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Article

Chemically Modified Silicone Oil with Enhanced Tribological and Anti-Foaming Properties

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Abstract: Two kinds of chemically modified silicone oil, diisooctyl phosphate-terminated silicone oil (UCP204) and dioctyl dithiophosphate-terminated silicone oil (UCT2003), were synthesized. The tribological properties of silicone oil were evaluated using an SRV tribometer, and the worn surface and chemical composition were examined by scanning electron microscopy (SEM) coupled with energy dispersive spectroscopy (EDS). The friction coefficients of UCP204 and UCT2003 were reduced by approximately 62.7% and 56.7% compared with untreated silicone oil. The results indicated that the incorporation of phosphorus and/or sulfur could significantly enhance the tribological performance of modified silicone oil. Benefiting from their unique chemical structure, chemically modified silicone fluids also exhibit good dispersion stability and excellent anti-foaming properties, which are mainly attributed to the low surface tension properties imparted by the silicone backbone and the better dispersion stability provided by the dialkyl dithiophosphate group in chemically modified silicone fluids. It is also found that lubricants containing modified silicone oil remain clear after three months of storage and still maintain good anti-foaming properties.

Keywords: silicone oil; chemical modification; tributological properties; anti-foaming; multifunctional

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1. Introduction

Silicone oil is a kind of polymer with repeating Si-O bonds as the backbone; methyl, phenyl and other organic groups are directly connected to the silicon atom [1–4]. It attracts significant attention from both academia and industry due to its good economy and excellent properties, such as good viscosity–temperature properties, low surface tension [5–7], and outstanding thermal and oxidative stability [8–12]. These characteristics give silicone oil a wide range of applications involving lubrication, textile dyeing, and fermentation, as well as in the petroleum industry [13–18]. In the field of lubrication, silicone oil can be used either as a base fluid or as a lubricant additive. As a high-performance functional fluid, silicone oil is widely used in the aircraft industry and aerospace satellites [1,19]. However, one major drawback of silicone oil is its poor boundary lubricating properties, especially for steel/steel contacts, because of its thin films under the condition of a high shear rate [19–21]. Furthermore, due to the wide variations in chemical structure, the majority of traditional lubricant additives used in hydrocarbon-based oil are unable to dissolve or disperse in silicone-based oil [22,23]. The above two disadvantages seriously limit the application of silicone oil in the field of lubrication.

Besides as a base oil, silicone oil also acts as an effective anti-foaming agent to prevent the accumulation of foams in lubricants. The low surface tension, insolubility and fast liquid drainage characteristics enable silicone oil to exhibit outstanding anti-foaming performance. However, a major disadvantage of silicone oil as an anti-foaming agent is its tendency to sediment after long-term storage by reason of its poor dispersibility in lubricants. Therefore, it is important to balance the anti-foaming properties and dispersion stability of silicone oils [22,24]. To improve the anti-foaming performance of silicone oil, researchers have demonstrated many approaches, including modifying the type of functional group, the structure of the side chain, and the ratio and means of conjunction between the polymer and silicone [25–29]. Wu et al. reported the breaking and inhibiting foam performance of a series of silicone oil (F-SO) modified with fluorine-containing alkyl in the mixture of diesel and engine oil and found that F-SO exhibit excellent foam-breaking performance because of its lower surface tension [29]. Ni et al. synthesized a polyether-modified polysiloxane using chloroplatinic acid as a catalyst. Most of the modified products are efficient in foam-breaking because they can disperse more rapidly in the oil system than untreated polydimethylsiloxane [30]. Hassan et al. reported that amide-modified silicone-based antifoams contributed to the formation of unstable bubbles, which can accelerate foam destruction [31]. Previous research has established that reducing the surface tension and improving the dispersity could boost the anti-foaming performance of silicone oil. However, the relationship between structure and performance has not been systematically studied.

To improve the tribological properties and anti-foaming performance, sulfur and phosphorus elements were selected to modify silicone oil. In this work, two kinds of silicone oil, diisooctyl phosphate terminated silicone oil (UCP204) and dioctyl dithiophosphate terminated silicone oil (UCT2003), were synthesized successfully. The introduction of long-chain alkyl phosphate significantly enhances the tribological properties of silicone oil because of the formation of a phosphorus boundary lubrication film. The chemically modified silicone oil also exhibited excellent anti-foaming behavior and storage stability compared with the commercial anti-foaming agent. We discuss the effects of structure on performance and the anti-foaming mechanism of phosphorus-modified silicone oil was also proposed.

