Lubricating properties of ester oil prepared from bio-based 2, 5-furandicarboxylic acid

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Abstract: The depletion of petroleum resources and the intensification of environmental problems have necessitated the development of renewable products from bio-based chemicals instead of petroleum resources. Herein, a new kind of ester lubricating oil, isooctyl furan dicarboxylate (isooctyl-FD), was prepared from bio-based 2, 5-furandicarboxylic acid. The structure of isooctyl-FD was evaluated using nuclear magnetic resonance imaging and high-resolution mass spectroscopy. Its physicochemical and tribological properties including thermal and oxidation stabilities, flash point and pour point, viscosity and viscosity index, and friction-reducing and anti-wear properties were systematically evaluated. The results show that isooctyl-FD has comparable thermal and oxidation stability to the synthetic ester lubricating oil, isooctyl sebacate (isooctyl-S). Its friction-reducing and anti-wear properties are superior to isooctyl-S; however, its viscosity-temperature and low-temperature properties are inferior to isooctyl-S.

Keywords: 2, 5-furandicarboxylic acid; synthetic ester; bio-based platform compound; lubricating oil

1 Introduction

There are many types of available lubricants including mineral oil, synthetic oil, and vegetable oil. Among them, mineral oil obtained from petroleum resources is the most commonly used lubricating oil. However, as a non-renewable resource, the total petroleum stock constantly dwindling [1–4]. In particular, the release of a large amount of waste mineral lubricating oil into the environment would result in adverse effects on the ecological environment [5–9]. Considering the relative shortage of petroleum resources and the increase in environmental problems, increasing attention has been focused on the development of renewable biomass-based materials to replace the traditional fossil resources [10–18]. Biomass is inexpensive and

ubiquitous. As a sustainable and renewable resource, biomass has great potential to facilitate the transition from non-renewable petroleum resources to renewable bio-energy sources [19–23].

2, 5-furandicarboxylic acid is one of the members of the furan family. This chemical is abundant and readily found in plants and plant straw resources. It can also be obtained from fructose and galactose. Moreover, it contains two carboxyl groups that are considered to be alternatives to p-phthalic acid. Its aromaticity is weaker than that of a benzene ring and it is easily degraded [24–32]. In 2004, 2, 5-furandicarboxylic acid was identified as one of the 12 priority chemicals by the US Department of Energy that would probably be used to build the future “green” chemical industry [33].

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2, 5-furandicarboxylic acid can be used to synthesize esters because it has two modifiable carboxyl groups. In the present work, 2, 5-furandicarboxylic acid was used to prepare an ester lubricating oil and the product was determined to have good lubrication performance in addition to high thermal and oxidation stability. To the best of our knowledge, there are no published reports on bio-based lubricants to date. The presented procedure is consistent with the requirements of environmental protection and sustainable development. Moreover, it has great significance with respect to the development of green and environment-friendly ester oils.

2 Experiments

2.1 Materials

2, 5-furandicarboxylic acid (98%) and p-toluenesulfonic acid (98%) were purchased from Energy Chemical. Isooctanol (98%), isooctyl adipate (isooctyl-A, 98%) and isooctyl sebacate (isooctyl-S, 97%) were procured from Aladdin Amethyst Chemicals. All the other chemicals were of analytical reagent grade.

2.2 Synthesis and structural characterization of isooctyl-FD

The specific preparation reaction equation of isooctyl-FD is shown in Fig. 1. 2, 5-furandicarboxylic acid (100 g, 0.64 mol), isooctanol (500 g, 3.85 mol) and p-toluenesulfonic acid (15 g, 0.07 mol) were mixed and stirred in a round bottom flask in an oil bath at 140 °C for 6 hours. The reaction mixture was cooled to room temperature (RT) at the end of the reaction. We extracted the crude product using ethyl acetate and subsequently washed it with water three times until it was catalyst-free. The excess isooctanol was removed by vacuum distillation. Finally, pure products were obtained by column chromatography. The molecular structure of the isooctyl-FD was examined using proton nuclear magnetic resonance spectroscopy (^1H NMR, 400 MHz) and carbon nuclear magnetic

resonance spectroscopy (^{13}C NMR, 100 MHz), performed using an Agilent 400 MHz nuclear magnetic resonance spectrometer (NMR) and high-resolution mass spectra (HRMS) were recorded using a Bruker Dalton micrOTOF-Q II instrument.

