A comparative study on tribological properties of leaf-surface waxes extracted from coastal and inland plants

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Abstract

Purpose – This paper aims to explore the leaf-surface wax as green lubricant additive and compare the tribological properties between coastal and inland leaf-surface waxes of the same species plant.

Design/methodology/approach – The leaf-surface waxes were extracted from the leaves of *Robinia pseudoacacia* cv. Idaho and *Populus nigra* in coastal and inland areas, and then the compositions of the four kinds of leaf-surface waxes were characterized using a gas chromatography–mass spectrometry. The tribological properties of these leaf-surface waxes as lubricant additives in the base oil of synthetic ester (SE) were investigated by an MFT-R4000 reciprocating friction and wear tester. As well as the surface morphologies and chemical compositions of the wear scars were characterized by a scanning electron microscope and time-of-flight secondary ion mass spectrometry, respectively.

Findings – The results indicate that all the leaf-surface waxes as additives can effectively improve the friction reduction and anti-wear performances of SE for steel–aluminum friction pairs. Therein, coastal leaf-surface waxes have better tribological performances than inland leaf-surface waxes, which are attributed to that the leaf-surface waxes extracted from coastal plants can form a better protective film on the worn surface throughout the friction process.

Originality/value – This paper investigated a new kind of environmentally friendly lubricant additive and compared the tribological properties of the leaf-surface wax extracted from coastal and inland plants. The associated conclusions can provide a reference to explore the tribological performances of leaf-surface wax as green lubricant additive.

Keywords Tribological properties, Coastal and inland, Green lubricant additive, Leaf-surface wax

Paper type Research paper

1. Introduction

Lubricant is an engineering material which has been widely introduced to reduce friction and wear between surfaces in mutual contact. In general, it may also have the function of removing heat, corrosion control, providing a liquid seal and so forth. Over the past decades, the global demand for lubricant has increased at a rate of 2 per cent per year, and because of the improper disposal, spills, volatility, etc., it is estimated that more than 50 per cent of the lubricants enter the environment every year (Mannekote *et al.*, 2017). The most of the lubricants enter the environment are petroleum-based, and the additives added into the lubricants contains sulfur and phosphorus, which could generate a bad influence on the soil, air, water and human and plant life (Nehme, 2004; Spikes, 2008). Therefore, it is imperative to explore environmentally friendly and nontoxic lubricant.

Numerous studies have suggested that synthetic ester (SE) is a kind of superior environmentally friendly lubricating oil and has been widely used in industrial lubrication (Eisentraeger *et al.*, 2002). Meanwhile, there is also some research about the lubricant additives which can improve the tribological performances of synthetic ester. Khemchandani *et al.* (2014) mixed safflower oil with synthetic ester and found that the mixture can effectively replace mineral oil-based lubricants. Waara *et al.* (2004) and Jiménez and Bermúdez (2008) found that fatty acids and imidazolium ionic liquids all can greatly improve the friction reduction and anti-wear abilities of synthetic ester. However, it is also reported that not all lubricant additives are suitable for synthetic esters. Borda *et al.* (2017) investigated the tribological properties of mineral oil and SE additivated with copper nanoparticles, and the results showed that nano-copper particles can improve the tribological properties of mineral oil but is not suitable for synthetic ester. Therefore, it is necessary to further carry out the study about the environmentally friendly additives for synthetic ester.

Leaf-surface wax can protect leaves from the damage of bacteria, fungus, ultraviolet radiation and water loss, and it is a kind of natural material which has complete biodegradability and nontoxic. Leaf-surface wax is a complex mixture which is usually composed of alcohols, hydrocarbons, esters and so forth (Kolattukudy, 1970; Vermeer *et al.*, 2003; Ji and Jetter, 2008).

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These single ingredients all have been evaluated as lubricants, and the results show that they all have some certain friction reduction and anti-wear abilities (Hu and Liu, 1998; Song, 2002; Wang *et al.*, 2012). Therefore, the leaf-surface wax as a mixture composed of these compounds may also hold the potential as environmentally friendly additives to improve the tribological properties of synthetic ester. Recently, some research suggests that leaf-surface wax as additive exhibited some certain friction reduction and anti-wear abilities (Xia *et al.*, 2015; Xia *et al.*, 2017).

