The instrumentation details of the methods used to characterize the synthesised additives, as well as morphological and analytical studies of worn surfaces, are described in the present chapter. The description of steel ball bearings, properties of the base oil, testing procedures utilized to assess the tribological performance of the examined additives, and different tribological parameters have also been provided.

2.1. Instrumentation Used for Characterization of the Synthesized Additives

Transmission electron microscopy (TEM)/high-resolution transmission electron microscopy (HR-TEM) using an FEI-Tecnai-G2 electron microscope and scanning electron microscopy (SEM)/high-resolution scanning electron microscopy (HR-SEM) using an FEI-Nova Nano SEM 450 were both utilized to study the morphological characteristics of the additives. Pellets of samples that contain KBr were used to record FTIR spectra using a Thermo Scientific Nicolet iS5 FTIR spectrometer. The additives UV-visible spectra were obtained in the DMSO solution using a Shimadzu Pharma spec. UV-1700 model and LAMDA 25 Spectrophotometer Perkin Elmer, Germany. Using ZEISS SUPRA 40, Oxford Instruments, energy dispersive X-ray spectroscopy (EDX) was employed to investigate the nearly stoichiometric composition of nanomaterials. In order to determine the phase, purity, and size of the crystalline material, powder X-ray diffraction (XRD) studies were carried out on the Rigaku Miniflex 600, XRD-System using Cu-Kα1 radiation (λ=1.54Å). To determine the chemical composition of the synthesized materials and the tribofilm that had formed on the worn steel surface, X-ray photoelectron spectra (XPS) were taken using the PHI 5000 Versa Probe II, manufactured by FEI, Inc.

2.2. Antiwear Testing

2.2.1. Base Oil

The lubricating base oil, neutral liquid paraffin oil (Qualigens Fine Chemicals, Mumbai, India) having kinematic viscosity, at 40 °C and 100 °C, 30 and 5.5 cSt, respectively specific gravity of 0.82 at 25 °C, viscosity index 122, pour point -8 °C, cloud point -2 °C, fire point 200 °C and, flash point 180 °C was used without further purification.

2.2.2. Parameter of Steel Ball Bearing

For tribological tests, 12.7 mm-diameter AISI 52100 steel alloy balls with hardness grades of 59–61 HRc were used. The balls were well rinsed with n-hexane before and after each test, and they were then carefully air-dried.

2.2.3. Test Procedure

At room temperature, the prepared admixtures were sonicated for an hour. Using a Four-Ball Lubricant Tester (Ducom Instruments Pvt. Ltd., Bangalore, India), the antiwear properties of the synthesized additives were tested in accordance with ASTM D4172 standards, prescribed by the American Society for Testing and Materials. The Ducom four-ball tester's manual provides the test parameters of applied load 392 N, sliding speed 1200 rpm, time 1 h, and temperature 75 °C, that are used to evaluate the relative wear preventative capabilities of lubricating oils in sliding contact. Average wear scar diameter of the three stationary balls was evaluated and represented as MWD.

According to ASTM D4172 standards, concentration optimization test for base oil were initially conducted with and without varying concentrations of the investigated additives.

Similar conditions and the optimized concentration were used for all tribological tests. As specified by ASTM D5183, the step-loading test was carried out. After the completion of the running-in period under the test conditions (temperature, 75 °C, applied load, 392 N; sliding speed, 600 rpm; and optimized concentration (w/v)), increments of 98 N load were added every 10 minutes until the commencement of seizure of tribo-surface was observed. In each case, the tribological testing was conducted three times generally.

Figure 2.1. Four-ball tester machine

2.3. Tribological Parameters

The arithmetic mean of each ball's diameter (d_1, d_2, and d_3) for each experiment was determined using equation (2.1). The three stationary balls were not moved while the readings were taken by the image acquisition system.

