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Mini Review

The Preparation and Electroanalytical Investigation of Lubricants: a Mini Review

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Lubricants play a very important role in industry. Different lubricants were synthesized for different working conditions. In this mini-review, we first introduced various synthesis methods for different types of lubricant preparation. Some of our works were included and discussed. Then, electroanalytical-based characterization techniques were summarized.

Keywords: Electroanalytical-based property; Lubricant; Preparation; Tribology

1. INTRODUCTION

Tribology is the study of mutual contact and relative motion between surfaces in relation to surface science and related technology, including the study of friction, wear and lubrication. Tribology involves many research fields such as materials, chemistry, mechanics, physics and mechanics [1–6]. Friction, wear and lubrication occur on the surfaces and interfaces of materials. Friction leads to a large amount of mechanical energy loss, and wear is an important cause of mechanical part failure [7–12]. It has been shown that there is a large economic loss every year from failure of mechanical parts due to friction and wear. Therefore, reducing the economic loss caused by friction and wear is a major subject of social and economic benefits [13–17].

According to different working conditions and lubrication conditions, lubrication can be generally divided into fluid lubrication, elastohydrodynamic lubrication, boundary lubrication and mixed lubrication, among which boundary lubrication is a very common lubrication state [18–21]. Under boundary lubrication conditions, lubricants containing extreme pressure anti-wear additives are often added to improve the friction and wear of friction pairs. This kind of lubricant not only plays the role of lubrication while providing anti-wear protection and improving bearing capacity but also transfers friction heat and cools the friction pair [22–25]. Compared with mineral base lubricants, synthetic lubricants usually have good low-temperature and viscosity-temperature performance, lower volatilization loss, higher thermal stability, fire resistance, radiation resistance and excellent lubrication and wear resistance [26–34].

Recent studies have shown that the addition of nanoparticles and their dispersants to lubricating oil after surface modification can greatly improve the extreme pressure performance of lubricating oil, which is obviously better than that of the additives on the market and can significantly reduce the friction coefficient. The nanoparticle lubricating oil additives used by domestic and foreign researchers mainly include nanometal powders; nanopowders, including diamond, etc.; nano-oxide; nanohydroxide and nanosulfide [35–40]. Due to the simple preparation of nanometre metal powder and its good compatibility with friction pair materials, research on nanometre metal powder is delving deeper and deeper, which provides prospects for broader applications.

Characterization of lubricants involves measuring their properties [41–47]. Recent research shows that electrochemical analysis is an effective method to characterize lubricants [48–52]. Electrochemical analysis is an important part of modern analytical chemistry. It uses the specific electrical or electrochemical properties of substances to analyse the composition and structure of substances. In this review, we first introduce the synthesis of lubricants. Then, we introduce the application of the electrochemical analysis techniques for lubricant property analysis in detail. We also discuss the future direction of the technology.

2. PREPARATION OF LUBRICANTS

2.1 PAO synthetic lubricants

Synthetic hydrocarbon-based lubricants, including polyalphaolefin (PAO synthetic oils) and alkylated aromatics, have better properties than mineral lubricants. Currently, companies mainly prepare PAO base oils with different physical and chemical properties through ethylene oligomerization and hydrogenation [53–56]. Due to the high purity, catalytic activity and selectivity of α-decene and the easy and strict control of process conditions, the performance of α-decene is superior to that of PAO products prepared by wax cracking, fractionation and repolymerization, which are mainly characterized by a high viscosity index, good low-temperature performance and good thermal oxidation stability. Compared with mineral oils, PAO has the advantages of a low pour point, low-temperature fluidity and oxidation stability [57–61]. In practical production, PAO products with different viscosities and vapor pressures can be prepared by regulating the molecular weight distribution of molecular chains to meet different application requirements. In addition, PAO synthetic oils have good lubricity and compatibility with additives.