2. Experimental

2.1. Materials

Epoxy group-terminated silicone oil (UC-276) was purchased from China Jiaxing United Chemical Co., Ltd. The epoxy value of UC-276 was 0.048~0.052 mol/100 g, the viscosity was 50 cs and the molecular weight was approximately 4000. Diisooctyl phosphoric acid (P204) was purchased from China Luoyang Zhongda Chemicals Co., Ltd., and the density of the P204 was 0.974 g/mL. The dioctyl dithiophosphoric acid (T2003) was purchased from China Liaoning Tianhe Fine Chemicals Co., Ltd., with a density of 0.960 g/mL. A poly(dimethylsiloxane) structure anti-foaming agent (T901) was purchased from China Jinzhou SNDA Chemical Co., Ltd. T901 is a commonly used commercial anti-foaming agent with good chemical stability and anti-foaming performance. It was selected as the control to discuss the anti-foaming properties of chemically modified silicone oil. Polyalpha olefin (PAO4), synthetic ester (5750) and mineral oil (MVI250) were purchased from America Exxon Mobil, France NYCO and China Petroleum & Chemical Corporation, respectively. The used gear oil was collected from a wind turbine gearbox, and was originally produced by Exxon Mobil Corporation.

2.2. Synthesis of the Chemically Modified Silicone Oil

The synthetic pathway of diisooctyl phosphate terminated silicone oil (UCP204) and dioctyl dithiophosphate-terminated silicone oil (UCT2003) is given in Figure 1. The procedure for the synthesis of the silicone oil was as follows: UC-276 (0.02 mol) and P204 (0.04 mol)/T2003(0.04 mol) were added into a 250 mL three-necked flask. The mixture was stirred at 100 °C under reflux conditions for 5 h. In addition, then, rotary evaporation was used to remove impurities. Finally, the viscous colorless liquid UCP204 and light-yellow liquid UCT2003 were obtained.

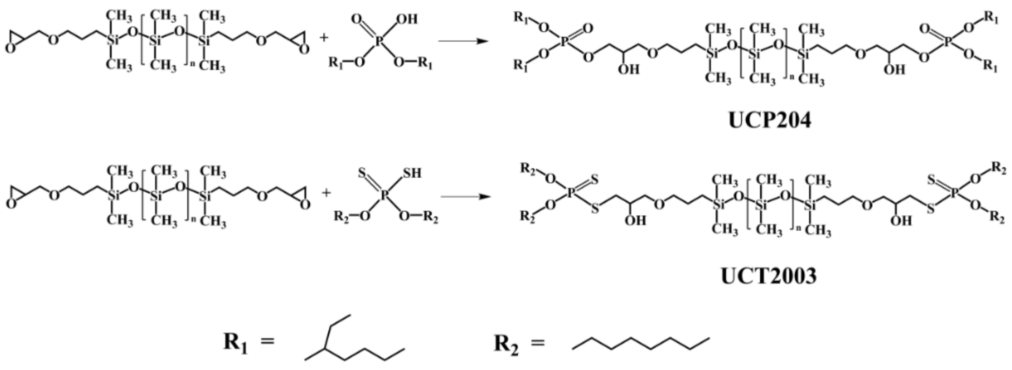


Figure 1. Schematic diagram of the preparation of UCP204 and UCT2003.

2.3. Characterization and Measurements

2.3.1. FTIR Analysis

The Fourier transform infrared (FTIR) spectra of UC-276, UCP204 and UCT2003 were analyzed using a Bruker Tensor 27 FTIR instrument. The spectral range was from 500 to 4000 cm^{-1} with a resolution of 4 cm^{-1}.

2.3.2. Tribological Tests

High-contact stress ball-on-disc oscillating friction and wear tests were carried out to measure the tribological properties using an Optimal-SRV-IV tribometer. The amplitude of the AISI 52100 steel ball is 1 mm. The ball has a diameter of 10 mm and hardness of 626 HV. The φ24.00 × 7.88 mm disc (AISI 52100 steel, hardness 700 HV) is fixed on the tribometer. All the tests were performed for 30 min at a frequency of 25 Hz, a load of 50 N and a temperature of 25 °C. Three drops of the silicone oil were dripped onto the contact area before the friction and wear experiment. In addition, the equipped computer was used to record the friction coefficient curve during the test. To ensure the accuracy of the data, each experiment was performed at least three times.

