the period of applied friction when oleic acid was used. For stearic acid, the friction coefficient decreased during the first 20 min., and then gradually increased to the value of additive-free lubricant.

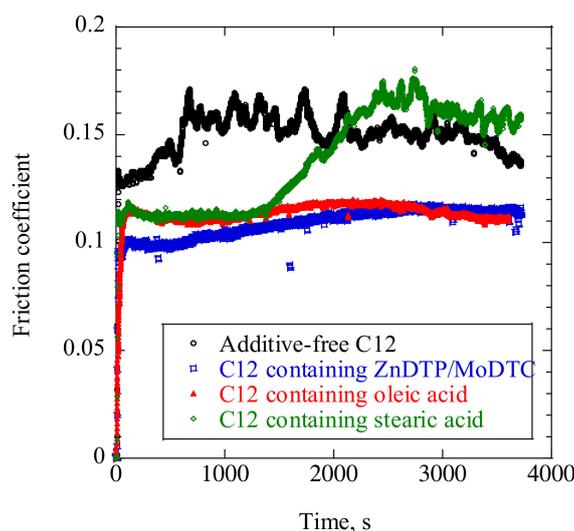


Figure 12. Effect of additives on friction coefficient.

Figure 13 shows microphotographs of the wear scars and surface profiles measured while using a confocal laser scanning microscope (OLS5000, Olympus, Tokyo, Japan). When ZnDTP/MoDTC was added, the width and depth in the wear scar both obviously decreased when compared with the additive-free lubricant. Oleic acid provided the lowest wear in this case and a minor friction scratch was formed on the disk surface. Stearic acid presented less reduction in wear, which was possibly because of the increase in friction coefficient.

In a traditional lubricant, such as PAO, the addition of ZnDTP and MoDTC have proven effective in reducing the friction coefficient and wear due to tribofilm formation [15]. In this case, we carried out XPS analysis after the friction tests when ZnDTP/MoDTC was used. Figure 14 shows the XPS spectra of the tribofilm. The main S 2p peak at 161.28 eV indicated that the sulfur essentially appeared as a metal sulfide [16]. The P 2p peak at 133.1 eV corresponded to the metal phosphate [16]. The Zn 2p spectrum featured the main peak at 1021.7 eV, which suggested the formation of ZnS [15,16]. The Mo 3d spectrum shows a strong peak at 231.88 eV, indicating the presence of molybdenum oxide, while that at 229 eV suggests the formation of molybdenum disulfide, although the peak was very weak in this case [15,16]. We conclude that the tribofilm consisted of metal sulfide, metal phosphate, molybdenum

oxide, molybdenum disulfide, and a carbon-rich phase. Therefore, the friction coefficient and wear decreased in comparison with additive-free C12 when ZnDTP/MoDTC was added. In other words, ZnDTP/MoDTC showed similarly good friction-reduction and anti-wear properties in ADEs as in traditional lubricants.