In this work, the leaf-surface waxes were extracted from *Populus nigra* and *Robinia pseudoacacia cv. Idaho* as additive in synthetic ester. Taking into consideration that the plant growth environment has a great influence on the compositions of the leaf-surface wax, therefore, this study also compared the tribological properties of the leaf-surface wax extracted from the same species plant growing in coastal and inland. Additionally, a gas chromatography–mass spectrometry (GC-MS) was used to identify the compositions of the leaf-surface wax, and a scanning electron microscopy (SEM) and time-of-flight secondary ion mass spectroscopy (TOF-SIMS) were used to characterize the worn surface to analyze the possible lubrication mechanisms in detail.

2. Experiments

2.1 Materials

The SE was obtained from Changsha Zhongcheng Petrochemical Co., Ltd, and its parameters are shown in Table I. Chloroform and petroleum ether were commercially obtained from Sinopharm Chemical Reagent Co., Ltd, and they were of analytical grade and without any further purification. CPN and CRP were obtained from the leaves of the saline–alkali soil on the Bohai Bay, China, whereas IPN and IRP were picked up from the inland area of the same city. Figure 1 presents the images of leaf samples.

The leaf-surface wax was extracted in a simple procedure. First, the collected leaves were rinsed with water to remove the dust and surface impurities and then dried in natural air. Second, the clean leaves were soaked in chloroform solution for 5-15 s at room temperature. After that, the mixed solution was filtered three times with filter paper to remove the possible solid impurities. Third, the filtered solution was placed in fuming cupboard to remove the chloroform. Finally, the precipitated substance would be the target product, and Figure 2 gives the samples.

The chemical compositions of leaf-surface waxes were characterized by using a GC-MS (Agilent Technologies Inc.). The leaf-surface wax was injected on-column into a constant

Table I The parameters of tested synthetic ester

Parameters	Value
Viscosity (mm^2/s)	
40°C	50-60
100°C	8.5-10
Viscosity index ≥	130
Acid value (mgKOH/g) ≥	0.2
Flash point (°C) ≥	270
Pour point (°C) ≥	−40

Figure 1 Leaves of CRP, IRP, CPN and IPN

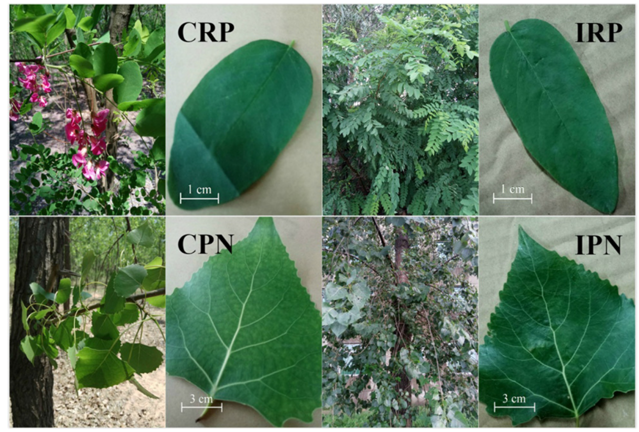
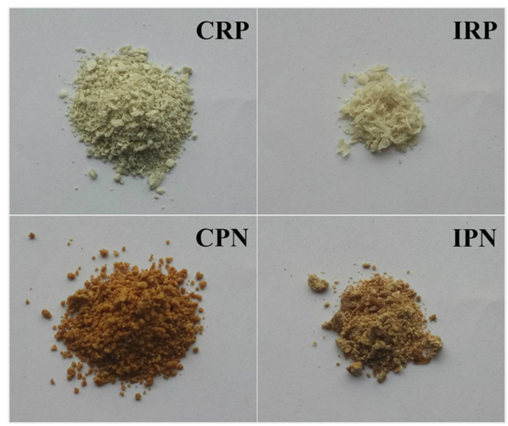


Figure 2 The leaf-surface waxes extracted from different leaves



flow of He of 1.2 mL/min. The initial temperature of the GC oven was set at 80°C, and the heating rate was set at 4°C/min. When the temperature reached 290°C, it was maintained for 20 min, and then a 5,973 N mass selective detector (EI 70 eV; ionization source temperature 230°C) was used to identify the chemical composition of the leaf-surface wax.

The lubricants were prepared by mixing the lubricant additives of leaf-surface waxes and base oil of SE. The mass fraction of lubricant additive was 0.4, 0.8, 1.2, 1.6, 2.0 and 2.4 per cent, respectively.