2.3 Physico-chemical properties

The viscosities of the isooctyl-FD and the reference samples (isooctyl-A and isooctyl-S) were determined based on the ASTM D7042-2012 method. The kinematic viscosities at 40 °C and 100 °C were measured with an SVM 3000 Stabinger viscometer and the viscosity index was automatically calculated by the instrument. The densities of the samples were also measured with the viscometer at 20 °C. The viscosities of the samples were tested twice to ensure the accuracy of the data.

The flash points of isooctyl-FD, isooctyl-A, and isooctyl-S were determined using a Stanhope-seta flash point tester (82000-0, U.K.) as per the method of ASTM D3828-09. Flash point refers to the lowest temperature at which a mixture of steam and air that escapes from heating oil flashes instantaneously in contact with a flame. The flash point was tested twice to obtain an average value.

The pour points of isooctyl-FD, isooctyl-A, and isooctyl-S were determined following the ASTM D97-09 method with an accuracy of 3 °C using a pour point test apparatus manufactured by Lawler Manufacturing (DR4-22 L). The pour points of the samples were measured in 3 °C decrements until pouring stopped. Duplicate tests were performed and the average value was obtained.

2.4 Copper strip corrosion test

The copper strip corrosion tests for isooctyl-FD, isooctyl-A, and isooctyl-S were evaluated using the ASTM D130 method. Polished standard copper strips (size = 75 mm × 12.5 mm × 3 mm) were completely immersed in the test samples and maintained at 150 °C for 3 hours in an oven. At the end of the experiment,

Fig. 1 Reaction equation of isooctyl-FD.

the copper strips were cleaned with acetone and images were acquired. The color and degree of corrosion of the copper strips were compared with the ASTM copper strip corrosion standard.

2.5 Oxidation stability

Rotating bomb oxidation tests of isooctyl-FD, isooctyl-A, and isooctyl-S were performed using a Stanhope-seta rotating bomb oxidation tester (15200-5, U.K.) following the method of ASTM D2272-09. The experiments were performed using a 50 g sample, copper catalyst, and 5 mL of distilled water. The vessel was sealed and filled with oxygen at a pressure of 620 KPa and subsequently immersed in an oil bath at 150 °C. The time was recorded when the pressure in the bomb has dropped by 175 KPa, and it is used to measure the oxidation stabilities of the samples.

2.6 Thermal stability

The thermal stabilities of isooctyl-FD, isooctyl-A, and isooctyl-S were measured using a Netzsch synchronous thermal analyzer system (DSC/DTA-TG, STA 449 F3) under a nitrogen atmosphere with a flow rate of 50 mL/min. The temperature was set to increase from RT to 600 °C with a heating rate of 10 °C/min. As the temperature was increased, the initial and complete decomposition temperatures of the samples were obtained. These values were used to measure the thermal stabilities of the samples.

2.7 Friction and wear test

The tribological properties of isooctyl-FD, isooctyl-A, and isooctyl-S for different friction pairs were performed using an Optimol SRV-V (Germany) oscillating reciprocating friction and wear tester at 25 °C. Prior to the tests, all the steel, copper, and aluminum discs were polished in using 400 Cw, 800 Cw, and 1500 Cw SiC sandpaper and cleaned with ethanol. An AISI 52100 steel ball bearing (diameter 10 mm, hardness 700 HV–800 HV, and mean roughness 20 nm) was used for sliding on the surface of a lower disc (AISI 52100 steel: $\varnothing$ 24 mm $\times$ 7.9 mm, hardness 850 HV–870 HV; ZQSn 663 copper: $\varnothing$ 24 mm $\times$ 7.9 mm, hardness 140 HV–160 HV; 2024 aluminum alloy: $\varnothing$ 24 mm $\times$ 7.9 mm, hardness 140 HV–170 HV). The sample was

dropped on the contact surface and tests were conducted under a load of 100 N, frequency of 25 Hz, amplitude of 1 mm and duration of 30 min. After the test, the wear volume of the lower test disk was measured using a non-contact surface mapping profiler (BRUKER-NPFLEX). Each sample was tested three times and the average value was reported.