We have recently studied the tribological properties of molybdenum dialkyl dithiocarbamate (MoDTC) [62]. The results show that molybdenum dialkyl dithiocarbamate can effectively reduce the friction coefficient of base oil and finished gasoline oil and has excellent friction reduction performance. An SEM diagram of the friction surface is shown in Figure 1. The state of the friction surface greatly improves after MoDTC is added. Scratches become shallower when the diameter of the plaque decreases, which shows the good anti-friction properties of MoDTC.

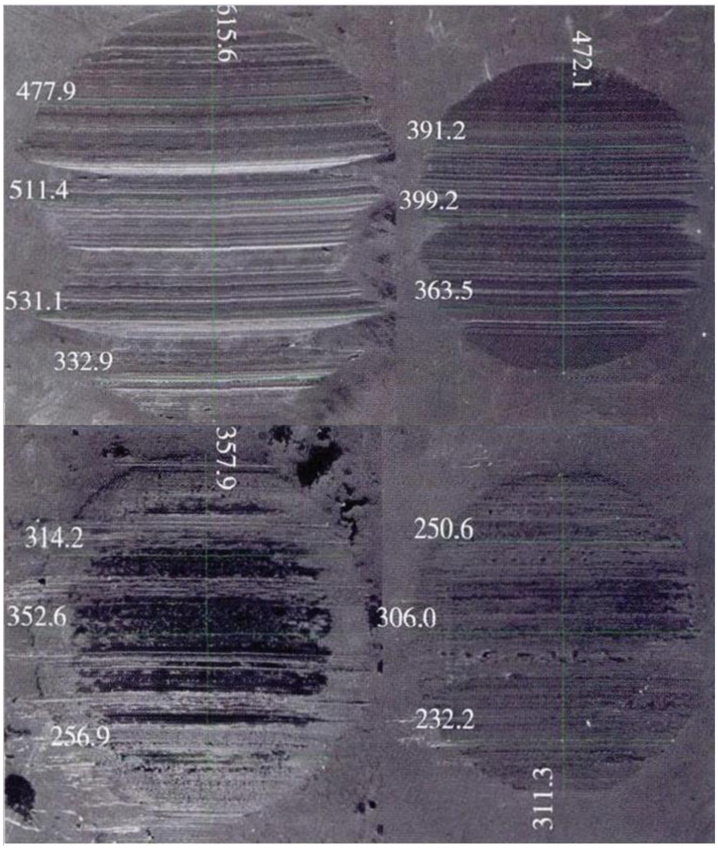

Figure 1. SEM images of two samples in the absence and presence of MoDTC [62]. Copyright 2019 CKNI.

PAO synthetic oils were first used in the military. The U.S. Department of Defense has begun using PAO as a base oil to prepare fire-resistant aircraft hydraulics. The PAO flash point, ignition point and automatic ignition point are significantly higher than those of petroleum-based hydraulic oil. Sophisticated space equipment systems require even more stringent performance requirements for lubricants, among which PAO is required to have excellent low-volatility properties, which in turn gives it an extremely long service life in space agencies; finding materials with this property has been one of the major challenges in space sports equipment for many years [63–67]. Synthetic hydrocarbon-based lubricants can significantly improve the life of lubrication equipment and reduce losses. PAO-based aviation lubricants 3001A and 3001 were studied by Michael et al., and their tribological properties were tested using a spiral orbit tester and a vacuum four-ball friction testing machine, respectively. The results showed excellent lubrication performance under a high vacuum environment. The latest research results indicate that PAO lubricating grease with additives can be used to give precision bearings a long life.

2.2 Alkyl aromatics

Alkylated aromatics mainly include alkyl benzene, alkyl naphthalene and alkylated cyclopentane. Pennzoil Co. Ltd. announced the first alkylated cyclopentane in 1991. Pennzoil announced a new high-performance synthetic hydrocarbon lubricant, whose production starts with cyclopentadiene, a by-product of the ethylene industry; cyclopentadiene is then dehydrated with advanced alcohols and subsequently hydrosaturated. This kind of lubricating oil has the advantages of good low-temperature fluidity, excellent heat and oxidation stability, good sensitivity to additives, and good lubricity and sealing compatibility, so its position in lubricating oil is becoming increasingly important.