2.2 Friction and wear test

A MFT-R4000 friction tester was used to investigate the friction reduction and anti-wear abilities of the leaf-surface waxes. The friction pair was a ball-on-disk configuration consisted of the upper ball (AISI 52100 steel, diameter 5 mm, hardness 710 Hv) and the lower disk (Al 2024, Ø 24 mm × 8 mm, hardness 136-141 Hv). The upper ball was driven to slide reciprocally against the lower disk at a stroke of 5 mm and a frequency of 5 Hz. The applied load was 20, 30 and 40 N, and the corresponding Hertzian contact area and pressure were 0.0232, 0.0304 and 0.0368 mm^2 and 1.29, 1.48 and 1.63 GPa, respectively. All the disks were polished with different grades of diamond paste to achieve a roughness of about 0.05 um. Before and after each friction test, the upper balls and lower disks were ultrasonically washed in petroleum ether for 10 min. The test temperature and humidity were 22-29.5°C and 26-39 per cent. About 0.5 g of to-be-tested lubricants was dropped into the contact zone of the friction pair. Each friction test was repeated thrice to get a more responsible data, and the mean values of the coefficients of friction (COFs) and wear scar widths (WSWs) with error bars were provided as well.

2.3 Characterization of wear scar

After friction test, an optical microscope was used to characterize the WSW on the lower disk. The high magnification surface morphologies of the wear scars were obtained by an EVO-18 scanning electron microscope (Zeiss, Germany) under an accelerating voltage of 10 kV with a 7.5 nm beam current. The chemical compositions of wear scars on disks were analyzed by using a TOF-SIMS (IONTOF, Germany). The acceleration voltage was 30 kV, and the analysis area was 300 μm × 300 μm.

3. Results and discussion

3.1 Chemical compositions of the leaf-surface waxes

As detailed in Table II, the major chemical compositions of tested samples are hydrocarbons, alcohols, esters, ketones, ethers and phenols. It is also found that CRP has more alcohols, hydrocarbons, esters and phenols, but fewer ethers and ketones than IRP, whereas CPN has fewer hydrocarbons and phenols but more alcohols, esters, ketones and ethers than IPN. The lengths of carbon chains are mostly 18-37, and the content of compositions more than 5 per cent are shown in Table III.

3.2 Friction reduction and anti-wear abilities

Figure 3 shows the COFs and the WSWs of the friction pair lubricated by SE containing different mass fractions of CRP or IRP at 20 N and 5 Hz. It was clearly visible that the COF and WSW of the SE with the addition of lubricant additives were greatly lowered as compared with pure SE, and the COFs and WSWs decreased first and then increased with the additive concentration growing. When the concentration of additive was 1.2 per cent, coastal and *inland Robinia pseudoacacia cv. Idaho* (CRP and IRP) all had the lowest COFs (0.065 and 0.068) and WSWs (0.232 and 0.262 mm). It was also found that CRP always exhibited lower COF and WSW than those of IRP at all concentrations. Compared with pure SE (0.085 and 0.43 mm), the addition of 1.2 percent CRP decreased the COF and WSW by 22.84 and 46.12 percent, respectively, indicating the superior friction reduction and anti-wear abilities.

Table II The chemical compositions and contents of wax samples (Wt.%)

	CRP (%)	IRP (%)	CPN (%)	IPN (%)
Alcohols	17.492	5.426	3.989	–
Hydrocarbons	53.993	10.312	39.839	85.513
Esters	16.742	–	26.274	6.344
Ketones	0.351	1.726	8.003	1.553
Ethers	–	35.552	2.269	2.306
Phenols	0.496	–	1.710	2.765
Others	10.926	46.984	17.916	1.519

Table III The content of the main compositions (>5 per cent, Wt.%)

Samples	Composition	Content (%)
CRP	Cyclooctacosane	28.816
	1,19-Eicosadiene	14.347
	.beta.-Amyrin	13.135
	Urs-12-en-24-oic acid, 3-oxo-, methyl ester, (+)–	6.037
	Terephthalic acid, di(4-octyl) ester	6.000
	Heptacosane	5.504
IRP	Oxirane, heptadecyl-	23.472
	Benzo[b]naphtho[2,3-d]furan	12.100
	Hentriacontane	10.312
	1-Heptacosanol	5.426
CPN	Nonacosane	19.092
	Terephthalic acid, 2-ethylhexyl octyl ester	10.046
	1,19-Eicosadiene	6.669
IPN	Nonacosane	41.252
	Tetracosane	15.302
	1,19-Eicosadiene	10.261

