**Influence of fatty acid methyl ester composition on tribological properties of vegetable oils and duck fat derived biodiesel**

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**Abstract**

To explore its potential as a biolubricant/additive, the study determines the frictional properties at various lubrication regimes for biodiesels derived from vegetable oils, hydrogenated vegetable oil and animal fat. It is found that the frictional characteristics for the biodiesels can be divided into *Group I* (feedstocks from winter crops) and *Group II* (feedstocks from summer crops, animal fat and hydrogenated vegetable oil). For each of the groups, with decreasing ratio of mono-unsaturated to total saturated fatty acid methyl ester content, the biodiesels’ friction force reduces while their load carrying capacity deteriorates. From the experimental results, it is deduced that soybean biodiesel shows great potential as a biolubricant/additive because it possesses low friction force with the highest possible load carrying capacity.

Keywords: *Green Tribology; Biodiesel; Frictional Mapping*; *Biolubricant*

1. **Introduction**

Global energy demand is estimated to increase from 557 quadrillion BTU (588 EJ) in the year 2014 to 703 quadrillion BTU (742 EJ) in the year 2040 [1]. During this period of time, the energy demand for the transportation sector is also predicted to grow by 27.8% [1]. Such growth pace will have a significant impact on the greenhouse gas emissions. The International Energy Agency (IEA) has identified decarbonisation, in allowing a high efficiency and low-carbon energy sector, to be the core of international efforts to combat climate change due to greenhouse gas emissions [2]. For transportation sector, decarbonisation can be achieved by means of: 1) improving fuel economy through reducing frictional losses in vehicles (higher efficiency) and 2) moving towards alternative fuels/lubricants in order to relieve the heavy reliance on fossil fuels (low-carbon).

Applying effective friction reduction strategies, Holmberg *et al.* estimate that a drop in CO2 emission, by as much as 290 million tons in the short term (5-10 years) and 960 million tons in the long term (15-25 years), could be achieved [3]. In a typical passenger car, one third of the fuel energy is used to overcome friction [3]. Aside from the pumping and hydraulic losses, the frictional losses in a passenger car also originate from the piston assembly, bearings, seals and valve-train. The strategies that can be adopted to reduce friction include the application of low friction coatings [4], surface texturing (friction reduction through lubricant micro-reservoirs) [5,6] and a more effective lubricant [7].

Evidently, all of the strategies mentioned above still involve the use of lubricant. The estimated lubricant global consumption for the automotive sector is around 22 million tons in 2015 [8], with a large majority still mineral-based lubricant. It is also found that lubricant-derived emissions could have serious impact on the exhaust-after-treatment system, which might eventually lead to emissions of toxic pollutants [9]. Therefore, in view of the impact of tribology to the environment, a new concept of *Green Tribology* has been introduced. *Green Tribology* refers to the science and technology of the tribological aspects of ecological balance and of environmental and biological impacts [10]. Twelve principles have been formulated for this concept, with one of them focusing on the use of biodegradable lubrication [11].

According to Nosonovsky and Bhushan [11], vegetable oil based or animal fat based natural lubricant should be considered for lubrication in automotive, hydraulic and metal-cutting applications because these lubricants are biodegradable. One possible alternative to petroleum-based lubricant/additive is biodiesel. Biodiesel is defined as monoalkyl esters derived from either vegetable oil or animal fat through transesterification [12]. Aside from producing lower emissions [13], biodiesel is also commonly used as an additive to improve the lubricity of petro-diesel [14,15]. Tribological improvements have been observed when small amount of biodiesel is added to petro-diesel [15,16,17,18]. Geller and Goodrum [19] and Hu *et al.* [20] explained that the biodiesels’ fatty acid methyl ester (FAME) composition plays an important role in imparting lubricity.

