Lubricity and Tribological Properties of Dicarboxylic Acid and Oleyl Alcohol Based Esters

(Sifat Pelinciran dan Tribologi Ester Berasaskan Asid Dikarboksilik dan Alkohol Oleil)

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ABSTRACT

Synthesis of new lubricants nowadays is increasing to improve the lubricity properties and the quality of lubricant. In the current study, eight diesters with different chemical structures were tested in terms of their suitability as lubricants. The esterification reaction was carried out using Dean-Stark distillation method with some modification. Fourier transformation infra-red (FTIR), proton and carbon nuclear magnetic resonance (¹H-NMR and ¹³C-NMR), were used to verify the chemical structure of the diesters. The results showed that the dioleyl pimelate (DOIP), dioleyl adipate (DOIA), dioleyl glutarate (DOIG), and dioleyl succinate (DOISuc) showed good low temperature properties with pour point values at -10, -12, -16 and -20°C, respectively. The dioleyl dodecanedioate (DOID) indicated remarkable flash point value at 305°C and had slightly high oxidative stability temperature (OT) at 183°C. The flash point increased with the number of carbons for dicarboxylic acid used, while oxidative stability was affected by the unsaturated of oleyl alcohol. Tribological study showed that the diesters were non-Newtonian except DOID, which was Newtonian fluid. All the diesters were found to be boundary lubricants with low coefficients of friction (COF). Overall, the results indicated that all the diesters studied can be used as lubricating base oils.

Keywords: Coefficients of friction; flash point; oxidative stability; pour point; tribological properties

ABSTRAK

Sintesis pelincir pada masa ini telah meningkat untuk menambahbaik sifat pelinciran dan kualiti sesuatu minyak pelincir. Dalam kajian ini, lapan jenis ester berlainan struktur kimia telah diuji kesetabilannya untuk kegunaan pelincir. Tindak balas pengesteran telah dilakukkan dengan menggunakan penyulingan Dean-Stark dengan sedikit pengubahsuian. Struktur kimia diester terhasil dibuktikan dengan menggunakan transformasi Fourier inframerah (FTIR), nuklear magnetik resonan proton dan karbon (¹H-NMR dan ¹³C-NMR). Hasil kajian menunjukkan dioleil pimelat (DOIP), dioleil adipat (DOIA), dioleil glutarat (DOIG) dan dioleil suksinat (DOISuc) mempunyai sifat suhu rendah yang baik dengan nilai takat tuang masing-masing pada -10, -12, -16 dan -20°C. Dioleil dodekanedioat (DOID) pula menunjukkan nilai takat kilat yang tinggi pada 305°C dan mempunyai kestabilan oksidatif (OT) yang agak tinggi pada 183°C. Takat kilat ester meningkat dengan peningkatan bilangan karbon asid dikarboksilik yang digunakan, sementara kestabilan oksidatif dipengaruhi oleh ketaktepuan alkohol oleil. Kajian tribologi menunjukkan diester yang terhasil bersifat bukan-Newtonian kecuali DOID, yang bersifat bendalir Newtonian. Kesemua diester menunjukkan sifat pelincir sempadan dengan pekali geseran (COF) yang rendah. Secara keseluruhannya, hasil kajian menunjukkan kesemua diester kajian boleh digunakan sebagai minyak asas pelincir.

Kata kunci: Kestabilan oksidatif; pekali geseran; sifat tribologi; takat kilat; takat tuang

INTRODUCTION

There is an increasing use of synthetic esters as base fluids for lubricants in applications. This is due to the performance advantage offered by the properties of the over mineral oils. The functional group in esters carries a molecular dipole, thus ester lubricants have higher polarity than mineral oils. It is possible to have a more precise control over the chemical structure of ester lubricants can be controlled more precisely than the chemical structure of mineral oils. This is due to the fact that ester lubricants are generated by chemical synthesis from controlled raw materials (Boyde 2005). Moreover, their ester base fluids have been reported to be highly affinite for metal surfaces. This feature is attributed to the high polarity of dicarboxylic acid. In comparing dicarboxylic acid ester to mineral oils or Polyalphaolefins (PAOs) of comparable viscosity, the first one typically provides lower friction and lower wear under boundary lubrication conditions than the second. Dicarboxylic acids ester which mainly contains linear acid or alcohol groups is capable of forming a more coherent, tightly packed surface film than branched group. Therefore, it has superior lubricity to esters which mainly contain branched groups (Rudnick 2006). 406