Alkylation of aromatic hydrocarbons is mainly performed through the Friedel-Craft alkylation reaction of benzene and naphthalene with halides, alkenes or olefin oligomers. Typical structures of alkylated aromatics are shown in Figure 2. Due to a shortage of petroleum-derived base oil, the early development of alkylation aromatics was promoted. After years of development, researchers gradually realized the advantages of these functionalized liquid compounds, and the development and application of alkylation aromatics also attracted increasing attention [68–73].

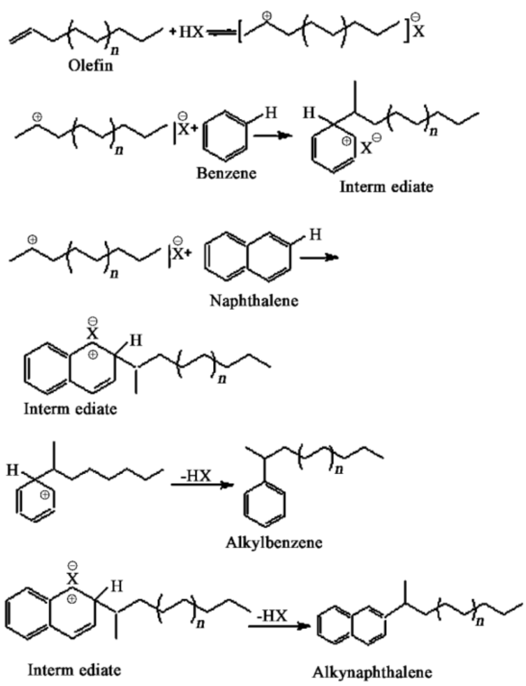

Figure 2. Syntheses of alkylated aromatics.

The structure of alkyl aromatics has a significant effect on its properties. For example, the electron-rich naphthalene rings in alkyl naphthalene capture hydroxyl or peroxy groups from hydrocarbon oxidation, which prevents further oxidative degradation of lubricants. Dialkyl benzene is widely used in industrial and metal working fluids due to its low price, absence of sulfur and high thermal stability, and it is also widely used in rolling bearings. Although synthetic alkyl benzene is less comprehensive than PAO, its compatibility with additives, low pour point, good oxidation resistance and high thermal stability give it great advantages at high and low temperatures [74–78].

2.3 Ester-based Lubricant

Ester synthetic oil is an organic compound composed of ester functional groups that is dehydrated by esterification under the action of organic acid and organic alcohol with a catalyst. According to the reaction product, the base ester and the location, the oils can be divided into double ester, polyol ester, aromatic carboxylic acid ester and complex ester categories. Because of their special molecular structure, ester synthetic oils have many advantages, such as high- and low-temperature performance, viscosity-temperature performance, thermal oxidation stability, lubricity and low volatility [79–86]. At the same time, ester oils also have the advantages of having excellent biodegradability and low toxicity and being renewable raw materials, which can allows them to meet the requirements of the current industrial development of new lubricating materials and be one of the most valuable synthetic lubricants with application prospects.

We recently investigated the friction-reducing and anti-wear properties of four kinds of thiophosphate in different base oils [87]. The results show that the friction-reducing property of thiophosphate was much better in Yubase 6 and PAO base oils than in different base oils. The anti-wear property of thiophosphate in Yubase 6 is superior to that in PAO and PAG. The adsorption behaviour of thiophosphate on a stainless-steel surface in hexane and ethanol was investigated using a quartz crystal microbalance with dissipation (QCM-D) monitoring. The adsorption film of thiophosphate was much more stable in hexane than in ethanol. It is not easy to desorb thiophosphate in hexane. Thus, the friction-reducing and anti-wear properties of thiophosphate in mineral oil were better than those in hexane.