Figure 4 further investigated the tribological properties of SE containing 1.2 per cent CRP or IRP under different applied loads. Observing Figure 4(a) and (b), the COFs and WSWs of all the lubricants increased with the applied load ramping from 20 to 40 N, and CRP still exhibited little better tribological properties than IRP under different loads. The biggest improvements on friction reduction and anti-wear abilities were achieved by SE + 1.2 per cent CRP which, respectively, reduced COF and WSW by 26.92 and 42.12 per cent under the applied load of 30 N. The results shown in Figure 3 and Figure 4 all suggest that CRP exhibited better tribological properties than IRP.

Figure 5 gives the tribological properties of another two kinds of leaf-surface wax coastal and inland *Populus nigr* (CPN and IPN) under applied load of 20 N and a frequency of 5 Hz. It was similar to the results shown in Figure 3: when the additive was used, the COFs and WSWs decreased first and then increased with the increasing additive concentrations. And SE containing 1.6 per cent CPN or IPN exhibited better tribologcial properties than others. It was also found that CPN exhibited lower COF and WSW than those of IPN under all the tested conditions. Compared with pure SE, 1.6 per cent CPN reduced the COF and WSW by 28.75 and 32.56 per cent, respectively.

Figure 6 presents the COFs and WSWs of friction pair lubricated by SE + 1.6 per cent CPN or IPN as a function of loads at 5 Hz. It was observed that the growing load resulted in an increase in COFs and WSW, whereas CPN always exhibited better tribological properties than IPN under all the tested loads. Compared with pure SE, CPN reduced COF and WSW by 28.32 and 34.38 per cent under the load of 30 N, indicating outstanding friction reduction and anti-wear abilities.

3.3 Wear scar morphology

The results shown in Figures 3-6 suggest that the leaf-surface wax extracted from the coastal plant leaves exhibited better tribological properties than that extracted from the inland plant leaves. To explore the lubrication mechanisms, the

Figure 3 (a) COFs and (b) WSWs of different mass fractions of CRP and IRP additives at 20 N

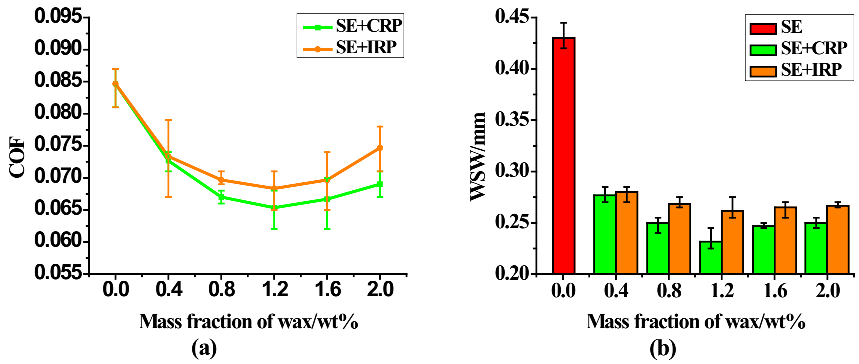


Figure 4 (a) COFs and (b) WSWs of CRP and IRP additives under different load

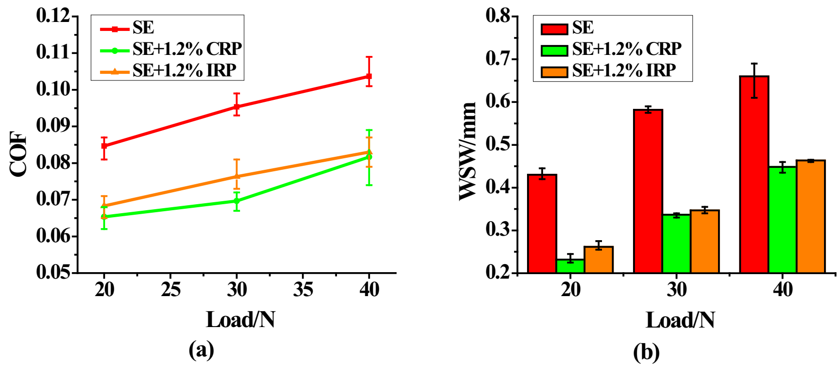


Figure 5 (a) COFs and (b) WSWs of the different mass fractions of CPN and IPN additives at 20 N