High Frequency Reciprocating Rig (HFRR) is typically used to understand the lubrication properties of biodiesels, especially to examine its role in fuel lubricity. The HFRR test is conducted along the boundary lubrication regime, focusing mainly on the wear scar diameter. As a result, this neglects the friction related information for a lubricated contact. The frictional properties of a lubricated contact are known to depend on the lubricant properties, applied normal load and also relative sliding velocity [21,22]. The combined effect of all these parameters can be summarised using a Stribeck curve (see figure 1), dividing the lubrication regimes into boundary lubrication (BL), mixed lubrication (ML) and elasto-hydrodynamic lubrication (EHL).

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| Figure 1. Stribeck curve (Lubrication regimes) |

Using a tribometer configured for a ball-on-disc, Maru *et al.* determined the BL and EHL phenomena contributions to the frictional properties for soybean and animal fat biodiesel in comparison with petro-diesel [23]. More recently, Maru *et a*l. compared the method in characterising lubricity for biodiesels using HFRR with a tribometer [24]. Instead of focusing on the wear scar diameter using HFRR, they generated the Stribeck curve to investigate the biodiesel friction performance. Through Stribeck curve test, they managed to observe the transition from mixed to boundary lubrication regime for their tested biodiesels. In order to better understand the boundary interaction along the BL regime of biodiesels, Maru *et al.* [25] and Chong and Ng [26] measured the nano-scale friction forces at different sliding velocities for biodiesel lubricated contacts using Lateral Force Microscopy (LFM).

As mentioned earlier, biodiesel is commonly used as a fuel lubricity enhancer. Knothe and Steidley explained that the good lubrication properties of biodiesels are attributed to their polarity imparting oxygen atoms [27]. The mechanism involved in imparting lubricity is similar to a friction modifier in a typical lubricant. A friction modifier possesses sufficiently high adsorptivity to metal surfaces due to its polarity, forming a sufficiently thick protective film [28]. Only limited work has been done to explore the capacity of biodiesel as possible alternative friction modifier [29]. The study here intends to determine the frictional properties under pure sliding motion at different lubrication regimes for vegetable oils (coconut, soybean, palm, olive and canola), hydrogenated vegetable oil (shortening) and also animal fat (duck fat) derived biodiesels. It is important to understand the effect of fatty acid methyl ester composition towards the lubrication properties for the tested biodiesels. Therefore, the study also attempts to generate a friction map relating each of the tested biodiesels to their fatty acid methyl ester compositions using a ternary plot. Hitherto, such tribological characterisation approach for biodiesel lubricity has yet been reported in literature.

1. **Experimental Approach**

*2.1 Vegetable oil and duck fat derived biodiesel*

Biodiesel can be produced from any triglycerides of vegetable oil or animal fat origins through the transesterification process. The process requires a simple global reaction involving the reactants of triglycerides and alcohol reacted at sufficient temperatures with the assistance of acid, alkaline or lipase catalysts. The reaction will produce methyl esters (biodiesel) and the co-product of crude glycerol. The possibility of using any combinations of triglycerides and alcohols mean that biodiesel can have a range of physical properties and chemical compositions.

In this study, biodiesels derived from commercially available feedstocks such as palm, coconut, soybean, olive, canola, hydrogenated vegetable oil and duck fat were selected. The fatty acid methyl ester compositions for the tested biodiesels are given in Table 1. The selection of various types of feedstocks is aimed at representing the entire saturated-unsaturated and monounsaturated-polyunsaturated FAME ranges as illustrated in the ternary plot in figure 2.

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| Table 1. Fatty acid methyl ester compositions for tested biodiesels | | | | | | | | | |
| **Type of biodiesel** | **Fatty Acid Methyl Ester Composition** | | | | | | | | |
|  | **8:0** | **10:0** | **12:0** | **14:0** | **16:0** | **18:0** | **18:1** | **18:2** | **18:3** |
| Coconut | 8.3 | 6 | 46.7 | 18.3 | 9.2 | 2.9 | 6.9 | 1.7 | 0 |
| Soybean | 0 | 0 | 0 | 0.1 | 10.3 | 4.7 | 22.5 | 54.1 | 8.3 |
| Palm | 0 | 0 | 0.9 | 1.3 | 43.9 | 4.9 | 39.0 | 9.5 | 0.3 |
| Olive | 0 | 0 | 0 | 0 | 11 | 3.6 | 75.3 | 9.5 | 0.6 |
| Canola | 0 | 0 | 0 | 0.1 | 3.9 | 3.1 | 60.2 | 21.1 | 11.1 |
| Shortening | 0 | 0 | 0 | 0 | 25.8 | 5.3 | 52.1 | 0 | 12.0 |
| Duck fat | 0 | 0 | 0 | 0.5 | 23.4 | 5.0 | 29.4 | 34.0 | 3.2 |