Figure 3 is the SEM image of the worn surface of the ball and disc in different base oils. Take Sample A as an example to observe the pattern of wear after the friction test. It can be seen from the figure that the abrasive surface of Sample A in Yubase 6 is relatively smooth. The ground surfaces of the samples in PAO and PAG were relatively rough, with large furrows and scratches and obvious wear. By comparison, the wear resistance of Sample A in Yubase 6 mineral oil is better.

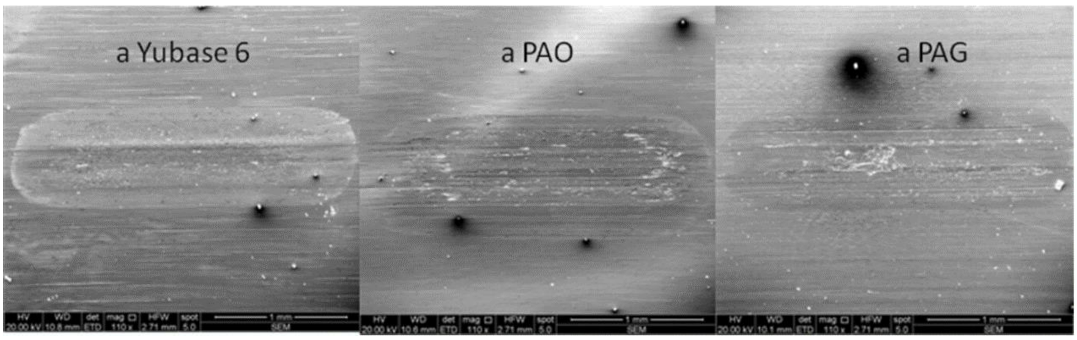

Figure 3. The wear pattern of thiophosphate a in Yubase 6, PAO and PAG base oils [87]. Copyright 2019 CKNI.

Synthetic ester lubricants have been widely used in the field of aviation because of their flexible structure and excellent comprehensive properties. At present, ester lubricating oil is not only used in high-tech national defence but is also widely used in automobile, petrochemical, metallurgy, machinery and other industrial fields [88–91].

2.4 Perfluoropolyether

Fluorogenic lubricants are synthetic lubricants containing fluorine elements, the most important of which are perfluorocarbon hydrocarbons, fluorochlorocarbon hydrocarbons and PFPE. PFPE is a synthetic polymer because the molecules do not contain hydrogen, so it has strong oxidation resistance, good lubrication properties, and good viscosity temperature properties along with a low freezing point. In addition, PFPE products have a high boiling point, so the evaporation loss is small; at normal temperature, it is a liquid and has been used as a space machine lubricant for more than 40 years [92–95]. All reported PFPE structures are linear, as shown in Figure 4.

PFPE plays an important role in some special fields because of its excellent viscosity-temperature performance, low pour point, low evaporability and good polarity [96–100]. It is widely used in the aviation, aerospace and military industries. The high stability, corrosion resistance and wear resistance properties allow it to be used in a harsh environment for a long-term use as a lubricant, so it has been widely used in engineering and industry.

Type	Molecular formula
Demnum	$CF_3-CF_2-CF_2-O(CF_2-CF_2-CF_2-O)_m CF_2CF_3$
Krytox	$CF_3-CF_2-CF_2-O(C(F)(CF_3)-CF_3-O)_m CF_2-CF_3$
Fomblin Y	$CF_3-O-(O(F)(F)-CF_3-O)_m-O(CF_2-O)_n-CF_3$ $m/n = 40/1$
Fomblin Z	$CF_3-O-(CF_2-CF_2-O)_m-(CF_2-O)_n CF_3$ $m/n = 2/3$

Figure 4. Types and molecular formula of PFPE.