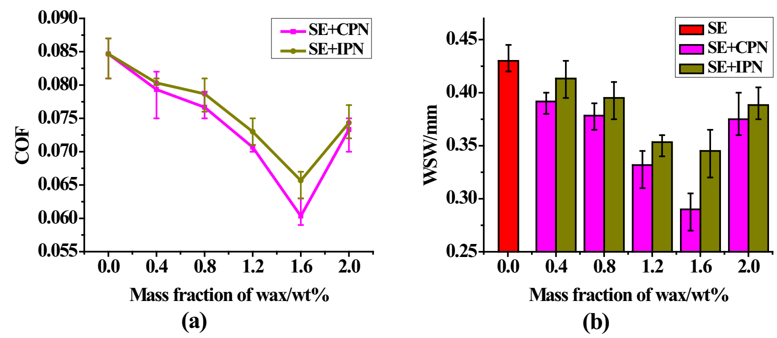
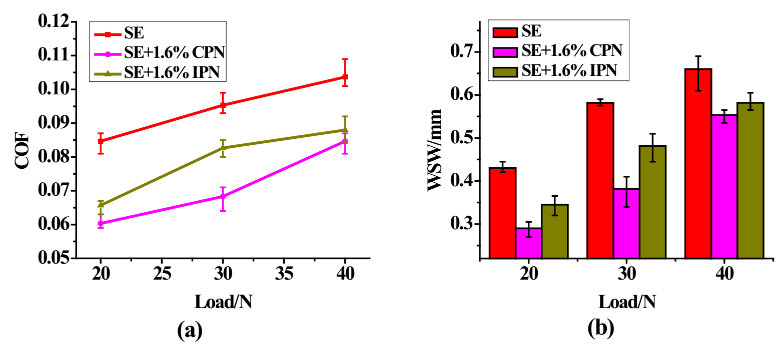
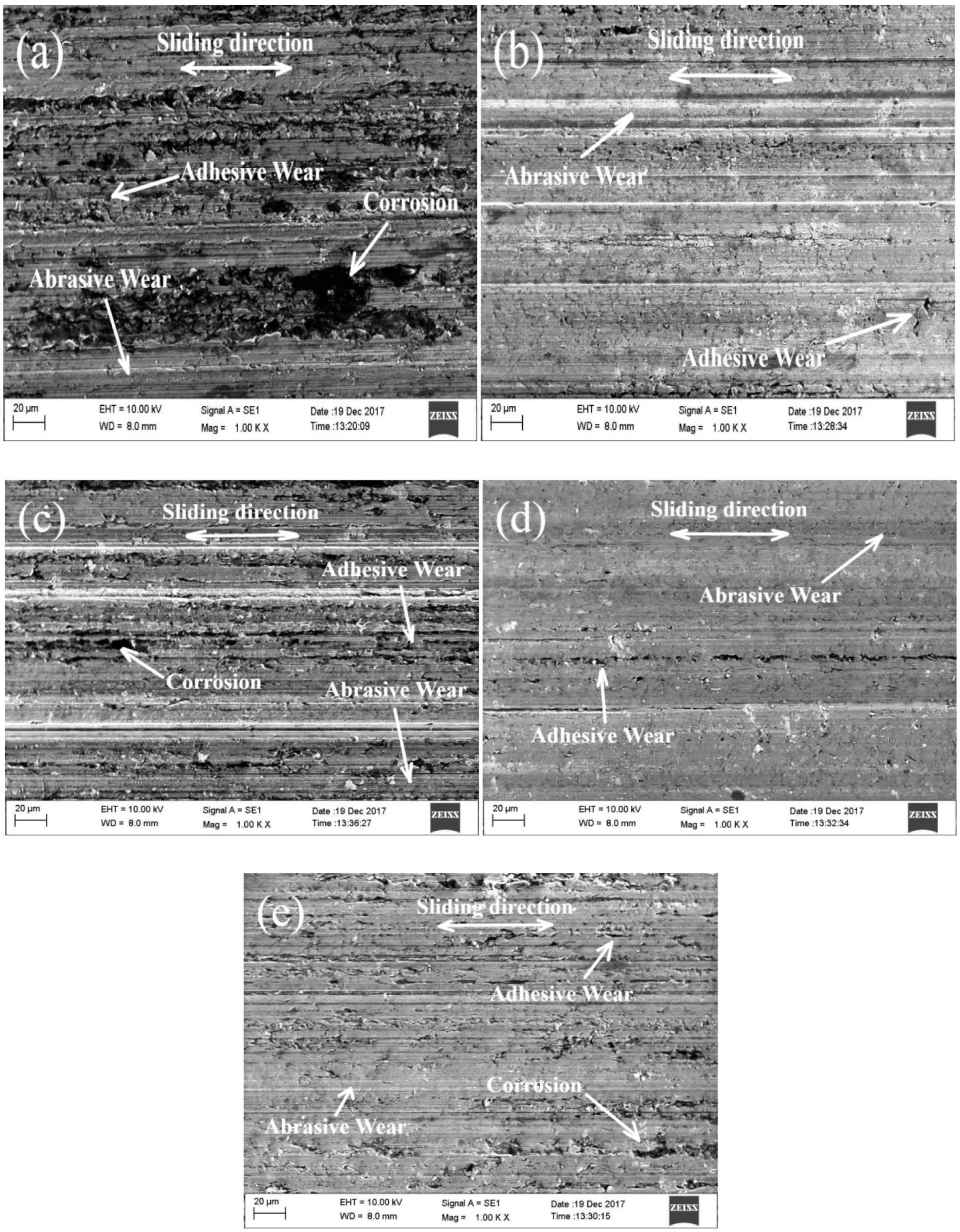


