

## Lubricity Characterizations of Sebacic Acid Based Ester

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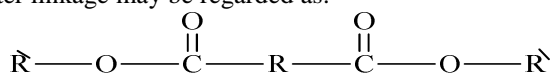
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**Abstract**— The current study aimed to evaluate the effect of long-chain and branch alcohol on lubricity of sebacic acid based ester. Since currently synthesis of a new lubricant to improve the lubricity properties and quality of lubricant is increasing, in this study, sebacic acid esters were synthesized by the conventional esterification method with some modification using sulfuric acid (SA) as catalyst. Twelve samples of sebacic acid esters with different chemical structures were synthesized including di-2-octyl sebacate, didecyl sebacate, di-2 ethylhexanol sebacate, di-2-ethylbutyl sebacate and dioleoyl sebacate. These diesters were tested in terms of their suitability as lubricant. The results showed that the pour point, flash point and oxidation stability were affected by linear and branch of the alcohol used. Di-2-ethylbutyl sebacate (D2EBS) and di-2-ethylhexanol sebacate (D2EHS) showed very low pour point at -44°C and -60°C respectively, while the same carbon number of dihexyl sebacate (DHS) and dioctyl sebacate (DOS) recorded high pour point at 8°C and 15 °C respectively. These differences were due to the presence of the branching. DOS recorded the highest oxidative stability at 290°C. However, there was a slight negative effect of the branching on the flash point.

**Keywords**— lubricity; Sebacic acid ester; pour point; flash point; viscosity index

### I. INTRODUCTION

By far the most important extension of the use of synthetic lubricants has been to employ compounds containing an ester linkage [the product of reaction an alcohol (R-CH<sub>2</sub>OH) with an organic acid (R'-COOH)]. The diester linkage may be regarded as:



In which the organic groups R\ come from the alcohol used and R' from the acid used.

Diesters are currently used in many applications including automotive and marine engine oils, compressor oils, hydraulic fluids, gear oils, and grease formulations [1]. The low toxicity and excellent biodegradability of ester molecules now afford added benefits to those of performance [2]. Reacting a linear diacid with a linear, but more usually a branched, monofunctional alcohol makes a diester. Diesters have very good viscosity indices (VIs) and pour points. This refers to their “dumb-bell” configuration. The linear diacid portion of the diester contributes to the good VI and high pour points, while the branched alcohol gives a good pour points and low VI. However, the branched alcohols are at the end of a linear acid, and this gives an excellent trade-off between VI and pour point due to the free rotation around the ester linkage is good [3].

Sebacic acid is a chemical compound which is primarily derived from castor oil with two primary uses as a reactant with other chemicals to produce distinct chemical compounds or polymers or directly in formulated products such as a corrosion inhibitor in cutting and metal working fluids [4]. Sebacic acid can be used directly in formulated products such as antifreeze coolants [5]. As a fatty chemical, it coats exposed metals with a thin film that helps protect against oxygen and electrolytic corrosion. The sebacate esters are frequently used as liquid proxies for the corresponding acid in making greases. Sebacate esters compete with the chemical derivatives of adipic acid, azelaic acid, and dodecanedioic acid as plasticizers. Derivatives of sebacic acid can be used in the production of corrosion inhibitors [6].

Long-chain compounds with branched or bulky moieties find uses in various commercial products such as biodiesel and an alternative diesel fuel derived from vegetable oils or animal fats [7]. The branched esters such as neat isopropyl or isobutyl esters have been applied to improve the low-temperature properties as documented by cloud and pour points [8]. They serve as one of the possible solutions for improving the low-temperature properties of biodiesel, and the others are diluted with conventional diesel fuel, the use of various polymeric additives [7], and winterization [9]. Other compounds with branched or bulky moieties, such as neopentylglycol diesters, are used commercially in lubricants [10], plasticizers and cosmetics [11].

Physico chemistry of esters depends upon the balance between hydrocarbon moiety and ester groups. Hydrocarbon chain length reflects in viscosity, viscosity index and compatibility while the pour point, oxidation and biodegradability are reflected by the extent of branching [12]. Presence of ester group means polarity, which reflects in evaporation, lubricity and solvency [13]. So in comparison to mineral oils, many esters have a series of technical advantages, such as natural high VI (over 160), low temperature properties (good cold flowing), pour point < -30 °C, low evaporation losses and good thermal stability i.e. high temperature properties, hydrolytic stability, good anti-friction and anti-wear characteristics, high flash point (>260 °C) compatibility and biodegradability [14]. It has been shown that fundamentally possible types (mono, di-, poly- and complex esters) have acceptance for biodegradable lubricants primarily for cost reasons [13].