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| Figure 2. Ternary plot describing the fatty acid methyl ester composition for the tested biodiesels |

All of the biodiesels are produced using the same method, where pre-heated triglycerides from vegetable oil, hydrogenated vegetable oil and duck fat are reacted with premixed lye and methanol. The triglycerides are preheated at a fixed temperature of 55°C for an hour to ensure that all of the feedstocks are in liquid phase. A 1 %wt catalyst loading of potassium hydroxide (KOH) and triglyceride-to-methanol molar ratio of 6:1 is used in the reaction to ensure minimal soap production. The reactants then undergo the transesterification process in a reacting vessel at 55°C for a residence time of four hours. Crude glycerol as co-product is removed and the biodiesel is washed to remove soap and other contaminant. The process of producing biodiesel is completed when water is completely evaporated away from the biodiesels at the high temperature of 120°C. This process is to ensure that the biodiesel yield is at least 96.5% (m/m) of ester content (as required by EN 14214 biodiesel standards). The kinematic viscosities and densities for the selected biodiesels are given in Table 2.

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| Table 2. Kinematic viscosity and density for the tested biodiesels | | |
| **Type of biodiesel** | **Kinematic Viscosity at 40oC (mm2/s)** | **Density (g/cm3)** |
| Coconut | 2.726 | 0.8742 |
| Soybean | 4.019 | 0.8854 |
| Palm | 4.439 | 0.8730 |
| Olive | 4.512 | 0.8779 |
| Canola | 4.255 | 0.8784 |
| Shortening | 4.174 | 0.8397 |
| Duck fat | 4.363 | 0.8785 |

*2.2 Friction test*

Friction tests were carried out for the selected biodiesels to characterise their frictional properties under pure sliding motion with respect to the three main lubrication regimes, namely BL, ML and EHL. The test is performed using a tribometer configured for a pin-on-disc setup (see figure 3). In this study, a wear disc, fabricated from JIS SKD-11 tool steel (75 mm diameter and 4 mm thickness), is rotated against a stationary cast iron pin (10 mm diameter and 32 mm length). The cast iron pin has a spherical end cap with curvature radius of 5 mm. Both the pin and the wear disc are grinded to a surface roughness, *Ra* of 0.2 μm. Before the friction test, the pin and wear disc are cleaned using an ultrasonic bath and then left to dry in a desiccator. This is to remove the residuals of tooling fluids from the machining process.

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| Top view | Side view |
| Figure 3. Schematic diagram for a tribometer configured for a pin-on-disc setup | |

The friction tests are conducted under room temperature condition. During the test, the pin and wear disc are subjected to a constant normal load of 20 N with wear disc rotational speed ranging from 20 rpm to 2000 rpm. The wear track is set at 20 mm, corresponding to wear disc linear sliding velocity between 0.042 m/s and 4.2 m/s. For the selected test conditions, the maximum Hertzian pressure for the contact is 0.96 GPa, calculated by taking the modulus of elasticity for cast iron and JIS SKD-11 tool steel as 110 GPa and 210 GPa with Poisson’s ratio of 0.21 and 0.27, respectively. A lubricated run-in test is conducted for three minutes at 1000 rpm with 20 N normal loading in order to flatten the initial high surface asperity peaks separated by deep valleys.