Previous research reported widespread applications of some esters of dicarboxylic acids. Such applications include adipic and sebacic acid esters. They have been used as high-quality synthetic oils for over 50 years (El-Magly et al. 1990). Oils which are generated from dicarboxylic acid esters are characterised by a low pour point, good oxidative stability, high flash point and good tribological and rheology properties. Within the increasing temperature, their viscosity gets slightly reduced. The result, their viscosity indexes are high (Rudnick 2006). The physical properties can be primarily attributed to the structure of the lubricant base stock. For the occurrence of the chemical properties of the finished or formulated lubricants, they occur as a result of using the additives with the base stock. Thus, the selected properties of interest are viscosity, surface tension, thermal properties, volatility, oxidative stability, thermal stability, hydrolytic stability, gas solubility, and inflammability (Bhushan 2013).

Oleyl alcohol was used as an antifoam agent and cutting lubricant (Lee et al. 2003; Sudimack et al. 2002). Methyl oleate ester melts at -20°C has fairly good oxidative stable. Such properties imply the suitability of oleate esters as candidates for biolubricants (Fatemi & Hammond 1980). There are variety of esters which used in neat oils. These include esters such as monoesters (methyl oleate or isopropyl palmitate), diesters (propylene glycol dioleate) and polyol esters (trimethylolpropane trioleate). They are also used as base oils, which is usually the result of some additional requirements including higher lubricity, minimizing misting or a desire for a high level of biodegradability. Friction modifiers for automotive applications include some carboxylic acids, sulfurized fatty acids, glycerol monoesters and oleate esters (Rudnick 2006). Among synthetic esters is the TMP trioleate which is recognized as very useful, and its usefulness is attributed to its ISO46 viscosity and a pour point that is about -50°C. Previous research provided a description of the use of polyol esters with high oleic content (Lawate & Lal 1998).

Studies have also highlighted the importance of boundary lubrication especially during the starting and stopping of a machine when high loads and low speed were applied to the contact surfaces. According to Bushan (2002), a thin layer of lubricant molecule absorbs onto the metal surfaces and provides some protection against wear. It was added so that under boundary lubrication, the physical and chemical interactions of the lubricant with metal surfaces have dominance over the tribological performances. The performance of lubricants under boundary conditions highly relies on their chemical structures. Unsaturation has an inverse relation to the liquidity of the ester or its pour point and at the same time, it has a direct relation to its solubility and chemical reactivity. The higher the unsaturation is, the lower the pour point becomes (higher liquidity) whereas solubility in certain solvents and chemical reactivity increased. However, some unsaturation for low temperature fluidity is required by long chain molecules.

There was no previous study that has been conducted on the lubricity and tribological behaviours on dieters of oleyl alcohol with dicarboxylic acid. Therefore, the current study examined the lubricity and tribological properties of oleyl esters of various long chain dicarboxylic acids (C_4-C_{12}) .

MATERIALS AND METHODS

Eight dicarboxylic acids (99%) (dodecanedioic acid, sebacic acid, azelaic acid, suberic acid, pimelic acid, adipic acid, glutaric acid and succinic acid) and oleoyl alcohol (85%) of analytical grade were obtained from Fisher Scientific. Other chemicals and reagents were obtained from Aldrich Chemical and were used directly without further purification.

SYNTHESIS OF DIESTER

The esterification reaction was carried out according to Dean-Stark distillation method. The reaction mixture of dicarboxylic acid and oleoyl alcohol (a mole ratio 1:2) was heated with stirring in an oil bath to desired reaction temperature at 120-130°C. A catalyst amount of concentrated H₂SO₄ (2% of diacid) was slowly introduced into the reaction vessel. The reaction was carried out for about 4 h. Water formed during the reaction was continuously distilled off from the system. The crude reaction product was allowed to cool at room temperature before it was purified and then transferred into a separating funnel. 50 mL of diethyl ether was added to the funnel followed by three times of 10 mL of saturated NaHCO₂ solution to neutralise the catalytic acid used and unreacted diacid. The saturated sodium chloride solution was added to avoid any emulsion formation. The aqueous layer was decanted and the diethyl ether layer was dried over the sufficient anhydrous Na₂SO₄ and the hydrated Na_2SO_4 was filtered off. The yield (%) of the diester product was determined. The final product of diesters was identified by its IR, ¹H-NMR, and ¹³C NMR spectral analyses.