The aim of the present work was to synthesize novel sebacic acid esters based lubricant and examine the effect of long-chain (C2-C8) and branched alcohol such as 2-butanol, 2-octanol, 2-hexanol, 2-decanol, 2-methyl-1-butanol, 2ethyl-1-butanol and 2-ethyl-1-hexanol on the lubricity properties of sebacic acid based ester. Thus, the study included evaluation of the advantages of the obtained diester of sebacic acid with respect to their application as components of lubricant.

## II. MATERIALS AND METHOD

### A. Chemicals

Sebacic acid (99%) was obtained from Fisher Scientific. All other chemicals and reagents such as oleyl alcohol, 2-ethyl-1-hexanol, 2ethyl-1-butanol, Decanol, 2-decanol, Octanol, 2-octanol, sec-butanol, cis-3-hexen-1-ol were obtained from Aldrich Chemical. They were used directly without further purification and the organic extracts were dried using anhydrous sodium sulfate (Aldrich Chemical).

### B. Ester Synthesis

The well known acid catalytic esterification method with a Dean Stark distillation setup was used in this study. A mixture of sebacic acid and alcohol (mole ratio 1:2) was initially placed into a three-necked round-bottom reaction flask fitted with this setup. 40-50 ml of toluene was added as a reaction medium. The reaction mixture was heated with stirring in an oil bath to desired reaction temperature at 120-130°C. A catalyst amount of concentrated H<sub>2</sub>SO<sub>4</sub> (2% of diacid) was slowly introduced into the reaction vessel. The reaction was carried out for about 4 hours. Water formed during the reaction was continuously distilled off from the system. The progress of the reaction was monitored by measuring the quantity of water which was collected in a dropping funnel. Reaction stopped when no water was further distilled off from the reaction vessel.

### C. Purification Of Sebacic Acid Ester

After the reaction stopped, the crude reaction product was transferred into a round-bottom flask. Toluene was firstly removed and the crude product was allowed to cool at room temperature before it was purified and was then transferred

into a separating funnel. The 50ml of diethyl ether was added into the funnel followed by three times of 10 ml of saturated sodium bicarbonate solution to neutralize the catalytic inorganic acid used in the reaction and remove unreacted starting sebacic acid. Saturated sodium chloride solution was then added to avoid any emulsion formation. The aqueous layer was decanted and the diethyl ether layer was dried over sufficient anhydrous sodium sulphate, and the hydrated Na<sub>2</sub>SO<sub>4</sub> was filtered off. The dried product was then passed through a column packed with silica gel 60 to remove any decomposed materials formed during the reaction and reduced the black color was caused by Sulphuric acid. The column product was rotary-evaporated to remove the solvent. Trace solvent was further removed under nitrogen gas and Excess alcohol in the mixture was removed using the distillation method. The yield% of the diester product was determined. The final diester purities were checked its IR, <sup>1</sup>H-NMR and <sup>13</sup>C NMR spectral analyses.

### D. Characterization

Fourier transforms infrared spectroscopy (FTIR) was carried out according to Aigbodion & Bakare [15]. The FTIR of the products was recorded on Prkin Elmer Spectrum GX spectrophotometer in the range 400-4000 cm<sup>-1</sup>. It was used to measure the functional groups of diester. A very thin film of product was covered on NaCl cells (25 mm.d × 4 mm thickness) and was used for the analysis. Nuclear magnetic resonance spectroscopy (NMR) for proton <sup>1</sup>H and <sup>13</sup>C has been carried out according to Aigbodion & Bakare [15]. <sup>1</sup>H and <sup>13</sup>C NMR experiment was performed with model Joel FCP 400 MHz with the solvent CDCl<sub>3</sub>. <sup>1</sup>H and <sup>13</sup>C NMR of diesters were recorded on Bruker 300 NMR spectrophotometer 20 mg of sample was dissolved in 1 mL of CDCl<sub>3</sub> and introduced into the NMR tube.