2.3.3. Surface Analysis

The wear debris formed on the worn surface was thoroughly removed with petroleum ether by sonication for 10 min. The wear scars produced on the discs during the wear experiment were examined by JSM-5600LV scanning electron microscopy (SEM), and the chemical composition (C, Si, O, Fe, P and S) of the worn surface was recorded by energy dispersive spectroscopy (EDS). To analyze the extent of wear and morphology of worn surfaces, a three-dimensional (3D) non-contact surface profilometer (America ADE Co.) was chosen to provide true 3D surface profiling results, wear volumes and topography information in this work.

2.3.4. Anti-Foaming Performance

The anti-foaming performance was analyzed by the foaming characteristics of a lubricating oil tester (Shanghai Shenkai petroleum instruments Co., Ltd., Shanghai, China). The experiments were carried out according to GB/T 12579-2002, which is equivalent to ISO 6247:1998. Specifically, 50 ppm silicone oil was added to the mixture of PAO4 and used gear oil (V/V = 3:1). Transparent solution was obtained after stirring on a magnetic stirrer for 10 min. Then, 190 mL of the lubricant was placed in a 1000 mL graduated cylinder. The foams were generated by blowing air at a flow rate of 94 ± 5 mL/min through a diffuser stone. After 5 min, the air pump was stopped, and the volume of foam was recorded. The initial foam volume and the further evolution of the foam with time were used to characterize the foaminess and foam stability, respectively.

All the equipment used for characterization was calibrated before the tests.

3. Results and Discussion

3.1. Characterization of Chemically Modified Silicone Oil

FTIR was used to characterize the chemical structure of modified silicone oil. FTIR spectra of UC-276, UCP204 and UCT2003 are depicted in Figure 2. As can be seen, the absorption peaks of Si-O-Si at 1017 cm^{-1} and 1095 cm^{-1} exist in all of the silicone oil. In addition, the peak at 1259 can be attributed to Si-CH_3. The bands around 2970–2873 cm^{-1} are associated with the asymmetric and symmetric stretching vibrations of methyl and methylene groups. Meanwhile, the peak at 916 cm^{-1} attributed to the epoxy groups is significantly weakened and replaced by the bands at 3200–3600 cm^{-1}, which correspond to the hydroxyl, indicating the occurrence of the ring-opening of the epoxy groups in silicone oil [32,33]. In addition, the peak of P = S located at 678 cm^{-1} is only observed in UCT2003. FTIR results confirm that epoxy silicone oils were successfully chemically modified.

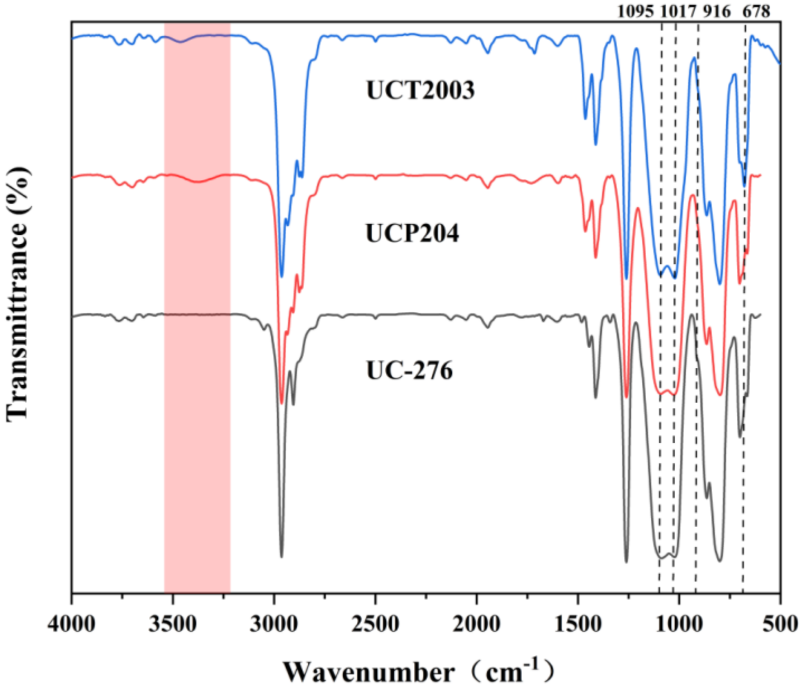


Figure 2. FTIR spectra of UC−276, UCP204 and UCT2003.