2.8 Four-ball friction and wear tester

The tribological properties of isooctyl-FD, isooctyl-A, and isooctyl-S were also tested using a four-ball friction wear tester (MRS-1J) at 25 °C. During the tests, the contact points of the four steel balls (diameter 12.7 mm) were immersed in the lubricating oil. The test speed was 1,200 rev/min, the load was 392 N, and the test time was 1 h. Upon completion of the test, the corresponding friction curve and wear spot diameter were obtained.

3 Results and discussion

3.1 Effect of emulsifier concentration

The chemical structures of isooctyl-FD, isooctyl-A, and isooctyl-S are listed in Table 1. The structure and purity of isooctyl-FD were confirmed by ^1H NMR, ^{13}C NMR and HRMS spectroscopic data and the details of the data are presented below: ^1H NMR (400 MHz, CDCl_3) δ (ppm), 7.16 (s, 2 H), 4.19–4.27 (m, 4 H), 1.66–1.72 (m, 2 H), 1.27–1.45 (m, 16 H), 0.86–0.93 (m, 12H). ^{13}C NMR (100 MHz, CDCl_3) δ (ppm), 158.34, 147.08, 118.18, 68.00, 38.92, 30.49, 29.02, 23.93, 23.04, 14.12, 11.10. HRMS (ESI) calculated for $\text{C}_{22}\text{H}_{36}\text{O}_5$ ($\text{M}+\text{Na}$) $^+$ 403.2461, found 403.2471.

Table 1 Chemical structures of isooctyl-A, isooctyl-S, and isooctyl-FD.

Samples	Chemical structure
Isooctyl-A
Isooctyl-S
Isooctyl-FD

3.2 Kinematic viscosity and viscosity index

Kinematic viscosity is the measure of the internal friction produced by the molecules when the lubricating oil is subjected to external forces due to relative movement. The moderate viscosity is helpful to form stable oil films between the sliding surfaces, which is favorable to prevent the friction surface wear during the sliding process. The viscosity index indicates the extent to which the viscosity of the lubricant varies with temperature. During the testing, it was determined that the viscosities of isooctyl-FD at 40 °C and 100 °C are higher than those of isooctyl-A and isooctyl-S. However, it can be seen from Table 2 that the viscosity index is extremely low. This is related to the molecular structure of the sample. In general, the greater the molecular weight, the higher the viscosity [34–38]. More importantly, the biggest difference between isooctyl-FD and the reference samples is that it contains an aromatic ring, which leads to a significant decrease in the viscosity index [39].

3.3 Flash point and pour point

The flash point is a safety indicator of lubricating oil during storage, transportation, and use. A higher flash point implies that the lubricant is safer. In Table 2, isooctyl-FD has a flash point of 201.5 °C, which is slightly higher than the values of isooctyl-A (190.0 °C) and isooctyl-S (194.0 °C). The associated temperature of this parameter is related to the molecular structure. The existence of furan ring can increase the flash point [40].

The pour point is a conventional indicator used to measure the low-temperature flow of lubricants. In general, the pour point of lubricating oil refers to the lowest temperature at which it can flow and when the temperature reaches a certain low level, it solidifies and loses fluidity. The lower this value, the better the low-temperature fluidity of the lubricant oil is. In Table 2, isooctyl-FD has a pour point of −13 °C, which

is obviously higher than the values for isooctyl-A (<−60 °C) and isooctyl-S (<−60 °C). This is because isooctyl-FD contains an aromatic ring and the introduction of the furan rings increases the pour point of the product [41, 42].

3.4 Oxidation stability

The oxidation stability refers to the ability of lubricating oil to resist a permanent change of its properties due to the effect of oxygen. As lubricating base oil, ester oils are often operated at high temperatures with strong oxidizing conditions in contact with metal surfaces and oxygen. Therefore, oxidation stability is also an important property of an ester lubricating oil. The oxidation stability of lubricating oil is measured in terms of oxidation life (T_r). The longer the test time (T_r), the better its oxidation life. In Table 2, isooctyl-FD has an oxidation life of 45.1 min, which is slightly higher than the value for isooctyl-A (39.0 min) and isooctyl-S (43.9 min). This indicates that isooctyl-FD has better oxidation stability than isooctyl-A and isooctyl-S. According to the studies in Refs. [43–45], the molecular containing shorter ester chains and branches on the ester chains can improve the oxidation stability of a sample. Moreover, the presence of an aromatic ring in the molecule can also improve the oxidative stability of lubricating oil.