### E. Lubricity Analysis

The well known acid catalytic esterification method with a Dean Stark distillation setup was used in this study. A mixture of sebacic acid and alcohol (mole ratio 1:2) was initially placed into a three-necked round-bottom reaction flask fitted with this setup.

The Pour point (pp), flash point, viscosity, viscosity index and oxidative stability values (oxidation temperature onset OT °C) were measured according to the ASTM method [16]. Triplicate measurements were made and the average values were reported with the standard deviation (±SD).

## III. RESULT AND DISCUSSION

### A. Chemical Reaction And Yield %

The esterification reaction of sebacic acid and alcohol (1:2) mole ratio was done out according to routine method, through 4 h at the range of 100-130°C with some modification using nitrogen. H<sub>2</sub>SO<sub>4</sub> was used as a catalyst. Figure 1 shows the reaction procedure of sebacic acid and 2-ethyl-1-hexanol. The formed water through reaction was removed by dean stark method using toluene as a zoetrope. After 4 h the reaction stopped and left for 2- 3 h to stable and cool, it was dissolved in 50ml of diethyl ether and put in separating funnel, and then the mixture was washing using

saturated NaHCO<sub>3</sub> (10ml X 3) followed by 10 ml of saturated NaCl until the pH of organic layer was 7.

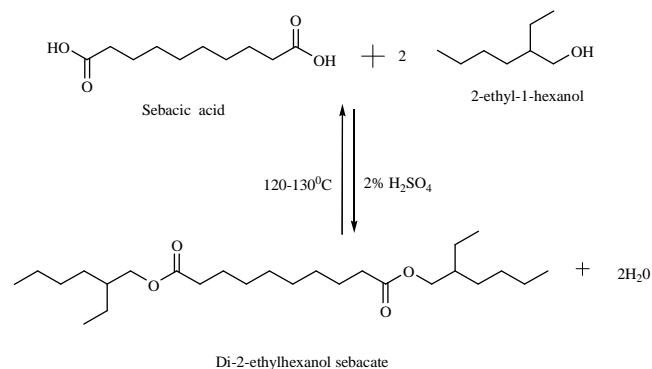


Fig. 1 Esterification reaction of sebacic acid and 2-ethyl-1-hexanol to form D2EHS.

The diester yield % values are shown in Table 1. The percentages of the yields of diester showed different values and this depends on the structure and the –OH position of the alcohols used. The esterification rate increased when the carbon number of alcohol grew. On the other hand, it was decreased when the secondary alcohols were used. So this means that large chain alcohols lead to get a more productive esterification, with a less reactive consumption. The effect of the position of the –OH group on esterification rate was as expected; the esterification extent obtained is small in the secondary alcohols because of the steric resistance [16].

### B. Characterization of the Ester Groups

The ester group of all compounds was characterized using FTIR and NMR. FTIR results showed that the absorption bands were due to the aliphatic carboxylic group (1710 and 1715 cm<sup>-1</sup>) were not observed. This result suggests that sebacic acid undergoes complete esterified under the reaction conditions. The production of diesters was characterized with the absorption bands of C=O bend of ester at 1735-1739 cm<sup>-1</sup>, C-O stretching vibrations of 1244 - 1171 cm<sup>-1</sup> and C–O–C bands in esters of 965–1101 cm<sup>-1</sup> for all synthesized diester.

The H NMR is the most valuable form of the technique for compound analysis. The frequency at which any given hydrogen atom in an organic compound resonates is strongly dependent on its precise molecular environment. Representative HNMR and <sup>13</sup>C NMR was for D2EBS and D2EHS. The <sup>1</sup>H NMR result also shows the main signals of assignments in some tested diester including D2EBS and D2EHS. The distinguishable peaks appeared at 3.69 and 3.68 ppm for C=O ester of D2EBS while D2EHS at 3.73 and 3.72 ppm. <sup>13</sup>C NMR spectroscopy is one of the less naturally-abundant isotopes of carbon which exhibits the phenomenon. However, until the emergence of comparatively recent development in instrumentation and data processing, <sup>13</sup>C NMR spectroscopy has been becoming much more accessible and since all carbon atoms in the organic compounds give distinctive signals regardless of being linked to protons, a great deal of structural information can be obtained from the spectra. The <sup>13</sup>C NMR results show the main signals of assignment of D2EBS and D2EHS, the

disappearance of carbon atom of the carbonyl group of D2EBS and D2EHS diester clearly appeared at 172.71 and 173.12 ppm respectively. The signals at 10.73 to 39.96 ppm refer to the saturated aliphatic carbons atoms for both D2EBS and D2EHS.