Adapting the approach by Kovalchenko *et al.* [30], the friction test starts at a wear disc rotational speed of 2000 rpm and decreased in a stepwise manner after three minutes of each speed until the minimum speed of 20 rpm is achieved. This procedure allows for the contact to move from elasto-hydrodynamic to mixed and then finally to boundary lubrication regime. The amount for each biodiesel prepared for the friction test through transesterification is 1.5 litres. For the whole test duration, the tested lubricant is continuously supplied to the pin-disk contact/wear track through a pump to ensure a fully flooded lubricated conjunction. This is mainly to avoid possible lubricant starvation that could induce higher friction, owing to the lack of lubricant entrainment into the contact conjunction, especially at higher rotational speeds.

1. **Results and Discussions**

In this study, the frictional characteristics of biodiesels derived from vegetable oils (coconut, soybean, palm, olive and canola), hydrogenated vegetable oil (shortening) and also animal fat (duck fat) are investigated under pure sliding motion using a tribometer. A detailed analysis is conducted to interpret the friction force and the transition points at each of the lubrication regimes.

*3.1 Pin-on-Disc friction test*

The curves plotting the coefficient of friction against linear sliding velocity for the tested biodiesels are given in figure 4. With increasing sliding velocity, it can be seen that all the tested biodiesels show a distinct transition from BL to ML and finally to EHL regime. However, these characteristic curves seem to shift horizontally along the linear sliding velocity axis, showing a varying transition velocity (to be known as critical velocity) from EHL to ML regime for different types of biodiesels. Through the friction test, coconut and duck fat biodiesels are observed to transit from ML to EHL regime at the lowest and highest critical velocity, respectively. This shows that coconut biodiesel has the capability of sustaining fluid film lubrication for a larger range of linear sliding velocities as compared to the other types of biodiesels. Roegiers and Zhmud [28] relate such behaviour to an increased friction modifier effect, where the protective layer, adsorbing to the metal surfaces, functions to expand the borders of the fluid film lubrication regime to withstand a larger operating range of sliding velocity, eventually delaying the onset of boundary lubrication.

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| Figure 4. Coefficient of friction (CoF) measured at varying sliding velocity at 20N load for tested biodiesels |

With the tested biodiesels showing different friction modifier effect (varying critical velocities), the influence of the critical velocity values towards the frictional properties for the tested biodiesels is investigated as shown in figure 5. The friction forces are taken as the average friction values along EHL and BL regimes respectively. For the tested biodiesels, figure 5(a) illustrates the increase in friction force along the EHL region when the critical velocity increases before plateauing at approximately 2 m/s. The relative increase for the highest friction (duck fat biodiesel) with respect to the lowest friction force (coconut biodiesel) is approximately 11.5%. Such increase in friction with higher critical velocity is because of the reduced friction modifier effect (see figure 4), resulting in a less effective fluid film lubrication along the EHL regime.

In figure 5(b), the boundary friction is plotted against the critical velocity for the tested biodiesels. Along the BL regime, lubrication is typically through the formation of boundary adsorbed thin film, which provides the final barrier to inhibit direct surface-to-surface interaction. For the selected test conditions at room temperature, coconut biodiesel, with the lowest critical velocity value, exhibits a more effective boundary lubrication property than the other types of tested biodiesels. Such characteristic portrayed by coconut biodiesel is similar to those of anti-wear agent and/or extreme-pressure additives. With escalating critical velocity, the boundary friction is observed to have increased by 5.9%. The increased friction force along this region could potentially lead to the rupture of the boundary-adsorbed film. This can be observed in figure 4 for both shortening and duck fat biodiesels, where the coefficient of friction begin to increase beyond their boundary friction values below sliding velocities of 0.5 m/s and 1.4 m/s, respectively, indicating dry contact occurrence as a result of the boundary adsorbed thin film rupturing along the contact region under pure sliding motion.