DIESTER CHARACTERISATION

Fourier transforms infrared spectroscopy (FTIR) and nuclear magnetic resonance (NMR) spectroscopy for proton ¹H and ¹³C were carried out according to Aigbodion and Bakare 2005.

LUBRICITY PROPERTIES

Flash and pour points, kinematic viscosity, viscosity index and oxidative stability of the diester were determined according to American Society for Testing and Materials (ASTM 2011) standard procedures. Triplicate measurements were made and the average values were reported with the standard deviation (±SD).

TRIBOLOGY AND RHEOLOGY ANALYSIS

Tribological properties of diester were performed according to ASTM method D4172-94 (ASTM 2011) and discussed based on their Stribeck curves at 40 and 100°C. The sliding speed was manipulated between $10^{-1}-10^3$ mm/s at 40 and 100°C (Kalin et al. 2009). Rheological properties of diesters were observed by the rheometer with cone and plate geometry (Coussot et al. 2009). The cone spindle used was CP 25-2 with a diameter of 0.051 mm. The shear rate was manipulated between 0 and 100 s⁻¹ at 25°C.

RESULTS AND DISCUSSION

ESTER SYNTHESIS

Eight different dicarboxylic acids were mixed separately with oleoyl alcohol at (1: 2) mole ratio, the mixture put in three necked conical flask contains 50 mL toluene as medium reaction and connected with Dean-stark apparatus. The esterfication reaction was done as it mentioned in the method section. Figure 1 shows the reaction procedure of dodecanedioic acid and oleyl alcohol to form DOID. This study was focused on the effect of dicarboxylic acid long chain carbon on the lubricity and tribological properties of diester. The diester yield percentage (%) is given in Table 1. The results showed high diester yield %. Those yields of diesters were affected by the electrophilic character of carbonyl carbon of dicarboxylic acids, which increases with the decrease in the long- chain carbon (Romero et al. 2011). Table 1 shows that DOISuc gave the highest yield percentage at 92%, while DOID gave the lowest yield percentage at 85% among all diesters.

ESTER CHARACTERISATION

All diester chemical structures were verified by FTIR, ¹H and ¹³C NMR spectroscopy. The FT-IR result gave significant peaks of carbonyl stretching vibrations (C=O) of ester at the range of 1735 to 1745 cm⁻¹ while, the peaks at 3003-3010 cm⁻¹ of C=C band were attributed to the unsaturated aliphatic carbons atoms of all diesters. The absence of hydroxyl (OH) stretching vibrations of alcohol (3230-3550 cm⁻¹) and the bonded hydrogen–oxygen stretching of acids (1700–1725 cm⁻¹) in the spectra suggest that the final products of diester were free of any unreacted alcohol and acid impurities. Bands representing -CH₃ groups (2850-2980 cm⁻¹), C-O stretching vibrations (1244 1171 cm⁻¹) and C-O-C (965–1101 cm⁻¹) were clearly visible in the spectra. Figure 2 shows the FTIR spectra of dioleyl dodecanedioate (DOID) as final product and dodecanedioic acid (DA) as raw material. The peak of carbonyl group (C=O) of DA appeared at 1700 cm⁻¹, while it was at 1738 cm⁻¹ for DOID.