### C. Lubricity Characterizations

Table 1 presents the lubricity properties such as viscosity, viscosity index, pour point, flash point, and oxidative stability of sebacic acid esters.

TABLE I  
THE LUBRICITY CHARACTERIZATION OF SEBACIC ACID BASED ESTER.

Diester	Viscosity by cSt		VI	Yield %	Pour point	Flash point	Onset temperature (PDSC) °C
	40°C	100°C					
DES	5.67±0.59	2.4±0.1	210±28	85±2	-10°C±1	150±5	196±1
DBS	6.11±0.19	3.3±0.1	301±8	87±2	-5°C±3	155±5	204±2
D2BS	8.41±0.39	3.3±0.1	246±12	86±3	-22°C±3	150±3	189±1
DHS	10.49±0.36	3.4±0.1	205±20	89±1	8°C±2	185±5	213±1
D2HS	11.58±0.35	3.2±0.2	164±19	86±1	-17°C±1	173±4	207±3
DOS	10.55±0.40	4.5±0.1	262±6	92±2	15°C±2	205±5	290±2
D2OS	11.55±0.42	4.4±0.1	250±4	88±1	-12°C±3	185±5	255±2
D2EBS	11.34±0.18	3.3±0.1	172±16	87±2	-44°C±2	175±5	208±2
D2EHS	11.71±0.46	4.9±0.1	243±3	90±1	-60°C±2	190±5	179±1
DOIS	45.54±0.45	11.9±0.1	166±1	91±3	14°C±2	290±5	175±2
D2EHS*	11.8	3.1	126	-	-60	-	-

DES: Diethyl sebacate, DBS: Dibutyl sebacate, D2BS: Di-2-butyl sebacate, DHS: Dihexyl sebacate, D2HS: Di-2-hexyl sebacate DOS: Dioctyl sebacate, D2OS: Di-2-octyl sebacate, D2EBS: Di-2-ethylbutyl sebacate D2EHS: Di-2-ethylhexanol sebacate, DOLD: Dioleyl sebacate. D2EHS\* previous study [19]

The efficiency of the lubricant in reducing friction and wear is greatly influenced by its viscosity. This viscosity of diester decreases as temperature increases [18]. For instance, kinematic viscosity ranges, at 100°C, from 3.8 to 20 cSt, from 4.1 to 40 cSt, or from 35.2 to 3520 cSt are demanded for engine oils, automotive gear lubricants or industrial gear lubricants, respectively [19]. Dynamic viscosities were measured in a rotational controlled strain rheometer. At least, three replicates of each test were performed on fresh samples. Kinematic viscosity values were obtained as the ratio of the dynamic viscosity to the density. The viscosity index (VI) was obtained according to ASTM D 2270 (ASTM 2005). The kinematic viscosities at 100 and 40°C were measured. The kinematic viscosity at 100°C was used to find the L and H values directly from an ASTM reference table [20]. Then, the VI of the sebacic acid esters was obtained on the basis of the L and H values, and the kinematic viscosity at 40°C (U) using the following equations [21]:

$$VI = \frac{L-U}{L-H} \times 100$$

The viscosity index (VI) indicates the effect of the temperature on the viscosity. A low viscosity index represents a relatively large change of viscosity with temperature. On the other hand, a high viscosity index represents a relatively little change in viscosity over a wide temperature range. The importance of the viscosity index can be easily shown by considering automotive lubricants. Ester having a high viscosity index resists excessive thickening when the engine is cold and consequently promotes rapid starting and prompt circulation. It resists excessive thinning when the motor is hot and thus provides