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| (a) Average friction force along elasto-hydrodynamic lubrication (EHL) regime | (b) Average friction force along boundary lubrication (BL) regime |
| Figure 5. Average friction force variation with critical velocity values at (a) EHL (b) and BL regimes for tested biodiesels | |

The results thus far focus mainly on the friction forces of the tested biodiesels. To determine the undermining factor affecting the varying friction modifier effect, figure 6 illustrates the influence of the FAME saturation level towards the frictional properties for each of the tested biodiesels. By using a rational function to curve fit the average friction force along the EHL region against percentage of the total saturated FAME content, two distinct groups of biodiesels are observed. For each of the distinct groups, average friction force reductions can be seen with higher saturation level of FAME composition. This is because the saturated molecules have higher tendency to align themselves to the surface to form a more compact and effective lubrication layer [31].

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| (a) Average friction force along elasto-hydrodynamic lubrication (EHL) regime | (b) Average friction force along boundary lubrication (BL) regime |
| Figure 6. Average friction force along (a) EHL and (b) BL regimes with the amount of saturated fatty acid composition | |

From figure 6, the first group (to be known as *Group I*) consists of canola, olive and soybean derived biodiesels while the second group (to be known as *Group II*) consists of duck fat, shortening, palm, and coconut derived biodiesels. The distinction between the two groups can be made based on the melting temperature for each of the feedstocks used to derive the tested biodiesels. Referring to Table 3, the melting temperatures for the feedstocks used to derive *Group I* biodiesels are observed to occur at sub-zero temperatures while the opposite can be seen for the feedstocks used to derive *Group II* biodiesels. It is to note that *Group I* biodiesels are derived mainly from winter crops (canola, olive and soybean) while *Group II* biodiesels are derived from summer crops (palm and coconut), animal fat (duck fat) and also hydrogenated vegetable oil (shortening).

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| Table 3. Melting temperature for feedstocks used to derive the tested biodiesels | |
| **Type of feedstocks** | **Melting Temperature\* (oC)** |
| *Group I* |  |
| Canola | -10 |
| Olive | -6 |
| Soybean | -16 |
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| *Group II* |  |
| Duck fat | 40 |
| Shortening | 48 |
| Palm | 35 |
| Coconut | 25 |

*\* The melting temperature for each of the feedstock refers to the temperature where the last compound of the feedstock changes from solid to liquid.*

Following the trend lines for each of the groups (blue and red dotted lines) respectively, the average friction force along the EHL regime is seen to decrease with lower ratio of mono-unsaturated to total saturated FAME content. In figure 6(b), similar characteristic is also being observed for the average friction force along the BL regime, where the tested biodiesels are divided into the two similar distinct groups as shown in figure 6(a). A decreasing boundary friction is also measured when the biodiesel composition consists of lower ratio of mono-unsaturated to total saturated FAME content.

As pointed out earlier, the critical velocity, where the transition from ML to EHL occurs, plays an important role in determining the friction modifier effect portrayed by a lubricant, especially for the tested biodiesels in this study. Figure 7(a) shows the critical velocity for the tested biodiesels as the function of the saturated FAME content. Similar to that of figure 6, the biodiesels can also be divided into the two similar distinct groups (*Group I* and *Group* *II*). The critical velocity values are also observed to reduce with decreasing ratio of mono-unsaturated to total saturated FAME content for both *Group I* and *Group II* biodiesels. This reflects on an improved friction modifier effect when the amount of mono-unsaturation level in the biodiesel is reduced with respect to the saturation level.

Along ML regime, both the fluid film and also the asperity pairs in contact share the load carrying capacity of the contact. In figure 7(b), the velocity span (where ML regime occurs) is shown to shrink with reduced ratio of mono-unsaturated to total saturated FAME content. A smaller ML velocity span (e.g. coconut biodiesel) would see to an abrupt leap from ML to BL regime, resulting in a near-sudden undesirable increase in friction. This could be as a consequence of the fluid film having smaller load carrying capacity, which would result in a faster and easier fluid film rupture when being sheared under high contact pressure, leading to increased boundary interaction that imparts higher friction.