Dioleyl dodecanedioate

FIGURE 1. Esterification reaction of dodecanedioic acid and oleyl alcohol to form DOID

Diester	DOID	DOIS	DOlAz	DOISub	DOIP	DOlA	DOIG	DOISuc
	18:12:18	18:10:18	18:9:18	18:8:18	18:7:18	18:6:18	18:5:18	18:4:18
Yield %	85	86	88	89	90	91	92	93

TABLE 1. The yield percentage (%) of diesters

Notes: DOID: Dioleyl dodecanedioate, DOIS: Dioleyl sebacate, DOIAz: Dioleyl azelate, DOISub: Dioleyl suberate, DOIP: Dioleyl pimelate, DOIA: Dioleyl adipate, DOIG: Dioleyl glutarate, DOISuc: Dioleyl succinat

Thus, the DA was completely esterified under the reaction conditions. The peak at 3008 cm⁻¹ of C=C band was attributed to the unsaturated carbons atoms (C=C) of DOID.

¹H and ¹³C NMR are the most valuable technique for chemical structure analysis. The ¹H and ¹³C NMR results of all synthesised diesters showed the main ¹H and ¹³C chemical shifts. The ¹H chemical shift of RCOO–CH₂ (ester) was clearly appeared at 4.02 to 4.04 ppm, while unsaturated aliphatic carbons atoms (HC=CH) appeared at 5.31-5.35 ppm. The ¹³C chemical shift for (C=O) of ester was appeared at 173-174 ppm, while the ¹³C chemical shift of unsaturated aliphatic carbons atoms (C=C) appeared at 129-130 ppm. Those chemical shifts were correspondent to the formation of ester carbonyl (Pavia et al. 2008). The ¹H and ¹³C NMR chemical shifts of DOID and DOIP are presented in details (Table 2).

LUBRICITY PROPERTIES OF DIESTER

The pour point, flash point, viscosity index and oxidation stability of diester are presented at Table 3. The lubricant ability in decreasing friction and wear is strongly affected by its viscosity. The viscosity of many esters depend on the temperature where decrease at high temperature (Salimon et al. 2011). Using of lubricants as industrial application depend on their kinematic viscosities at high temperature, for example the kinematic viscosities from 3.8 to 20 cSt at

100°C are suitable for engine oil while, from 35.2 cSt and high are demanded for automotive gear (Rudnick 2006). In the current study, the values of kinematic viscosities of diesters were calculated as the ratios of their dynamic viscosity to the density. They showed around 28 to 46 cSt at 40°C (Table 3), those values made them suitable to be used as hydraulic fluids and engine oil. However, they were low for some other applications such as automotive gear lubricants and grease oil (Bair 2000). Therefore, improvement the kinematic viscosities of esters are needed to mount their applications as lubricants. The viscosity results in Table 3 shows that the viscosity at 40 and 100°C increased with the high chain length (number of acid carbons). For example, DOID recorded the highest viscosity values of 46 and 12.8 cSt, while DOISuc showed the lowest values of 28 and 6.2 cSt at 40 and 100°C, respectively.

The viscosity of diesters increased with the increase in molecular weight and chain length of dicarboxylic acid (Table 3). The viscosity indexes (VIs) values of the diester were dependent on the difference in viscosity at 40 and 100°C. The VIs of diester in this study was in the range of 155 to 178. Synthesis of esters with high VI can be done by the suitable selection of raw materials that have high molecular weight with linear and branch chemical structures (Buenemann et al. 2003). The pour point is the most important property of any oil used as a lubricant.



4000 3750 3500 3250 3000 2750 2500 2250 2000 1750 1500 1250 1000 750

FIGURE 2. Comparison FTIR spectrum of DOID and DA

13 C chemical shift δ (ppm)		Functional group	¹ H chemica	l shift δ (ppm)	Functional group	
DOIP	DOID	r unetional group	DOIP	DOID	i unetional group	
129.97	129.9	C=C (aliphatic)	5.31	5.32,5.31	HC=CH (aliphatic)	
173.9	173.3	C=O (ester)	2.26	2.27, 2.25	H,C-C=O (ester)	
29.77	29.83	CH ₂ -C=O (ester)	2.23	2.24	-H,C-C=C-CH,-	
29.12	24.6-28.70	-CH,	4.03	4.04,4.02	-O- CH ₂ (ester)	
64.43,62.99	65.9, 64.5	O- $\tilde{\mathbf{C}}$ (ester)	1.59	1.26 -1.98	-CH,	
14.14	14.4	-CH _(aliphatic)	0.86	0.84, 0.86	-CH, (aliphatic)	