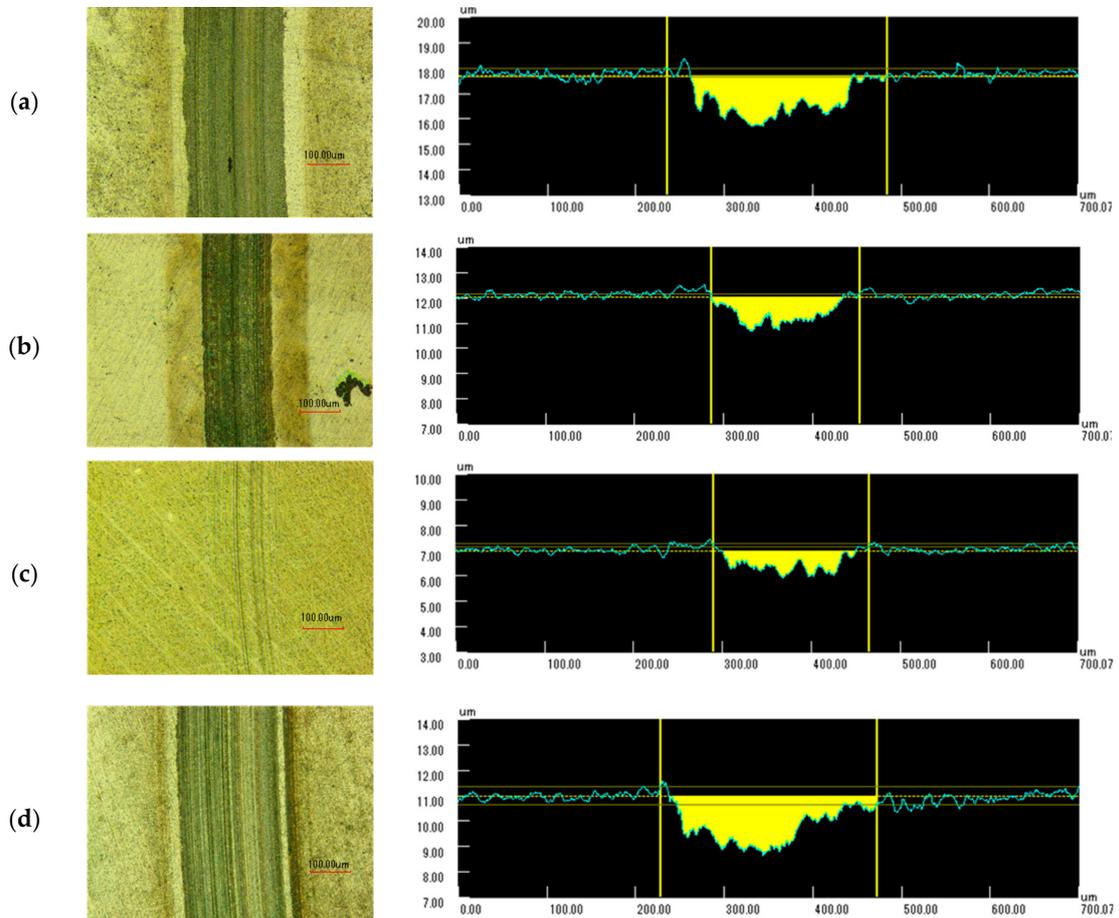


Figure 13. Micrographs and surface profiles of wear scars on disk: (a) additive-free C12, (b) C12 containing zinc dialkyldithiophosphate (ZnDTP) and molybdenum dithiocarbamate (MoDTC), (c) C12 containing oleic acid, and (d) C12 containing stearic acid.

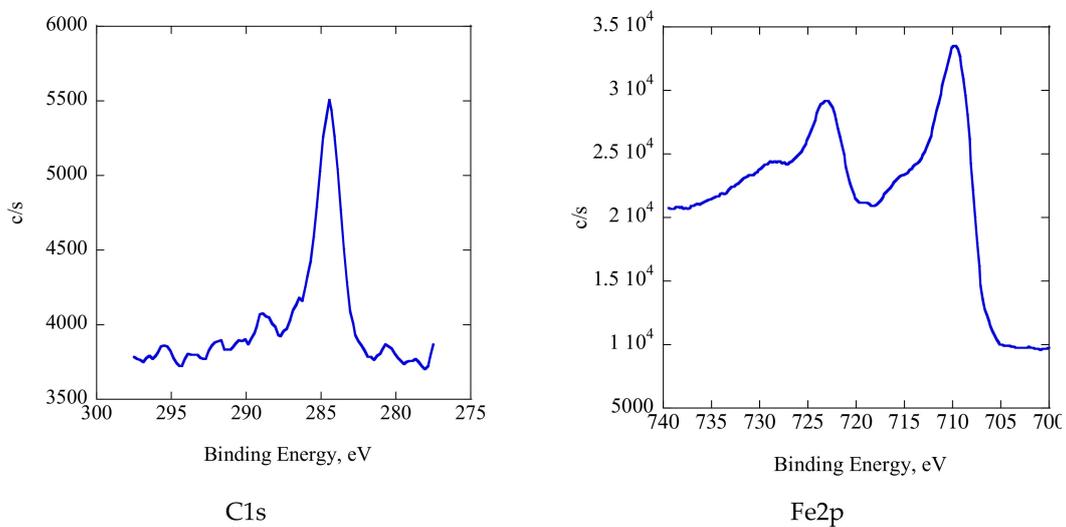


Figure 14. Cont.