full lubrication and prevents excessive oil consumption. Sebacic acid esters have viscosity values around 5 to 45 cSt at 40 °C that make them suitable to be used as hydraulic fluids. However, these viscosities are low for some other applications such as gear lubricants. Therefore, viscosity modifiers are clearly needed to expand the applications of diester as lubricants [22] and [23]. As summarized in Table 1, the viscosity of sebacic acid ester increased by increasing the molecular weight, chain length and the degree of branching of the alcohol. Linear sebacic acid ester tends to have indices in the region of 210 to 301 while the branched ones tend to have VIs in the region 172 to 243. The viscosity index highlights how a lubricant's viscosity changes with variations in temperature. Many lubricant applications require performing across a wide range of conditions (e.g. in an engine). Automotive lubricants must reduce friction between engine components when it is started from cold (relative to engine operating temperatures) as well as when it is running up to 200 °C [24]. Table 1 shows that the viscosity at 40°C and 100°C increased with increasing the chain length (number of alcohol carbons). For example, DOS of (10.55 and 4.5 cSt) was higher than DHS of (10.49 and 3.4 cSt) and dibutyl sebacate (DBS) of (6.11 and 3.3 cSt) respectively. A high viscosity index indicates small viscosity changes with temperature. This was noticed in the case of DBS and DHS which were recorded the high VI among the sebacic acid esters at 354 and 308 respectively. Esters of a good VI can be obtained by the proper selection of the starting materials that have linear structures [25]. The results revealed that the viscosity at 40°C and 100°C was not significantly affected by the branching such as D2EBS of (11.34 and 3.3 cSt) and D2EHS of (11.71 and 4.9 cSt) compared to isomer diester such as DHS of (10.49 and 3.4 cSt) and DOS of (10.55 and 4.5 cSt) respectively. The main advantage of the good viscosity index is that the base oil does not need a viscosity improver to maintain its viscosity at high temperatures [26].

Pour point (PP) is the most important low temperature property of any oil used as a lubricant. It is the temperature below which the oil ceases to flow when it is cooled. As per ASTM D97 method pour points are measured in steps of 3 °C. The pour points of sebacic acid esters in this study are summarized in Table 1. The flow property of some sebacic acid esters with different long chain alcohol is extremely poor and this limits their use at low operating temperature especially as automotive engine oils. However, the branched diester such as D2EBS and D2EHS were the most effective ones in terms of decreasing of the PP at -44°C and -60°C respectively. Nagendramma & Savita [13] showed that the branching in the alcohol fragments leads to a very good low pour point. Linear long-chain of DHS and DOS showed high pour points values at >5°C. This variation of pour points indicates that the branching of alkyl chain of esters plays a significant role in decreasing pour points. Cermak et al. [27] showed that the branched esters have lower pour points compared to their linear chain analogous members. It can be assumed that the presence of a large branching point on the diester creates a steric barrier around the individual molecules and inhibits crystallization, resulting in lower pour point [28]. Rizvi [29] reported that the pour points of esters depend on the structure and weight of alcohols used.

The pour points of sebacic acid esters in this study increased with the long-chain carbons of alcohol. This increase was expected due to the saturation and the high molecular weight. All sebacic acid esters were in the liquid state at a room temperature. Recently, such solid diesters have been used as novel organic phase change materials for thermal energy storage [30]. The effect of unsaturated alcohol used was clear in the dioleil sebacate (DOIS) of 14°C. Despite the high of molecular weight and long-chain carbon number, it showed slightly low pour point, and this can be attributed to the unsaturated in DOIS. These results were supported by [29], who reported that the unsaturated diester cause decreased in pour points. The low-temperature fluidity properties of di-2-butyl sebacate (D2BS) of -22°C and D2OS of -10°C were noticed compared to the same of carbon number of DBS of -5°C and DOS of 15°C due to the presence of the branching. The flash point is useful in determining the volatility and fire resistance of a lubricant. It can be used to determine the transportation and storage temperature requirements for lubricants. It should be high to ensure the safe operation and minimum volatilization at the maximum operating temperature. For the most demanding applications, such as in aviation jet engines, an effective flash point range of greater than 300 °C may be required [18].