TABLE 2. The ¹H NMR and ¹³C NMR functional groups of DOIP and DOIS

The pour points of diesters in this study are summarised in Table 3. The results indicated that the unsaturated aliphatic carbons atoms (C=C) in diesters play a significant role in decreasing pour points. But, those pour points increased with the high length of carbon chain of the dicarboxylic acid used. All diesters were in the liquid state at room temperature. The low-temperature fluidity properties of DOISuc at -20°C were the lowest among all diesters. This decrease was due to the low molecular weight. Despite the high molecular weight of diesters, it gave a good pour point that was attributed to the unsaturated aliphatic carbons atoms. Flash point uses to control the required temperature for transportation and storage of lubricants. Salimon et al. (2010) reported that the appropriate values of flash points for many applications such as in aviation jet engines must be higher than 300°C. Flash point is indicator for the volatile and flammable materials in the non-volatile material (Bloch 2009).

The results in Table 3 shows that the flash points of the diesters increased with the high molecular weight. The dioleyl dodecanedioate (DOID) indicated remarkable flash point value at 305°C, which was due to the high molecular weight and long chain acid used, while the lowest point was recorded at 205°C for DOISuc. The flash points of other diesters (e.g. DOID at 305°C, DOIS at 290°C, DOIAz at 275°C, DOISub at 257°C, DOIP at 243°C, DOIA at 225°C, DOIG at 210°C and DOISuc at 205°C), in addition to other properties such as low pour point and high viscosity index, made them suitable to be used as lubricants at hightemperature applications. The low of lubricant's volatility is preferred to reduce the need to replenish the lost ester, the increase in viscosity during use and the negative effect on the environment (Bloch 2009; Buenemann et al. 2003). Oxidative stability is very important property for lubricant's quality, especially for long-time use (turbines, transformers, hydraulic and heat transfer units). The rate of oxidation depend on the chemical structure of esters (Murrenhoff 2003). In this study, the diesters were screened to measure their oxidative stability using PDSC through determination of oxidation temperature (OT), which is defined as the temperature at which a rapid increase in the rate of oxidation is observed. High oxidative stability value means more stability to be oxidised (Adhvaryu et al. 2000).

The results from Figure 3 was analysed for the OT. Effects of evaporation indicated by tailing, split peaks and change in baseline were due to loss of sample mass. However, these effects were apparent at temperatures higher than the initial oxidation peak. Moreover, these effects did not interfere with the measurement of the OT values by extrapolation of peak onset temperatures. The OT was calculated from a plot of heat flow (W/g) versus temperature generated by the sample upon degradation. Murrenhoff (2003) stated that the chemical structure of diester has significant effect on the rates of oxidation. Kubouchi et al. (2002) reported that the long-chain carbon of the acids and alcohols used in the esterfication increases the oxidative stability. Table 3 and Figure 3 show the values of oxidative stability as (OT) of all diester in this study. The DOID (OT 183°C) indicated the highest oxidative stability among all diesters due to the increase in the molecular weight. In general, the oxidative stability of all diesters was slightly low because of the presence of unsaturation (C=C). Moser (2009) stated that the effect of unsaturated group on the oxidative stability increases with the high number of double bond. For example, the mono-unsaturated as methyl

Diester	KV at 40°C	KV at 100°C	VI	OT (°C)	F.P (°C)	P.P (°C)
DOID 18:12:18	45.99	12.8	168	183±2	305±5	20±2
DOIS 18:10:18	44.66	11.8	167	175±2	290±5	15±2
DOlAz 18:9:18	38.55	9.9	165	173±2	275±5	10±2
DOISub 18:8:18	33.23	9.6	178	172±2	257±3	7±2
DOIP 18:7:18	32.52	9.2	175	170±2	243±6	-10±1
DO1A 18:6:18	31.04	8.8	176	169±1	225±5	-12±3
DOIG 18:5:18	30.49	7.8	171	166±1	210±5	-16±1
DOISuc 18:4:18	28.28	6.2	155	151±3	205±5	-20±2
PAO	33	6.0	140	167	190	-57

TABLE 3. The lubricity properties of synthesised diesters

Notes: VI: viscosity index, KV: Kinematic Viscosity, OT: oxidative temperature. F.P: flash point, P.P: poure point, values are mean ± SD of triplicate determinations. PAO: polyalphaolefin as real lubricant (Rudnick 2006).