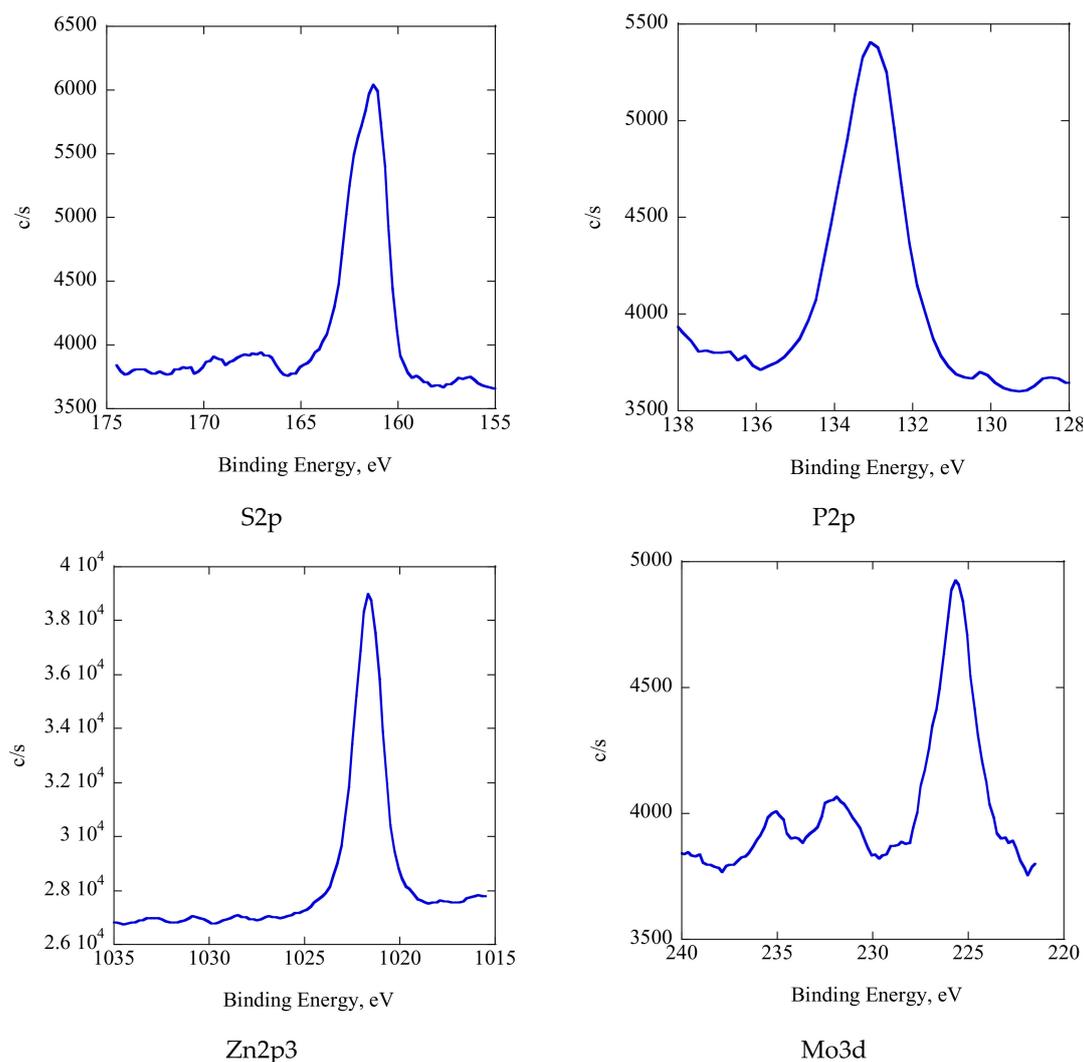


Figure 14. X-ray photoelectron spectra detected in the wear track on the disk lubricated with C12 containing zinc dialkyldithiophosphate (ZnDTP) and molybdenum dithiocarbamate (MoDTC).