3.2. Dispersion Stability Test

The dispersion stability of silicone oil in base oil is an important factor affecting their anti-foaming performances. Here, the dispersion stability of different silicone oil in different types of base oil was studied. Silicone oil UC-276, UCP204, UCT2003 and T901 were added directly into three base oils (mineral oil MVI 250, PAO4 and synthetic ester 5750) with a concentration of 100 ppm and stirred for 30 min at ambient temperature. The images of the combinations are shown in Figure 3. It can be observed that UCP204 and UCT2003 can be completely dispersed in all kinds of base oil. In contrast, the dispersibility of UC-276 and T901 is relatively poor. For base oil with T901, obvious opacity can be observed. We consider the enhanced dispersibility of chemically modified silicone fluids to be closely related to the incorporation of long-chain alkyl groups in silicone fluids, which can enhance the interaction of silicone fluids with base oils. It is worth mentioning that the base oil containing UCP204 and UCT2003 remained clear after 6 months of storage, which further proves the outstanding dispersibility of the chemically modified silicone oil.

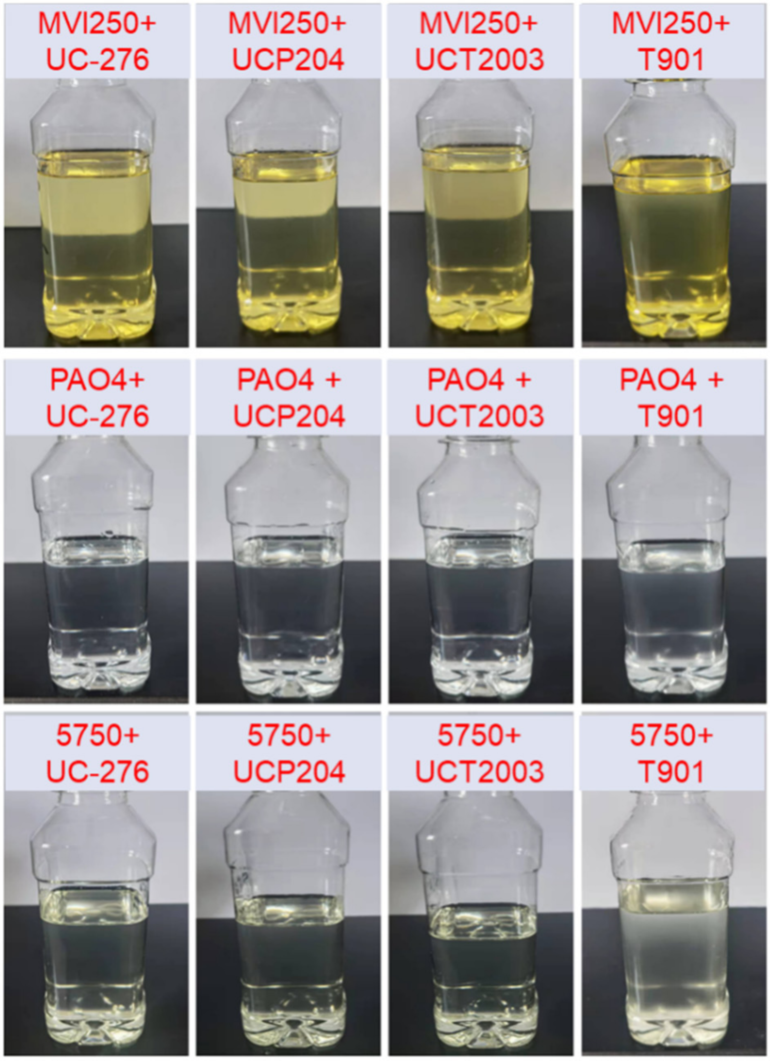


Figure 3. Dispersion stability of silicone oil in different base oils.

3.3. Tribological Properties

The variations in the coefficients of friction (COFs) with test duration and the wear volume of different silicone oils are displayed in Figure 4. It can be observed that the COF curves of all silicone oils rise rapidly within the first 100 s, then fall and become stable. This phenomenon is caused by the high contact pressure and surface roughness of the initial testing period. The friction curve of UC-276 fluctuates significantly, and the mean COF is greater than 0.35. The chemically modified silicone oil shows superior tribological properties compared to UC-276. After a sharp peak in the early stage, the COF of UCP204 and UCT2003 becomes low and stable, which may be due to the formation of a boundary lubrication film initialized by high contact stress. The mean COFs of UCP204 and UCT2003 are 0.131 and 0.152, respectively, representing reductions of 62.7% and 56.7% compared to UC-276. These findings suggest that both sulfur and phosphorus are beneficial in mitigating the frictional properties of silicone oil. Besides friction-reducing performance, chemically modified silicone oil also exhibits good anti-wear properties. It can be observed that the wear volumes of UC-276, UCP204 and UCT2003 are 8.60×10^{-4} mm^3, 5.77×10^{-4} mm^3 and 4.82×10^{-4} mm^3. UCT2003 achieves the lowest wear volume, which can probably be attributed to the formation of a sulfur-phosphorus boundary lubrication film.

