VEGETABLE OILS AS BASE OIL FOR INDUSTRIAL LUBRICANTS- EVALUATION OXIDATIVE AND LOW TEMPERATURE PROPERTIES USING TGA, DTA AND DSC

Jayadas N H1, K Prabhakaran Nair2, Ajithkumar G1

1 School of Engineering, Cochin University of Science and Technology, Kochi, India
2 K Prabhakaran Nair, National Institute of Technology, Calicut, India.

ABSTRACT
In this paper oxidative degradation of saturated and unsaturated vegetable oils is studied by thermo gravimetric analysis (TGA) and differential thermal analysis (DTA) and their low temperature properties are studied by differential scanning calorimetry (DSC). Both non-isothermal and isothermal thermo gravimetric analysis procedures are used for the study. The onset temperatures of oxidative degradation and weight gain in isothermal TGA are taken as measures of oxidative and thermal stabilities. Three vegetable oils namely coconut oil, sesame oil and sunflower oil with varying degree of unsaturation are selected for the study. The results are found to agree with reported results obtained by conventional methods. Several standard methods are currently available for determining the oxidative and low temperature properties of base oils. These methods are extremely time-consuming and data reproducibility between laboratories is poor. TGA, DTA and DSC are excellent tools to evaluate base oils in respect of their oxidative and low temperature behavior in that they produce results quickly, reproducibly and the sample requirement is very low.

INTRODUCTION
There has been growing concern for the use of mineral oils as lubricants because of the world wide interest in environmental issues [1]. This has promoted research into developing and using vegetable oils as alternative base oils for environmentally benign lubricants. Vegetable oils in general have excellent properties such as high viscosity index, high lubricity, high flash point, low evaporative loss, high biodegradability and low toxicity with regard to their use as base oils for lubricants. On the negative side they are known to possess low oxidative stability and poor low temperature characteristics [2, 3]. In developing new lubricants, it is not usually possible to screen a large number of base oils and antioxidant quantity by running expensive and time-consuming performance tests using mechanical hardware. For these reasons, it has become necessary to seek development of new oxidation stability tests, which are capable of representing field performance within a short testing time. Actually, thermo analytical methods as differential thermal analysis (DTA) and thermo gravimetric analysis (TGA) have received considerable attention [4]. These methods are advantageous in relation to the conventional ones because they present a higher precision and sensitivity as well as use smaller amount of sample and the results is obtained faster. Though TGA and DTA are extensively used for analyzing the thermal and oxidative stabilities of edible oils these methods are so far not regularly used for the analysis of base oil for lubricants. Several standard methods are currently available for determining the low temperature properties of vegetable oils, such as pour point (ASTM D97) and cloud point (ASTM D2500). These methods are extremely time-consuming and data reproducibility between laboratories is poor [5]. Differential scanning calorimetry (DSC) is a relatively simple and reproducible technique capable of providing a direct measurement of the ΔH (change in enthalpy) for a system undergoing physical and chemical changes during heating or cooling [5]. Results of studies on the cooling behavior of different vegetable oils using DSC for determination of pour point by DSC are discussed in Adharvayu A et al (5)and references therein).

EXPERIMENTAL DETAILS
The equipment used for the experiments are 1) Thermo gravimetric analyzer TGA-DTA, model Diamond TG/DTA, PerkinElmer Instruments, USA 2) DSC, model Q100, TA instruments, USA. TGA/DTA tests were conducted for coconut oil, sunflower oil and sesame oil from ambient to 700°C at a heating rate of 10°C/min under oxygen environment with flow rate 100ml/min. TGA was also done at isothermal conditions (100°C) for one hour for the above oils. DSC testsfor the above oils were conducted form 50°C to -50°C at a cooling rate of 10°C/min.

RESULTS & DISCUSSION
Results of the TGA/DTA analyses of coconut oil and sunflower oil are plotted in Fig 1 and Fig 2. Oxidative
Degradation of vegetable oils takes place in three distinct stages namely degradation of saturated fatty acids, unsaturated fatty acids and oxidation of carbonaceous residue [4]. Since coconut contains nearly 90% saturated fatty acids its TGA curve is steep associated with a high endothermic peak. For sunflower oil the TGA curve is flatter indicating weight gain by formation of oxidation products from unsaturated fatty acid constituents. Isothermal TGA graphs in Fig 3 also indicate comparatively high weight gain for sunflower oil compared to coconut oil. These results show that coconut oil has better oxidative properties because of the saturated nature of its fatty acid constituents. The pour point of coconut oil, sunflower oil and sesame oil are found to be correlated to the exothermic peak of DSC curves as shown in Fig 4. The values of pour point obtained from DSC corresponding to the exothermic peak and that from ASTM D97 tests are tabulated in Table 1. The values from either method show good agreement.

CONCLUSION

TGA/ DTA technique provide a fast, reproducible and reliable method to test the oxidative stability of base oils for lubricants. Similarly DSC can be effectively and quickly used to find out the pour point of base oils. Since these methods are fast, reliable and reproducible and the sample requirement is very low these can be used for fast development of new lubricants.

Table 1: Pour point by ASTM D97 method and DSC method

<table>
<thead>
<tr>
<th>SL No</th>
<th>Oil</th>
<th>Pour point in °C by ASTM D97 method</th>
<th>Temperature (°C) of occurrence of exothermic peak by DSC</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Coconut oil</td>
<td>23</td>
<td>24</td>
</tr>
<tr>
<td>2</td>
<td>Sunflower oil</td>
<td>-17</td>
<td>-17</td>
</tr>
<tr>
<td>3</td>
<td>Sesame oil</td>
<td>-14</td>
<td>-15</td>
</tr>
</tbody>
</table>

Figure 1: TGA and DTA curves of coconut oil

Figure 2: TGA and DTA curves of sunflower oil

Figure 3: Isothermal TGA curves of coconut oil, sesame oil and sunflower oil.

Figure 4: DSC curves of coconut oil sesame oil and sunflower oil.

REFERENCES