2.5 Ionic liquid lubricant

Ionic liquids have been emerging synthetic lubricants in recent years that have the advantages of extremely low volatility, a low melting point, incombustibility, antioxidation properties, and good thermal stability; they also do not generate pollution and hence may be an ideal new type of synthetic lubricant [101–105]. This is a promising multi-functional lubricant, but their corrosion and oxidation resistance have not been explained well. The main reason is that most of the current ionic liquid lubricants use alkyl imidazole as a cation and hexafluorophosphate or tetrafluoroborate as an anion. Although this kind of ionic liquid also has good lubrication and anti-wear performance, its anion hydrolyses easily to produce phosphoric acid or boric acid in the presence of water and releases hydrogen fluoride, thus causing corrosion of the metal friction pair, which limits the practical application of this kind of ionic liquid [106–109].

To reduce the corrosion problem of ionic liquid and retain its excellent lubrication performance, it is necessary to design and replace different cations and anions to improve the purity of the prepared ionic liquid to achieve the purpose of reducing corrosion and improving oxidation resistance. Figure 3 shows the molecular structures of several typical ionic liquids. The application of ionic liquid lubricants is still in its infancy. From the performance of ionic liquids, they are expected to be used as a lubricant, lubricating film and lubricating additive in harsh environments.

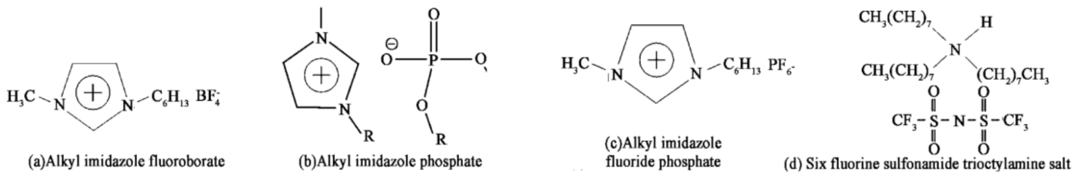

Figure 3. Molecular structure of some ionic liquid lubricants

3. ELECTROANALYTICAL-BASED PROPERTY INVESTIGATION OF LUBRICANTS

3.1Conductivity method

A transient current is generated when a steady voltage is applied to electrodes placed in lubricants. With the migration of charged particles in a lubricant, the current between electrodes will gradually become stable. A conductivity method is used to evaluate the degradation of lubricant by the difference between the test current and stable current [110–113]. According to the changes, the degradation mechanism of lubricants can be divided into three categories: the degradation of lubricants is caused by an increase in charged particles; the degradation of lubricant depends on the increase in conduction particles [114–117]; and the degradation of lubricants is related to the formation of large micelles. It was found that the conductivity between electrodes was affected by the aggregation structure of polar particles, such as alkaline additives and carboxylic acid formed by oxidative degradation, and the conductivity had a good linear relationship with pH. The conductivity was the smallest when the acid value of lubricant was equal to the base value. The reciprocal of conductivity and temperature show a good linear relationship with the dynamic viscosity of lubricant [118–122]. The oxidative degradation of lubricants can be effectively evaluated and detected by the conductivity method.

3.2 Dielectric constant method

With the accumulation of oxidation products and thermal degradation products of lubricants, foreign pollutants continue to increase, and polar compounds and polarized molecules also increase, which leads to changes in the dielectric constant of lubricants [123–127]. The introduction of metal wear particles and other conductive compounds caused by normal friction and abnormal wear of the machine will also change the dielectric properties of the lubricant and the dielectric constant. The dielectric constant of lubricant depends on the composition and content of base oil, additives and contaminants. The results show that there is a good correlation between the change in the dielectric constant and the mileage of the lubricant and the changing trend of the physicochemical index. The change in the dielectric constant reflects the deterioration of lubricants. The change in the dielectric constant of lubricants can be used to evaluate the performance of engine oil, monitor the change of lubricant properties, and determine the best oil change period of engine lubricants [128–133].