Figure 6 (a) COFs and (b) WSWs of CPN and IPN additives under different load



morphologies of the wear scars were obtained by a SEM, and Figure 7 gives the results. Figure 7(a) gives the SEM images of the wear scar lubricated pure SE. It can be seen that there were a lot of deep furrows and corrosion pits, indicating that severe abrasive and adhesive wear and tribocorrosion occurred during the friction test. In addition, there were some spalling and plastic deformation appearing in the occasion (Pritchard, 1970; Fellah *et al.*, 2017; Fellah *et al.*, 2014). Compared with Figure 7(a), the worn surfaces [Figure 7(b-e)] lubricated by SE containing additives were improved to some extent. Especially, under the lubrication of SE containing CRP or CPN [Figure 7(b) and (d)], there were just some furrows and small amount of pits, indicating leaf-surface wax extracted from the coastal plant leaves indeed exhibited better tribological properties, which was consistent with the previous experimental results.

Figure 7 The wear scar surface morphologies of the aluminum discs lubricated with (a) SE, (b) SE + 1.2 per cent CRP, (c) SE + 1.2 per cent IRP, (d) SE + 1.6 per cent CPN and (e) SE + 1.6 per cent IPN



3.4 Time-of-flight secondary ion mass spectrometry analysis

TOF-SIMS is very sensitive to a range of species. To further analyze the possible lubrication mechanisms, TOF-SIMS was used to characterize the chemical compositions on the worn surfaces lubricated by different lubricants. For quantitative analysis and on account of the base material of aluminum, data were standardized with Al, and so that the Al^{+} highest intensity was chosen as a normalized intensity of the positive ions graphs, whereas the AlO^{-} highest intensity as a normalized intensity of the negative ions graphs. Figure 8 and Figure 9, respectively, give the spectra of the positive and negative ions on the worn surfaces lubricated by different lubricants. It can be seen that all the chemical compositions of the worn surfaces were composed of short-chain and medium-chain ions. However, the intensities of the some positive and negative ions on the worn surface lubricated by coastal and inland leaf-surface wax were different. As for the positive ions, the intensities of short positive ions (C1-C6) lubricated by CRP and CPN are higher than that lubricated by IRP and IPN. In terms of the negative ions, all the intensities of the detected negative ions on the surfaces lubricated by CRP and CPN were higher than that lubricated by IRP and IPN. The TOF-SIMS spectra suggest that these ions form a protective film on the worn surface to improve the tribological properties during the friction test, and the reason for the better tribological properties of CRP and CPN than those of IRP and IPN was attributed to the difference in the amounts of ions on the worn surface.