3.5 Copper strip corrosion test

The copper corrosion test is an important method in the evaluation of the corrosive property of lubricating oil on a metal surface. If a copper strip is kept in lubricating oil for 3 hours at 150 °C, the higher the corrosivity, the deeper the appearance of the color of the copper strip. Images of corroded copper strips and the reference samples are shown in Fig. 2. The copper strip that was corroded by the isooctyl-FD is light yellow, whereas the strips corroded by isooctyl-A and

Table 2 The densities at 20 °C (d_{20}), kinematic viscosity at 40 °C (KV_{40}), at 100 °C (KV_{100}), viscosity index (VI), pour point, flash point, and rotating bomb oxidation time (T_r) of the samples.

Lubricants	d_{20} (g/cm ³)	KV (mm ² /s)		VI	Pourpoint (°C)	Flashpoint (°C)	T_r (min)
		KV_{40}	KV_{100}				
Isooctyl-A	0.9317	7.6336	2.3022	122.1	<−60	190.0	39.0
Isooctyl-S	0.9142	11.4910	3.2029	153.4	<−60	194.0	43.9
Isooctyl-FD	0.9890	31.3160	4.4807	4.50	−13	201.5	45.1

Fig. 2 Images of (a) new polished copper strip, (b) copper strip immersed in isooctyl-A, (c) copper strip immersed in isooctyl-S, (d) copper strip immersed in isooctyl-FD.

isooctyl-S are darker. A comparison with the ASTM corrosion standard color plate reveals that isooctyl-A and isooctyl-S are more corrosive to copper than isooctyl-FD.

3.6 Thermogravimetric analysis

Thermal stability is an index that represents the resistance of a lubricant to cracking and decomposition in a long-term high temperature environment. The thermal stability of an ester lubricating oil depends on its composition, purity, etc. Figure 3 represents the thermogravimetric curves for the thermal decomposition process of isooctyl-FD, isooctyl-A, and isooctyl-S. Based on Fig. 3, it is evident that isooctyl-FD begins to lose weight from 200 °C and decomposes completely at approximately 300 °C. Its onset decomposition

Fig. 3 Thermal decomposition temperatures of isooctyl-FD, isooctyl-A, and isooctyl-S.

temperature and total decomposition temperature are approximately the same as that of isooctyl-S. In contrast, the thermal stability of isooctyl-A is relatively poor. The results show that the thermal stability of isooctyl-FD is similar to that of the existed lubricating oil and superior to that of isooctyl-A.

3.7 Friction and wear test

The lubricating properties of the samples can be evaluated by measuring their friction coefficients and wear volumes. Friction and wear tests for isooctyl-FD, isooctyl-A, and isooctyl-S were performed on steel/steel, steel/copper, and steel/aluminum friction pairs at RT. Figure 4 shows the friction coefficients and wear volumes of isooctyl-FD, isooctyl-A, and isooctyl-S on steel/steel friction pairs. From Fig. 4(a), the friction coefficient of isooctyl-FD is much lower than the values of isooctyl-A and isooctyl-S. The shape of the curve is relatively smooth and the running-in time is very short, while the friction coefficients of the reference samples are relatively high with an increasing trend. This indicates that isooctyl-FD can form a stable oil film on the friction surface. It is evident from Fig. 4(b) that the wear volume of isooctyl-FD is also smaller than those of the reference samples. This indicates that isooctyl-FD has better anti-wear properties compared to isooctyl-A and isooctyl-S.

Under the same conditions, isooctyl-FD, isooctyl-A, and isooctyl-S were applied on copper/steel friction pairs. It is evident from Fig. 5(a) that the friction coefficient of isooctyl-FD is much lower than that of isooctyl-A and isooctyl-S. The shape of the friction coefficient curve is very smooth. The running-in time is short and negligible. In contrast, the friction coefficient of isooctyl-A and isooctyl-S is larger, and the curve is unstable. Moreover, as can be seen in Fig. 5(b), the wear volume of isooctyl-FD is also smaller than that of isooctyl-A and isooctyl-S.