3.3 Gas sensor method

In the oxidation and degradation of lubricating oil, the generation of low-molecular-weight gaseous oxidation products is always present. Therefore, a cheap, solid metal oxide semiconductor gas sensor can be used to simply and continuously monitor the oxidation and degradation of lubricating oil [134–138]. According to the output voltage of the detector, the gas sensor method can reasonably predict the oxidation induction period of lubricating oil. Before the induction period, the sensor output is relatively small and stable. After the induction period, the sensor signal will rapidly increase due to the oxidation of lubricating oil and the formation of a large amount of low-molecular-weight gases. The difference between the time when the tangent line to the oxidation gas release curve at its maximum rate intersects with the baseline and that time at the maximum rate is the oxidation induction period of the lubricating oil. There is a good correlation between the gas-sensitive sensor method and the traditional method to measure the antioxidant stability of lubricating oil. The gas sensor method has the advantages of a smaller sample size along with less time and cost than other methods.

3.4 Voltammetric method

Voltammetry is an electrochemical analysis technique based on the relationships of current, voltage and time for a microelectrode. For voltammetry, the base agent and antioxidant are dissolved in electrolyte solution, and the working electrode, reference electrode and auxiliary electrode are inserted [139–141]. Then, a voltage that increases linearly with time is applied to the auxiliary electrode. When the electrode voltage is equal to the oxidation potential of the lubricant antioxidant in the electrolyte system, the antioxidant will undergo electrochemical oxidation, leading to an increase in current at the working electrode, thus resulting in the voltage-current characteristic curve of the lubricant [142, 143]. The voltage value of the characteristic voltage-current peak depends on the type of antioxidant, and the peak amplitude depends on the concentration of the oxidant. The sensitivity of antioxidants in voltage-current analysis is related to the type of lubricant antioxidant, the electrolyte system and the voltage-current analysis parameters [144, 145]. The potential of electrochemical oxidation of different lubricant antioxidants depends on the type of antioxidant. Voltammetry can not only determine the antioxidant activity of antioxidants but also predict and evaluate the service life of lubricants [146–148].

The variation in the content of iron, zinc, copper, chromium, tin, lead and other metal elements in lubricants is an important index for the performance of lubricants and engines. After ashing and acid treatment, the content of metal elements in a lubricant can be determined by dissolution voltammetry in a specific electrolyte system. This method is simple and fast.

3.5 Impedance method

The impedance method is an electrochemical analysis method that takes an alternating potential of small amplitude as an excitation signal [149, 150]. On the one hand, due to the disturbance of the system by the small amplitude electrical signal, a large impact on the system is avoided; on the other hand, this signal also makes a disturbance, and the response of the system is approximately linear. The impedance spectrum results from a frequency domain measurement technique [151–153]. It can obtain more dynamic information and electrode interface structure than other conventional electrochemical method by measuring the impedance spectrum with a wide frequency range. Even for simple electrode systems, time constants can be obtained from measurements, and information about electrodes, inter-electrode solution impedance, double-layer capacitance, and electrode reaction impedance can be obtained at different frequency ranges [154,155]. Therefore, impedance spectroscopy is a kind of electrochemical analysis method that chemical workers attach great importance to, and its scope of application is continuously expanding. The impedance spectrum is also widely used in lubricant monitoring. The impedance characteristics and properties of lubricants at high, medium and low frequencies can be found, and wear particles, pollutants and oxidation degree have a good correlation [156–159].

At low frequencies, the response mainly comes from the conductivity of the lubricant. At this time, moisture is the main factor affecting the conductivity of the lubricant, and the moisture content is proportional to the conductivity drift [160, 161]. At high frequencies, the response is mainly due to the change in the dielectric constant of the lubricant caused by wear of metal particles. Impedance spectra respond to oxidation products at both low and high frequencies [162, 163]. Using the obtained impedance spectrum information and appropriate stoichiometric methods allows for satisfactory predictions to be made for the moisture, viscosity, oxidation products and iron content of the measured lubricants [164–166].

4. CONCLUSION

In this review, we first introduce preparation methods for synthesizing lubricants. Then, we focus on the recent development of characterization methods. Several advanced analysis techniques for lubricant property investigations are introduced.

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