3.5 Discussion

As the results of friction test show, all the leaf-surface waxes can effectively improve the friction reduction and anti-wear abilities of base oil. The lubrication mechanism can be explained by the results of GC-MS, SEM and TOF-SIMS.

All the leaf-surface waxes could effectively reduce the damage of worn surface as the SEM shows, and this can be attributed to the protective effects of lubricating film formed by leaf-surface waxes. With the addition of additives, not only the plowing of wear debris was reduced but also the corrosion was inhibited. Therein the coastal waxes had better protective abilities than the inland waxes, and this may be attributed to that plants growing under harsh conditions generally have thicker wax coats to preserve the water balance resulting in the different chemical components of coastal and inland waxes (Eglinton and Hamilton, 1967; Xu *et al.*, 2009).

Based on the results of GC-MS and TOF-SIMS, it can be inferred that the very long carbon chains of waxes were divided into shorter carbon chains in the process of friction, and these organic compound ions and other ions bound to the worn surface through physical adsorption and chemical reaction, reducing the direct contact between two friction pairs to resist wear (Kanno, 1981; Fellah *et al.*, 2018). Therein different length carbon chain ions were bound to the worn surface forming a micro-rugged lubricating film, improving friction reduction and anti-wear capacities.

The polar groups (−COOH, −OH and so on) with stronger chemical activity can absorb on the metal surface more firmly

Figure 8 The positive ions normalized intensity diagrams on worn surfaces lubricated with (a) SE + 1.2 per cent CRP, (b) SE + 1.2 per cent IRP, (c) SE + 1.6 per cent CPN and (d) SE + 1.6 per cent IPN

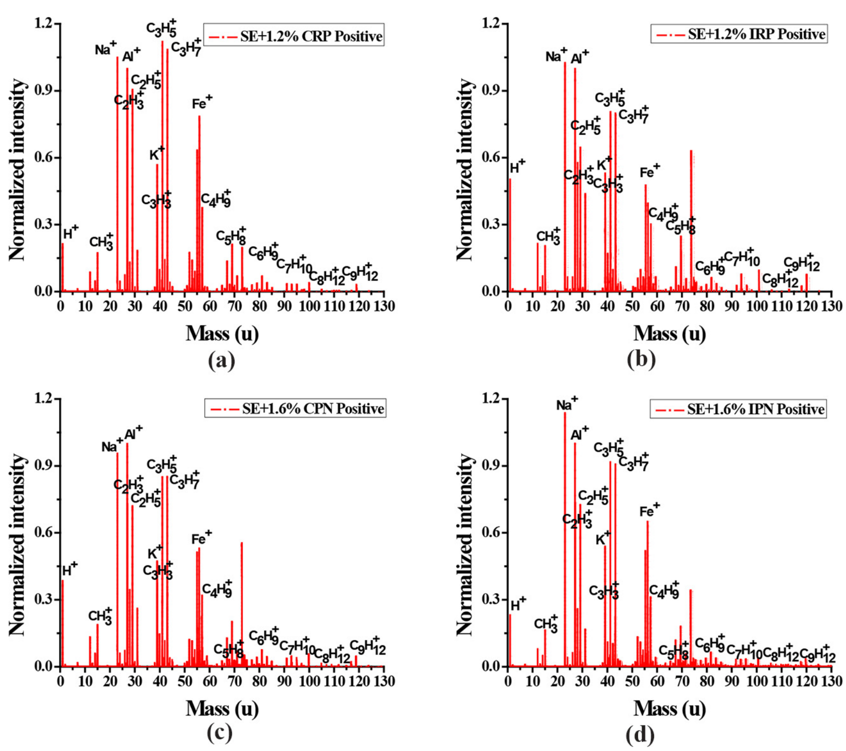
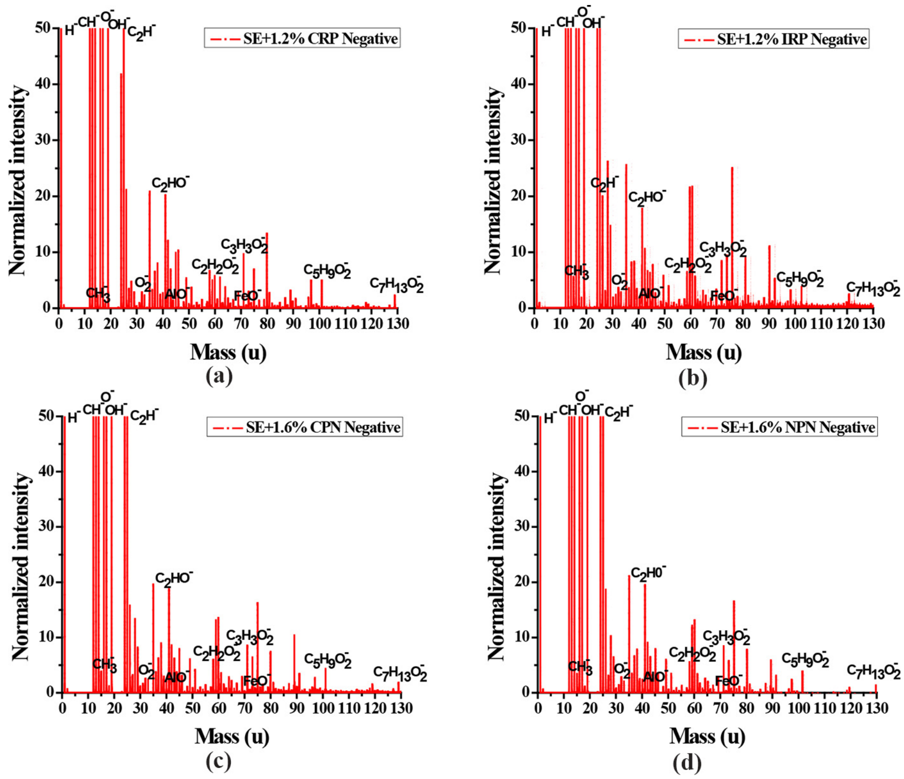
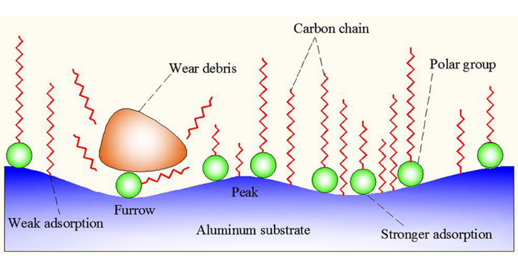