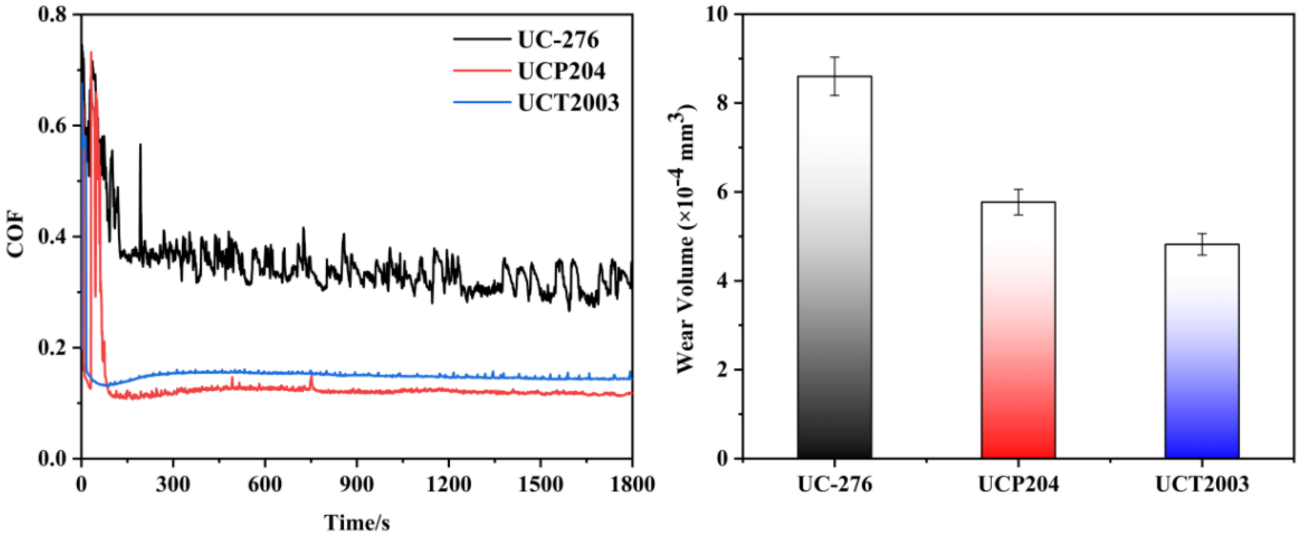
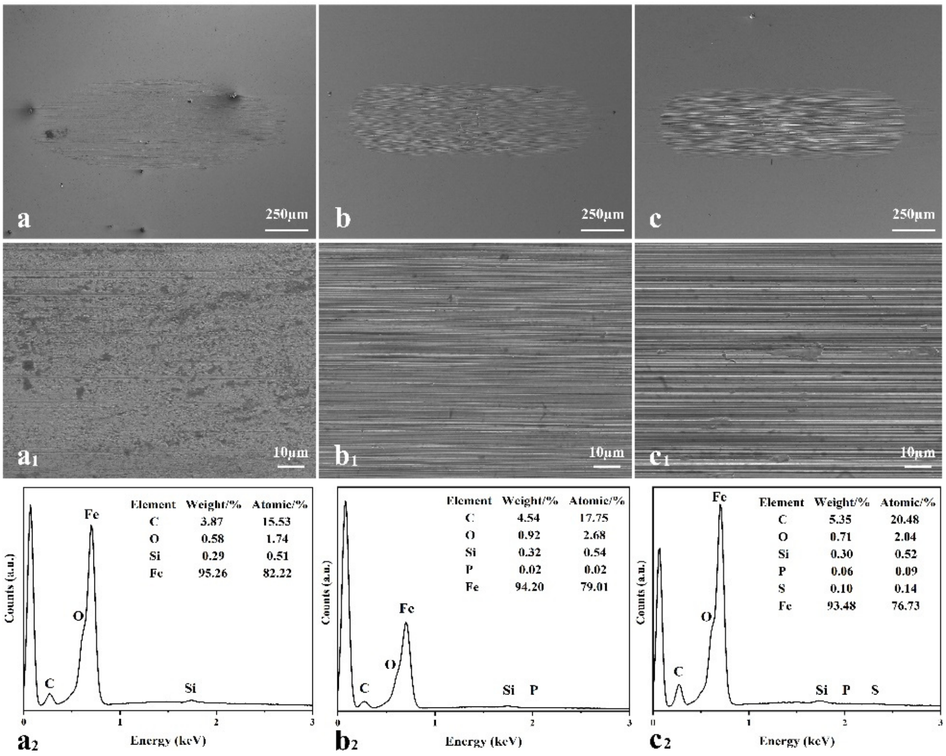


Figure 4. SRV curves and wear volumes of UC−276, UCP204 and UCT2003.