Oleic acid and stearic acid are generally used as friction modifiers to reduce the friction coefficient by the formation of a chemisorbed film. In this study, we confirmed that oleic acid effectively decreased the friction and wear of ADE; however, the friction coefficient increased after 20 min. when stearic acid was used. It was reported that stearic acid could reduce the friction coefficient of paraffinic oil to less than 0.1 at a concentration as low as $0.001 \text{ mol}\cdot\text{L}^{-1}$ [17]. In this case, the concentration of stearic acid in C12 was only 0.1 wt %, owing to its poor solubility in ADEs, which corresponds to $0.003 \text{ mol}\cdot\text{L}^{-1}$. Stearic acid reduced the friction coefficient of C12 for a short duration of applied friction. We analyzed the acid concentration at the contact interface while using an FTIR spectrometer in ATR mode. For both oleic and stearic acids, the acid concentration at the contact interface was found to be around $0.06 \text{ mol}\cdot\text{L}^{-1}$, as shown in Figure 15. The bulk concentration of oleic acid in the lubricants was $0.034 \text{ mol}\cdot\text{L}^{-1}$, so enough oleic acid molecules were provided to adsorb on the contact surface, leading to low friction throughout the tests; in contrast, the concentration of stearic acid in the lubricants was significantly lower than that at the contact interface, so the supply was inadequate or not timeously available during friction. In other words, the surface coverage of stearic acid at the contact surface became insufficient, which resulted in the short duration of the period of low friction [18].

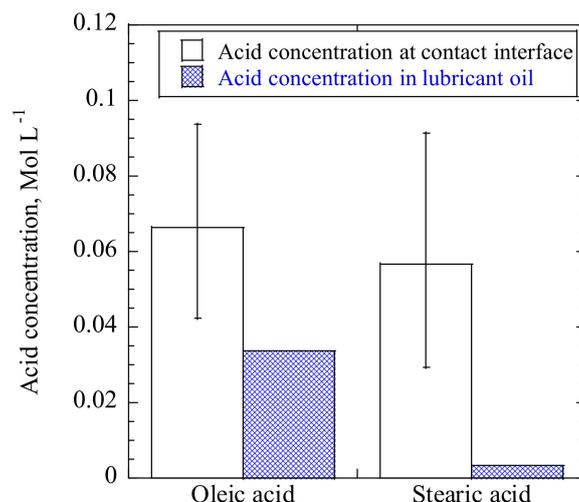


Figure 15. Acid concentration at contact interface.

4. Conclusions

We investigated the tribological properties of ADEs with different alkyl chain lengths and different numbers of attached alkyl chains. The main conclusions that can be drawn are as follows.

The alkyl chains strongly affected the friction coefficients of ADEs under reciprocating sliding conditions. The friction coefficients decreased with an increase in alkyl chain length. Moreover, more attached alkyl chains further reduced the friction coefficient, even if the attached alkyl chain length was short. However, we did not find a dependence of wear on the length or number of alkyl chains under reciprocating sliding conditions. We think that the debris could not be removed as quickly from the friction area and abrasion occurred in this case. As a result, we observed severe wear on the disks for all of the sample oils.

Under continuous sliding conditions, the same effect of alkyl chains on the friction coefficient of ADEs was observed as for reciprocating sliding. Moreover, the wear on the disks obviously depended on the alkyl chains. Longer alkyl chains and a greater number of attached alkyl chains decreased the wear on the disks. Furthermore, a relatively smooth surface in the friction track was generated when ADE was used. ADEs showed good anti-wear properties when they were used as lubricants or additives. FTIR analysis suggested that the phenyl group in ADE molecules adsorbed parallel to the steel surface and the attached alkyl chains consequently lay on the surface, which led to higher surface coverage, and therefore protected the surface from wear. On a worn steel surface, the ether groups were attracted to the metal oxide surface, phenyl groups became perpendicular, and the attached alkyl chains repelled other substances and prevented further wear of the surface.

Traditional additives, such as ZnDTP/MoDTC and oleic acid, promoted the tribological properties of ADEs. The introduction of ZnDTP/MoDTC in C12 obviously decreased the friction coefficient and reduced wear due to the formation of a tribofilm that was derived from the additives. A friction reduction of 20% and no obvious wear was achieved while using oleic acid as the additive; however, stearic acid could only reduce the friction for a short duration, because of insufficient supply at the contact interface due to its poor solubility in ADEs.

These results show that ADEs feature good lubricating performance and high adaptability with traditional additives.

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

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