Figure 9 The negative ions normalized intensity diagrams on worn surfaces lubricated with (a) SE + 1.2 per cent CRP, (b) SE + 1.2 per cent IRP, (c) SE + 1.6 per cent CPN and (d) SE + 1.6 per cent IPN



than hydrocarbons because of their stronger polarity (Wen and Huang, 2012; Zhong *et al.*, 2018). Meanwhile, the metal surface loses electrons to produce metal positive ions in the process of friction, so that the negative ions are attached to the metal surface more easily to form a protective film because of their charge. The coastal waxes contain more alcohols and esters than inland waxes. These alcohols and esters could be divided into more polar group in the process of friction , and the wear scar composition analysis confirms this point. There were stronger binding forces between ions and worn surfaces lubricated by coastal waxes than those lubricated by inland waxes, and the stronger binding forces could form stronger lubricating film to reduce friction, as Figure 10 shows.

Figure 10 The lubricating film on the worn surface lubricated with leaf-surface waxes



4. Conclusions

Two pairs of coastal and inland leaf-surface waxes were extracted, and the waxes of different mass fractions were added to the base oil of SE to prepare lubricants. The friction reduction and anti-wear properties of wax lubricants and base oil are investigated, and the main results from test and analysis were summarized as follows:

- All the leaf-surface wax additives can effectively improve the friction reduction and anti-wear abilities of SE under the test conditions. At the load of 20 N, 1.2 per cent CRP, 1.2 per cent IRP, 1.6 per cent CPN and 1.6 per cent IPN decreased the COF of SE by 22.84 , 19.30, 28.75 and 22.44 per cent, respectively, and, respectively, decreased the WSW of SE by 46.12, 39.15, 32.56 and 19.77 per cent. This result is attributed to the different lubricating film formed by leaf-surface waxes through physical adsorption and chemical reaction.
- The leaf-surface wax additives can not only reduce the plowing of wear debris but also inhibit the corrosion of SE, presenting superior tribological properties. The reason is attributed to that the long carbon chains were divided into short carbon chain in the process of friction. Therein the coastal waxes have more alcohols and esters than inland waxes, and these alcohols and esters can be divided into polar groups with strong adsorption capacity to form stronger protective film than hydrocarbons.

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