



Lubricant properties of biodegradable rubber tree seed (*Hevea brasiliensis* Muell. Arg) oil

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ABSTRACT

The rapid exhaustion of mineral reserves and the growing concern for the environment makes it essential to find a replacement for mineral oil lubricants and non-edible vegetable oils meets the requirements. The present work investigated the physicochemical, thermal and tribological properties of Rubber seed (*Hevea brasiliensis* Muell. Arg) oil (RSO), which is available in plenty in countries like India, Malaysia, Thailand, Peru and Ecuador. Properties like iodine value, acid value, saponification value, pour point, cloud point, viscosity and viscosity index were estimated for characterization of the oil. The fatty acid analysis was carried out using quantitative GC–MS and it was found that linoleic, oleic and linolenic were the major unsaturated fatty acids. Thermo gravimetric analysis was performed and it was found that RSO remains thermally stable upto 250 °C. Dynamic, kinematic viscosity and the viscosity index were calculated. The coefficient of friction and the wear preventive characteristics of the oil were estimated using a four ball tester. It was seen that RSO has characteristics which make it well suited to be developed into a bio-lubricant. Its properties can be improved further with the addition of suitable additives.

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1. Introduction

Lubricants formulated from vegetable oils show better performance over commercial mineral oil based lubricants in properties such as lubricity leading to reduced wear and friction, higher viscosity indices and elevated flash points which correlate to low volatility and vapor pressure making it safer for use (Ponnekanti and Savita, 2012). It is the polar nature of these oils that makes them good lubricants and their molecular makeup ensures a high natural viscosity. The development of a lubricant from a non-edible vegetable oil feedstock is advantageous as it can overcome the economic, environmental and food versus fuel issues associated with edible vegetable oils (Gui et al., 2008). Mineral oils, being a product of the distillation of crude oil are limited resources. The disposal of these lubricants effects aquatic and terrestrial eco systems drastically. Vegetable oils are available abundantly throughout the world. However vegetable oils are limited by poor oxidative stability, unpleasant smell, filter clogging tendency and low temperature fluidity (Mobarak et al., 2014). These properties have to be improvised by the addition of suitable additives such as sulphur–phosphorous compounds, organo-zinc compounds and

aromatic amine compounds for improving oxidative stability and polymethacrylates which function as viscosity index improvers and pour point depressants (Ahmed and Nassar, 2011). These non-edible crops can be grown in rural, unproductive lands or degraded forests as they are well adapted to semi-arid and arid conditions and require low moisture and fertility (Mohibbe Azam et al., 2005).

Hevea brasiliensis Muell. Arg commonly known as rubber tree belongs to the Euphorbiaceae family and is a primary source of 99% of the world's natural rubber. A healthy tree gives about 500 g of seeds containing 50–60 wt% oil on average annually (Kumar and Sharma, 2011). RSO, though rich in essential fatty acids cannot be consumed because of its inherent toxicity (Salimon et al., 2012). The latex from the rubber tree has high economic importance. Recently RSO has found a variety of applications in areas like biodiesel production (Ikwuagwu et al., 2000), foaming agent, synthesis of alkyd resin for paints and coatings (Aigbodion and Pillai, 2000). Rubber trees are grown abundantly for commercial purposes in different parts of the world India, Malaysia, Sri Lanka, Thailand Indonesia, Peru and Ecuador. Rubber seed (*H. brasiliensis* Muell. Arg) oil, has been chosen to study its potential as a biodegradable lubricant and compared with coconut oil, sunflower oil (Jayadas and Prabhakaran Nair, 2006b) and SAE20W40 a commercial mineral oil based lubricant. The tests employed in this study have been chosen from a broad portfolio of base oil tests defined by ASTM and these include tests for physical properties, chemical properties, rheological

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properties and tribological properties (Rand, 2010). Thermo gravimetric analysis was chosen to analyze the thermal properties as it has relatively better sensitivity and precision and requires only minute quantity of samples. Differential scanning calorimetry is simple and reproducible and enables a direct measurement of the enthalpy of a system; when compared with the conventional time consuming techniques which have poor reproducibility (Jayadas and Prabhakaran Nair, 2006b)

2. Materials and methods

2.1. Materials

Rubber seed oil was procured from M/s. Pavalam and Co., India. Coconut oil and sunflower oil were obtained from local dealers. Acetone, *n*-heptane, methyl ethyl ketone, pyridine, conc. sulphuric acid, starch indicator, standard sodium thiosulphate solution, ferric chloride solution, calcium chloride solution, phosphate buffer solution, standard potassium dichromate solution, manganous sulphate solution were procured from M/s. SRL Pvt. Ltd., India. All the solvents and reagents purchased were of analytical grade and were used directly without further purification.