Oxidative temperature (OT)

FIGURE 3. PDSC exotherm curve of oxidation temperature (OT) for the diesters

oleate (OT 174.9°C) is more stable than di-unsaturated as methyl linoleate (OT 142.6°C), while the general rule in the saturated esters, the ease of oxidation of C–H bonds follows the following order: -CH3 (most stable) > -CH2-> CH– (most reactive) (Rudnick 2006).

TRIBOLOGY AND RHEOLOGY OF DIESTERS

Boundary lubrication is defined as a condition where the solid surfaces are so close together that surface interaction between single or multimolecular films of lubricants and the solid asperities dominates the contact (Bhushan 2013). The oleate diesters in this study have polar structures. This polar structure is able to form an effective boundary layer. Durak et al. (2004) state that vegetable oils exhibit good lubricity because they contain organic straight-chain compounds with polar end groups. These polar end groups can be adsorbed on a metallic surface, which decreases or suppresses the surface energy and causes a reduction of the friction coefficient. Salimon et al. (2012) state that the long fatty acid chain and the presence of polar groups in the structure of plant oil makes it amphiphilic in nature, therefore allows it to be used as both boundary and hydrodynamic biolubricants. The results of the COF for the diesters with varying chemical structure are presented in Table 4. For the tests at different temperature (40 and 100°C), the COF of the diesters was higher at 100°C than 40°C due to the decrease in the viscosity of the diesters. The high value of COF of DOISuc was attributed to the low molecular weight and the viscosity. The COF increased with low molecular weight and viscosity of diesters and vice versa. Generally, all of the diesters recorded low COF below than 0.5 at 40 and 100°C. These results indicated that all products had a good quality of their tribological properties even when the temperature was increased. The results showed that all the diesters were boundary lubricants with low COF at 40 and 100°C.

Figure 4 shows the rheology analysis results of diester's (DOISuc and DOID) shear stress vs. shear rate plot. From the plot, fluids can be decided as Newtonian or non-Newtonian fluids. Classification of fluids as Newtonian and non-Newtonian depends on their viscosity by increasing shear rate, where the constant viscosity is Newtonian, while nu-constant viscosity is non Newtonian (Batchelor 2000). Based on Figure 4, DOISuc was identified as a non-Newtonian fluid while DOID was a Newtonian fluid. The rest of the diesters namely DOIS, DOIAz, DOISub, DOIP, and DOIG were identified as non-Newtonian fluids.

TABLE 4. Tribological properties of the diesters

Diester		DOID	DOIS	DOlAz	DOISub	DOIP	DOIA	DOIG	DOlSuc
COF	40°C	0.15	0.16	0.17	0.18	0.19	0.20	0.22	0.25
	100°C	0.19	0.20	0.21	0.23	0.25	0.27	0.30	0.32



FIGURE 4. Shear stress (τ) vs. shear rate (γ) plots of DOID and DOISuc respectively at 25°C

CONCLUSION

This study showed that the esterification of oleyl alcohol and dicarboxylic acid produced high yield of diester with good tribological and lubrication properties. Based on the results, it is possible to use the synthesised diesters as lubricants for both cold and warm climates without any additives. The results also showed that, despite the unsaturation (C=C) of diester, they had a high oxidative stability. This study suggests that the improvement of the lubricity properties (e.g. the pour point at low temperatures) and oxidative stability and flash point at high temperatures) of the synthesised diesters can be investigated by the epoxidation followed by oxirane ring opening reactions.

ACKNOWLEDGEMENTS

We would like to thank Universiti Kebangsaan Malaysia (UKM) for the project funding under University Research Grants No. DPP-2014-058, GGPM-2014-033 and the School of Chemical Science and Food Technology, Faculty of Science and Technology, Universiti Kebangsaan Malaysia for their support and encouragement.

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Received: 23 April 2014 Accepted: 26 September 2014