3.4. Worn Surface Analysis

SEM was used to examine the worn surface morphology after tribological tests. Figure 5 shows the SEM morphologies of the worn surface lubricated by UC-276, UCP204 and UCT2003. The SEM images of a_1, b_1 and c_1 are high-magnification images (1000×), and can provide distinct evidence of the abrasive and adhesive wear. It can be seen from Figure 5a,a_1 that the worn surface lubricated by UC-276 presented the largest wear scar, with furrows formed due to the abrasive and adhesive wear. However, when lubricated with chemically modified silicone oil, the wear scar is significantly narrower and smoother. More furrows appear on the worn surface of UCP204 and UCT2003, and the adhesive wear is significantly alleviated.



Element	Weight/%	Atomic/%
C	3.87	15.53
O	0.58	1.74
Si	0.29	0.51
Fe	95.26	82.22

Element	Weight/%	Atomic/%
C	4.54	17.75
O	0.92	2.68
Si	0.32	0.54
P	0.02	0.02
Fe	94.20	79.01

Element	Weight/%	Atomic/%
C	5.35	20.48
O	0.71	2.04
Si	0.30	0.52
P	0.06	0.09
S	0.10	0.14
Fe	93.48	76.73

Figure 5. SEM and EDS results of UC-276 (**a**,$\mathbf{a_1}$,$\mathbf{a_2}$), UCP204 (**b**,$\mathbf{b_1}$,$\mathbf{b_2}$), UCT2003 (**c**,$\mathbf{c_1}$,$\mathbf{c_1}$).

Figure 5a$_2$–c$_2$ displays the typical element composition of the worn surfaces detected by EDS. Compared with pure silicone oil, the worn surface lubricated by UCP204 contains a small amount of phosphorus and the surface lubricated by UCT2003 contains both phosphorus and sulfur. Based on EDS results, it can be concluded that the formation of P- and/or S-containing boundary lubrication film could effectively prevent the direct contact of metal friction pairs and reduce the adhesive wear, thus endowing UCP204 and UCT2003 with good lubricating and anti-wear properties.

3.5. Anti-Foaming Performance

The anti-foaming performances of silicone oil are evaluated in a mixed lubricant composed of used gear oil and fresh PAO 4 base oil. The used gear oil was collected in a wind turbine gearbox, which tends to produce a large amount of foam. Figure 6 gives the foam of different oil at the end of the foam test. As observed in Figure 6, the pure lubricant displayed the worst foam characteristics, with a foam of 480 mL after the test. The addition of UC-276 slightly alleviated the foaming tendency and the foam value was reduced to 420 mL. The lowest foam value of 20 mL was achieved by UCT2003, which was superior to the commercial anti-foaming agent T901.