Figure 6 shows the friction reducing and anti-wear properties of isooctyl-FD, isooctyl-A, and isooctyl-S on aluminum/steel friction pairs. It is evident from Fig. 6(a) that the friction coefficient of isooctyl-FD is lower than the values for isooctyl-A and isooctyl-S. Overall, the shape of the friction coefficient curves for the three samples on the aluminum friction pair has an increasing trend, but the increase of isooctyl-A

Fig. 4 The evolution of friction coefficients (a) and wear volumes (b) for different samples on steel/steel friction pairs at RT.

Fig. 5 The evolution of friction coefficients (a) and wear volumes (b) for different samples on copper/steel friction pairs at RT.

Fig. 6 The evolution of friction coefficients (a) and wear volumes (b) for different samples on aluminum/steel friction pairs at RT.

and isooctyl-S is more pronounced compared to that of isooctyl-FD. From Fig. 6(b), it can be seen that the wear volume of isooctyl-FD is much less than that of isooctyl-A and isooctyl-S by nearly a quarter of the wear volume of isooctyl-S. These results show that isooctyl-FD has better friction reducing and anti-wear properties compared to isooctyl-A and isooctyl-S on steel/steel, copper/steel, and aluminum/steel friction pairs.

3.8 Four-ball friction and wear test

In order to further confirm the good lubricating properties of isooctyl-FD. The tribological properties of the samples were tested using a four-ball friction and wear tester at RT. Figure 7 shows the results of the friction coefficient of the samples. It is evident that the friction coefficient of isooctyl-FD is lower than that of the two reference samples, and a decreasing

Fig. 7 The evolution of friction coefficients on the four-ball friction and wear tester at RT.

trend is observed in the shape of the curve. The wear spot diameter of isooctyl-A, isooctyl-S, and isooctyl-FD were 1.026 mm, 0.850 mm, and 0.810 mm, respectively, indicating that isooctyl-FD has better anti-wear performance than the others. These results are consistent with the results obtained using the SRV-V.

3.9 Mechanism analysis

In order to further explain the reduction of friction and the anti-wear mechanism of isooctyl-FD on the different friction pairs, the elemental composition of the worn surface was analyzed using X-ray photoelectron spectroscopy (XPS). It is evident from Fig. 9 that there is no significant difference between the Cu2p and O1s peaks measured on the worn regions lubricated by isooctyl-FD and the reference samples before and after the application of friction. Therefore, it can be concluded that no reaction films other than

that due to oxidation were formed on the sliding surfaces. This implies that there were no obvious tribological reactions other than oxidation on the surfaces. Identification of the source of oxygen is difficult because both the atmosphere and the ester oils are possibilities. However, it can be concluded that physical adsorption films were formed on the metal surfaces due to the polarity of the ester molecules and the aromatic rings contained in isooctyl-FD. The physical adsorption films play a major role, while the chemical reaction (oxidation) films play a minor supporting role in the friction reducing and anti-wear efficiency of the ester oils. The former was ultrasonically cleaned prior to XPS testing [46]. XPS testing was also performed on the steel and aluminum metal surfaces (as shown in Figs. 9 and 10), and we arrived at the same conclusions.

4 Conclusions

In this experiment, a new synthetic ester lubricating oil, isooctyl-FD, was synthesized by esterification reaction of isooctanol with the bio-based 2, 5-furandicarboxylic acid. After a series of physicochemical and tribological property tests, it was determined that isooctyl-FD has comparable thermal and oxidation stability to the synthetic ester lubricating oils isooctyl-A and isooctyl-S. Its friction reducing, and anti-wear properties are better than that obtained for the latter two, but its viscosity-temperature and low-temperature properties are inferior. The preparation of the lubricating base oil is consistent with the requirements of current environmental protection

Fig. 8 The XPS spectra of the worn copper surfaces lubricated with different samples at RT.

Fig. 9 The XPS spectra of the worn steel surfaces lubricated with different samples at RT.

Fig. 10 The XPS spectra of the worn aluminum surfaces lubricated with different samples at RT.

and green sustainable development because the main material used is one of 12 priority chemicals identified by the US Department of Energy for the establishment of a “green” chemical industry.

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