The Flash points data can indicate the possible presence of highly volatile and flammable material in a relatively nonvolatile or nonflammable material. The mixture can be ignited only if the concentration of fuel vapor in the air is more than about 1% or less than about 6% by volume [32]. The flash point is an important factor in determining how well oil will behave as a potential lubricant. It is often used as a descriptive characteristic of oil fuel, and it is also used to describe oils that are not normally used as fuels. Flash point refers to both flammable oils and combustible oils. There are various international standards for defining each, but most agree that oils with a flash point less than 43 °C are flammable, while those having a flash point above this temperature are combustible [31]. Table 1 presents the flash points of sebacic acid esters. As shown, it increased with long-chain alcohol. The highest point recording 290 °C for DOIS was due to the high molecular weight while the lowest point was at 150°C for DES of short-chain alcohol. In general, the good volatility of some sebacic acid esters was because of their uniform or close to uniform composition. The flash points were slightly affected by the isomers. For instance, D2EHS of 190°C, di-2-octyl sebacate (D2OS) of 185°C and DOS of 205°C having equal molecular weight nearly recorded same flash points. Values of the flash points of sebacic acid esters were above 150°C, mostly approaching the level 150 to 290°C. The flash points of the some sebacate acid ester e.g. DOIS of 290°C, DHS of 185°C, DOS of 205°C and D2OS of 185°C are suitable to be used in lubricants at high temperature applications with the rest properties such as a low pour point and a high viscosity index.

Oxidation stability is the resistance of a lubricant to molecular breakdown or rearrangement at elevated temperatures in the ordinary air environment. It is very important oil characteristic; especially where life extended is required (e.g. turbines, transformers, hydraulic and heat transfer units, etc). In this study, the synthesized sebacic acid

esters were screened to measure their oxidation stability using PDSC through determination of oxidation temperature (OT). The OT (°C) is defined as the temperature at which a rapid increase in the rate of oxidation is observed. It indicates a greater stability to oxidation [33]. Results from each scan were analyzed for the OT. The OT data presented in this work are means of replicating scans conducted on at least three fresh samples. Effects of evaporation indicated by tailing, split peaks and a change in baseline due to loss of sample mass were observed for some samples. However, these effects were apparent at temperatures higher than that of the initial oxidation peak and did not interfere with 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 that was generated by the sample upon degradation. Figure 2 present the OT curve of DOS (290°C) and D2OS ( 255°C).

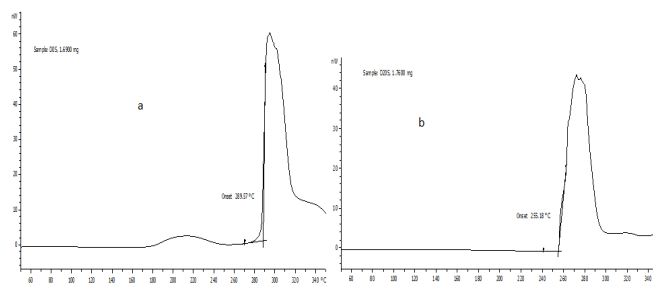


Fig. 2 Differential scanning calorimetry curve of oxidation temperature (OT) for DOS (a), D2OS (b)

P-DSC results showed that the magnitude of the increase in the OT between DBS and D2BS was significantly less than that between DOS and the isomer of D2EHS. That was due to the degree of branching. These results showed that the sebacic acid esters had a good stability to oxidation. However, those diesters differ in their rates of oxidation (e.g. DBS (OT 196 °C), DHS (OT 207 °C), and DOS (OT 290 °C) were more stable to oxidation than the isomer sebacic acid esters such as D2BS (OT 189°C), D2HS (OT 204°C) and D2OS (OT 255°C) respectively. Similar results were obtained by Murrenhoff & Remmelmann [34] as those researchers showed that diesters differ in their rates of oxidation, depending on the structure and the branching of the hydrocarbon chains. Kubouchi et al. [35] also showed that the oxidative stability increased with increasing the chain length of the esterified acid and alcohol. The branched sebacic acid ester such as D2EBS indicated a high oxidative stability of the OT at 208 °C while, D2EHS showed low OT at 179°C . Such this difference attributes to the degree of branching. In the case of DOIS (OT 175°C), the rates of oxidation were low due to the presence of unsaturation in DOIS molecule (due to C=C from oleyl alcohol).

#### IV. CONCLUSIONS

Esterification of sebacic acid and alcohol using sulphuric acid produced high yield of diester at the optimal conditions at 120°C for 4h. The branched sebacic acid esters showed good flash points and very good pour points. All values of the viscosity index recorded above 160. Based on the results, it is possible to modify the lubricity properties by altering

the length of the alcohol chain provided by the branching. In practice, the usable liquid range is limited by the pour point (PP) at low temperatures, oxidation stability and flash point at high temperatures. Thus, in the current study, sebacic acid esters indicated some excellent lubrication properties (e.g. good flash point and high viscosity index, without any additives).

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