2.2. Methods

The commercially obtained crude oil contains fiber, resin, moisture from the plant which makes it dark and opaque. The crude oil was allowed to stand undisturbed for a few days to remove the unwanted impurities by clarification. The upper layer was decanted, filtered and used for the study.

2.3. Physicochemical characterization

Standard AOCs (American Oil Chemists' Society) and ASTM (American Society for Testing and Materials) methods were followed to study the physicochemical properties which included the iodine value, acid value, saponification value. All the analysis was carried out in triplicate and an average was taken.

2.4. Analysis of thermal properties

A Cleveland open cup apparatus was used for the measurement of flash and fire point as per ASTM D92. The flash point is taken as the lowest temperature at which the oil emits enough vapors to ignite. It decreases with an increase in the fuels volatility and vice versa. The pour point was measured to an accuracy of $\pm 3^\circ\text{C}$ adhering to ASTM D97–96a. A test jar with 50 ml of the sample was placed into a cylinder submerged in a cooling medium. The temperature of the sample at the top was measured in increments until the sample stopped pouring. The pour point is the temperature at which the sample in the test jar does not flow when it is held horizontally for 5 s. The pour point was validated by conducting differential scanning calorimetry (DSC). A Mettler Toledo DSC 822e instrument was used to perform the DSC measurements, using 5–10 mg of sample sealed in hermetic aluminium pans. The selected temperature window ranged from -50°C to 220°C . ASTM D2500–99 was followed for determination of the cloud point. The experimental setup was similar to that of pour point calculation and the cloud point was taken as the coldest temperature at which the oil remained opaque at the bottom of the test jar.

2.5. Quantitative analysis of RSO

GC–MS analyses were carried out to determine the fatty acid profile of RSO. A Hewlett Packard 6890 instrument equipped with HP-5 crosslinked 5% phenyl methyl siloxane capillary column

($30\text{ m} \times 0.32\text{ mm} \times 0.25\text{ }\mu\text{m}$) and MSD 5973 equipment (Hewlett Packard) operating in EI mode at 209 eV was used and 1 μl of each sample was injected into the column. The carrier gas used was Helium at a constant flow rate of 1.4 ml/min. The components were identified based on their retention time of peak.

2.6. Thermo gravimetric analysis (TGA)

TGA was employed to give an insight into the change in weight with respect to change in temperature. The measured weight loss curve analyses the thermal stability of the sample. TGA of RSO was performed under an oxygen environment on a PerkinElmer STA 6000 in a temperature range of $0\text{--}500^\circ\text{C}$ with a 3.75 mg initial oil sample.

2.7. Rheological property

Dynamic viscosities were calculated in accordance with ASTM within a temperature range of 40 and 100°C . A Redwood viscometer was employed for the estimation of viscosity. The viscosity index (VI) was determined according to ASTM D 2270.

2.8. Biodegradability assay

Biodegradability study for RSO was performed as per the protocols mentioned in American Public Health Association (APHA) standards. The values for biochemical oxygen demand (BOD) and chemical oxygen demand (COD) were analyzed.

2.9. Tribological properties

A four ball tester machine was employed to determine the coefficient of friction (COF) and anti-wear properties of RSO. The apparatus consists of a mechanism by which a ball is rotated in contact with three fixed balls which remain immersed in the sample. Several loads are applied on the balls using a load lever. The ball test material was chrome steel alloy of diameter 12.7 mm and HRC of 61–64 with a high surface finish. The COF test was carried out at a load of 40 kg at 600 rev min^{-1} as per ASTM D 5183–05. A wear test was carried out at a load of 40 kg at 1200 rev min^{-1} with test duration of 60 min as per ASTM D 4172–94. Both the tests were carried out at 75°C lubricant temperature and duration of 60 min. The four ball tribometer recorded the frictional torque. This data was used to compute COF and the wear scar diameters (WSD) were analyzed with the help of scanning electron microscope (SEM).

3. Results and discussions

The present study explored the potential of the unsaturated non-edible *Hevea brasiliensis* Muell. Arg oil for the development of a biodegradable lubricant base stock. Table 1 shows the results of the physicochemical characterization of RSO. The color of rubber seed oil was light brown after clarification and it remained liquid at room temperature. The color of the oil may be attributed to the presence of plant pigments like tocopherols, carotenoids and derivatives (Okolie et al., 2012). The density of RSO was determined using a calibrated pycnometer and found to be 0.922 g/cm^3 which implies that RSO is less dense than water and that there is no heavy element present in the oil (Aigbodion and Bakare, 2005). The physicochemical characters seem to be similar to that of common vegetable oils and hence validate the claim that RSO can be exploited as a basestock for developing an environment friendly lubricant. Iodine number gives an idea about the degree of unsaturation of the oil and its oxidation stability. The iodine value of $135\text{ gI}_2/100\text{ g}$ indicates that RSO is a semi drying oil. The acid number gives an insight into the level of lubricant degradation while

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