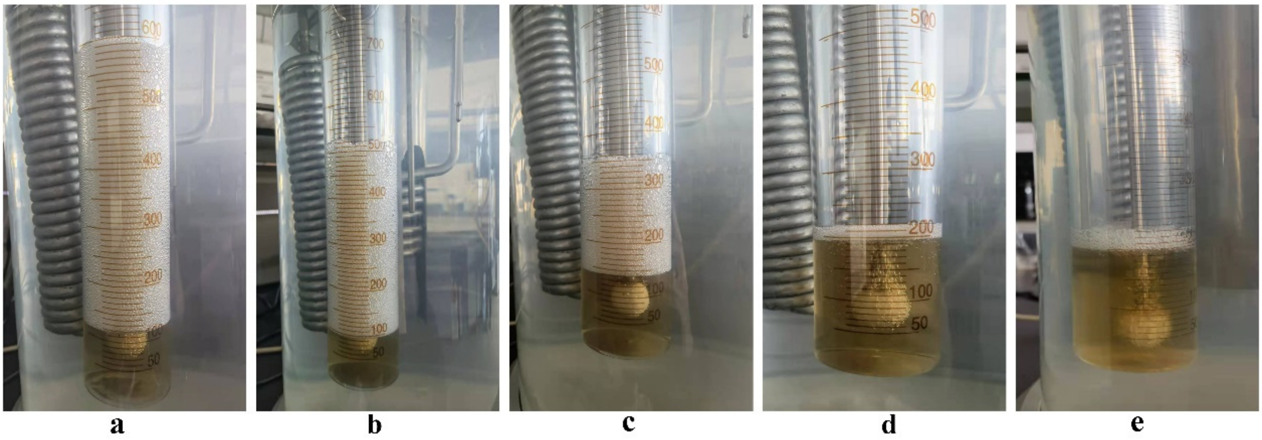


Figure 6. The foam volume of (**a**) pure oil, (**b**) UC-276, (**c**) UCP204, (**d**) UCT2003 and (**e**) T901.

Besides the formation of foam, another crucial feature of the foaming property of lubricants is defoaming speed. As can be seen in Figure 7, it took more than 600 s for all the foams to disappear for pure lubricant. The addition of silicone oil greatly shortened the defoaming time of pure lubricant. For lubricant containing UCT2003, it only took less than 60 s for the foam to completely disappear. The above results revealed that chemically modified silicone fluids exhibit excellent anti-foaming properties, and their anti-foaming capacity is closely related to their chemical structure.

Generally speaking, defoaming of lubricating oil involves two processes, the collision of the silicone oil droplets with foam is the first step in the anti-foaming process [34,35]. Then, silicone oil droplets attach to the air bubble and form a lens to break the foams. The first process is highly dependent on the dispersion status of the silicone oil in lubricant, and the second process is determined by the surface tension of the silicone oil. Therefore, there are two essential factors determining whether silicone oil is suitable as an anti-foaming agent. Firstly, they should have low surface tension, and secondly, the silicone oil should be immiscible, but be well dispersed in lubricants [29].

An automatic surface tensiometer was used to measure the surface tension and the results of UC-276, UCP204, UCT2003 and T901 are 22.1 mN/m, 23.0 mN/m, 21.8 mN/m and 21.0 mN/m, respectively. Judging from the surface tension data, the anti-foaming ability of these silicone oils should follow the order T901 > UCT2003 > UC-276 > UCP204. However, this is not consistent with the anti-foaming test results, which is probably due to the difference in dispersion stability of the silicone oil.

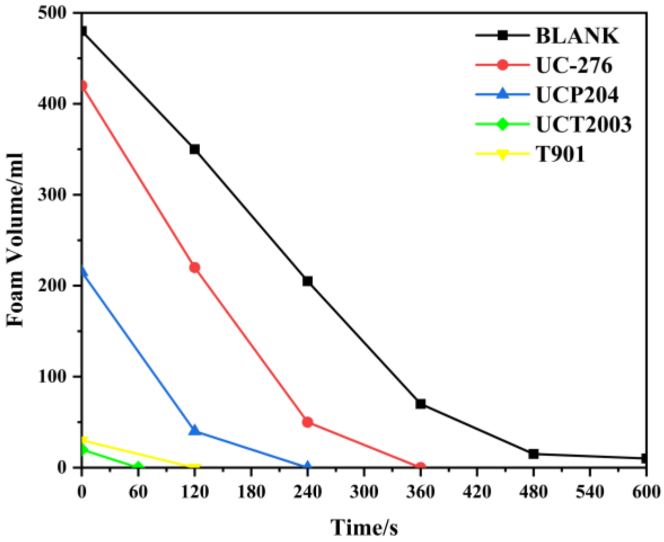


Figure 7. The foam volume as a function of time for lubricants.

Dispersion stability is crucial for a silicone oil type anti-foaming agent, if the silicone oil is completely dissolved in the lubricant, it cannot act as an anti-foaming agent. On the other hand, poor dispersion stability can lead to the sedimentation of anti-foaming agents and consequent deterioration of anti-foaming performance after long-term storage [36–39]. Therefore, how to balance the dispersion stability and solubility is of vital importance for anti-foaming agents. The dispersion stability of these silicone oils in different types of base oils in Figure 3 demonstrates that chemically modified silicone oil UCT2003 and UCP204 exhibit better dispersion stability. To further verify this phenomenon, the anti-foaming performances of silicone oil were tested after 3 months of storage. It was found that the foam volume of UCT2003 and T901 solution is 30 mL and 100 mL respectively. It is obvious that UCT2003 still retains outstanding anti-foaming performance after a long period of storage while the anti-foaming capability of T901 deteriorates with time due to the sedimentation of the additive. Furthermore, the mixture of the gear oil used and UCT2003 remains clear after 3 months of storage, while lubricant containing T901 starts to get cloudy. Therefore, it is reasonable to conclude that the outstanding anti-foaming performance of UCT2003 is attributed to both low surface tension and good dispersion stability.