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| (a) Critical velocity | (b) Mixed lubrication velocity span |
| Figure 7. Friction modifier properties: (a) Critical velocity and (b) Mixed lubrication velocity span of the biodiesels | |

From the measured frictional properties, it is to note that no trend could be found relating the weighted average chain length to the lubrication properties of the tested biodiesels (each of the biodiesels in *Group I* and *Group II* has an average chain length of 18 and 17 carbon atoms, with the only exception being coconut derived biodiesel, which has an average chain length of 13 carbon atoms), which is aligned to the suggestion by Wadumesthrige *et al.* [31]. For the analysis conducted thus far, there is also no net evidence that could be observed in terms of the effect of polyunsaturated FAME compositions towards the lubrication properties of the tested biodiesels.

*3.2 Ternary plot mapping of frictional properties of biodiesels*

Both sets of figures above show a relation between the fatty acid composition and the frictional properties for each of the tested biodiesels, dividing them into *Group I* and *Group II.* To further understand the influence of the fatty acid composition on the biodiesel lubrication properties, figure 8 maps the critical velocity and ML velocity span on a ternary plot, indicating the fatty acid composition for each of the tested biodiesels. Adopting a ternary plot in this study allows for the mapping of the frictional properties with respect to the tri-axial saturated, mono-unsaturated and poly-unsaturated FAME compositions for each of the tested biodiesels.

As an initial step to map the critical velocity and ML velocity span using a ternary plot, two global axes, showing: 1) increasing ratio of unsaturated to saturated and 2) mono-unsaturated to poly-unsaturated FAME content, are determined from the fatty acid composition for the selected biodiesel types. It is interesting to note that the axis representing the increasing ratio of mono-unsaturated to poly-unsaturated FAME content splits the biodiesel types into the two groups (*Group I* and *Group II*) identical to the ones obtained in figure 6 and figure 7. This shows a reasonably good correlation between the two distinct approaches considering that the ternary plot approach takes into account only the FAME composition of the biodiesels.

In the ternary plots given in figure 8(a) and (b), the direction of the blue (*Group I*) and red (*Group II)* dotted lines represent the decreasing ratio of the mono-unsaturated to total saturated FAME content, calculated from the FAME composition of the tested biodiesels, which coincidentally also indicates a trend in decreasing critical velocity (see figure 8(a)) and ML velocity span (see figure 8(b)) for the selected biodiesels. Both these trends obtained are consistent with the observations in figure 6 and figure 7. This shows further proof that the frictional properties of the tested biodiesels are influenced by the ratio of mono-unsaturated to total saturated FAME content, once they are categorised into the two distinct groups based on the melting temperatures of the feedstock used to derive the respective biodiesels.

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| (a) Critical velocity | (b) Mixed lubrication velocity span |
| Figure 8. Ternary plot describing the change in critical velocity with the fatty acid composition for the tested biodiesels | |

1. **Conclusions**

The study investigated the frictional properties of vegetable oil and duck fat derived biodiesels at various lubrication regimes under pure sliding motion. Based on the feedstocks’ melting temperatures, it is shown that the frictional properties of the biodiesels can be divided into two distinct groups: *Group I* (feedstocks from winter crops) and *Group II* (feedstocks from summer crops, animal fat and hydrogenated vegetable oil). For each of the groups, when the ratio of mono-unsaturated to total saturated FAME content decreases, friction forces for both EHL and BL regimes reduce because of improved friction modifier effect, delaying the onset of ML and BL regimes. Among the selected biodiesels, coconut biodiesel exhibits the better friction modifier effect with the smallest friction force along both the EHL and BL regimes.

While these properties improve with lower ratio of mono-unsaturated to total saturated FAME content, the load carrying capacity of the lubricated contact along the ML regime reduces, increasing the rupturability of the fluid film. In the study, canola and duck fat biodiesels display the better load carrying capacity with coconut biodiesel being the worst. It is recommended that an ideal lubricant should have the lowest possible friction force values with the highest possible load carrying capacity [32]. Therefore, by coupling the friction force curves (see figure 5) together with the ternary plots (see figure 8), it could then be deduced that soybean biodiesel actually boast a more balanced set of lubrication properties, showing better potential as a bio-lubricant/additive when compared with the other tested biodiesels.

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