2.3.1. Mean Wear Scar Diameter (MWD)

$$d = \frac{d_1 + d_2 + d_3}{3} \qquad (2.1)$$

2.3.2. Coefficient of Friction (μ)

The coefficient of friction for particular additives is found directly from the Software of four-ball tester using equation (2.2)

$$\mathrm{F} = \mu \times \mathrm{N} \qquad (2.2)$$

Where F = Frictional force, μ = Coefficient of friction, and N= Normal force

2.3.3. Analysis of Worn Surface

For magnified images of the wear scar surface of steel balls, scanning electron microscopy (SEM), and for elemental compositions of tribofilm formed on the wear scar, Energy-dispersive X-ray spectroscopy (EDX) was applied by using a scanning electron microscope (ZEISS SUPRA 40 electron microscope). To examine the roughness of the worn surfaces of steel balls with the Si_3N_4 cantilevers that have tip radii greater than 10 nm and a spring constant of approximately 0.1 Nm^{-1} (Nanosensor, CONTR type), a contact mode atomic force microscope (Model no. BT 02218, Nanosurf easyscan 2 Basic AFM, Switzerland) was used. For studying the chemical composition of the tribofilm formed on the worn surface, X-ray photoelectron spectroscopy (K-alpha X-ray photoelectron spectrometer) was used.

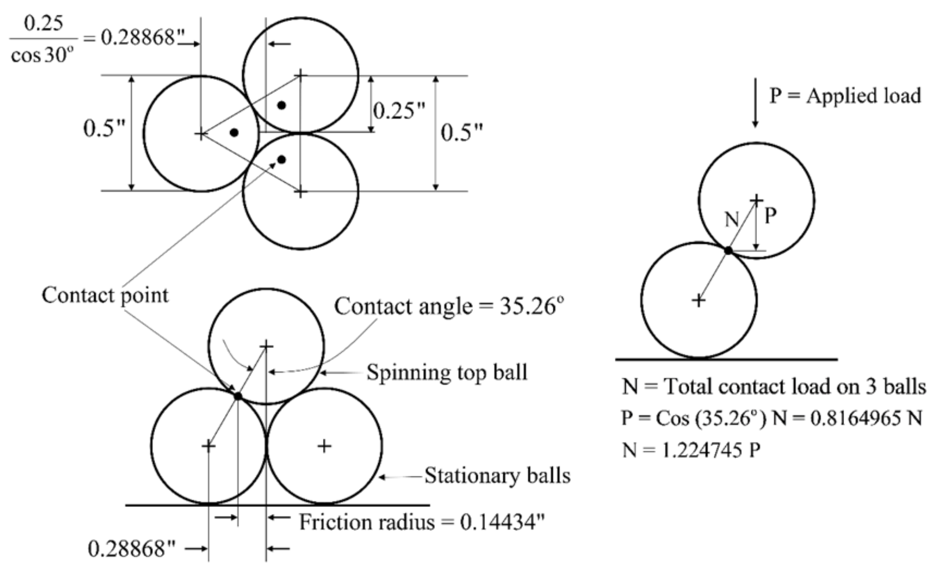
2.4. Frictional Power Loss (P)

There is more power consumption because of the loss of frictional heat. To reduce power consumption, COF has to be reduced. In the presence of additives blends in paraffin oil, COF has significantly reduced; consequently, the power consumption has decreased.

The frictional power loss is calculated by the given equation

$$P = 221 \times \mu \text{ (W)}$$

The theory for the calculation of the frictional power loss is being given.



The antiwear test was performed under the load of 392 N at 1200 rpm for one h using a four-ball tester. The total sliding distance of 1.656 km was covered during each test run.

Total Frictional Power Loss = Work done by rotating steel ball on the other three stationary steel balls by Frictional force

= - (work done by three stationary steel balls on rotating steel ball by Frictional force)

Contact load on all three stationary steel balls (N) = p/cos θ

p = Applied load and θ = contact angle

For angular motion, Work done = T.θ

T = Frictional torque (N.m) = F.r, Where, F is Frictional force = μN (μ is coefficient of friction, N is the contact load on the three balls (N), N = 1.22475p, where p is actual applied load = 392 N) and r is friction radius = 0.14434 (inch) = 3.662 x10^{-3} (m)

Since power consumed is given by work done per unit time

Thus

$$P = T.\ \theta/ t = T.\ \omega = \mu N \times r \times \omega \qquad (2.3)$$

Where, P = Frictional power loss (N.m.s^{-1}),

ω = angular velocity (rad/s) = 2πn/60, where n = 1200 RPM,

Substituting all the values in equation (2.3), The frictional power loss

$$P = 221 \times \mu \text{ (W)} \qquad (2.4)$$

$$1\text{kWh} = 3.6 \text{ MJ} \qquad (2.5)$$