Considering that UCT2003 has excellent anti-foaming properties, we are interested in understanding its anti-foaming mechanism. Actually, the anti-foaming mechanisms are complex and inconclusive. Many different mechanisms, such as “bridging–stretching”, “bridging–dewetting”, “spreading–fluid entrainment” and “spreading–wave generation” have been proposed [40–43]. We consider that the possible mechanism for UCT2003 is “bridging–stretching”. The unique structure of UCT2003 enables it to possess better anti-foaming characteristics. As shown in Figure 8, the molecular structure of UCT2003 can be divided into two parts, one is the main silicon–oxygen skeleton, and the other is the dialkyl dithiphosphate part. The silicon–oxygen skeleton partially endows the silicone oil with low surface tension which is favorable for the rapid rupture of the air bubble. The lipophilic nature of the dialkyl dithiophosphate parts enhances the interaction with the base oil and aids in the excellent dispersion of UCT2003 in hydrocarbon-based lubricants. When the silicone oil droplets attach to the oil film of the bubble, the alkyl groups cause it to form a biconcave bridge [40], and by virtue of the low surface tension of the UCT2003, the traction of the silicone oil bridge in the radial direction will contribute to forming a weak and unstable film in the middle of the bubble. With the continued spreading of the silicone oil, the film becomes thinner and thinner and eventually ruptures.

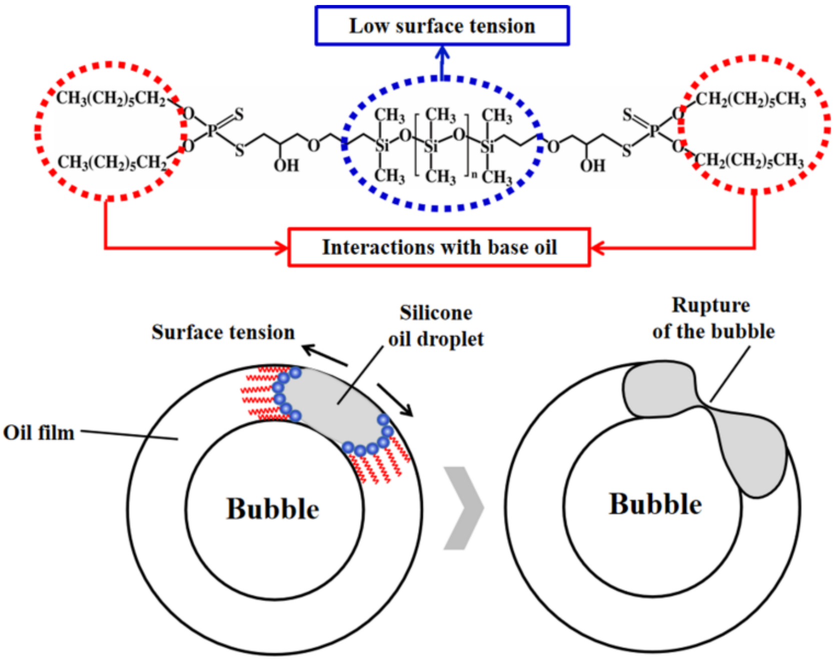


Figure 8. The anti-foaming mechanism of UCT2003.

4. Conclusions

Two kinds of chemically modified silicone oil were successfully synthesized. Tribological tests show that the introduction of phosphorus and/or sulfur can greatly enhance the tribological performance of silicone oil due to the formation of phosphorus and/or sulfur containing boundary lubrication films. The friction coefficients of UCP204 and UCT2003 were reduced by approximately 62.7% and 56.7% compared with untreated silicone oil. In addition, the chemically modified silicone oil UCT2003 exhibits outstanding anti-foaming properties and storage stability compared with commercial silicone oil-based anti-foaming agents. The dialkyl dithiophosphate groups are beneficial for the improvement of the dispersion stability which accelerates the collision chance of the silicone oil droplets with foams and the low surface tension nature of silicone oil leads to the formation of a weak film and the perforation of the foam. In conclusion, the chemically modified silicone oil can be used as a high-performance anti-foaming agent for lubricating oil.

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