This chapter provides an overview of the equipment utilized for characterizing the synthesized additives, as well as conducting morphological and analytical studies on worn surfaces. Information regarding the specifications of the steel ball bearing, attributes of the base oil, methodologies employed for testing the effectiveness of the investigated additives in terms of tribological efficiency, and diverse tribological parameters, have also been supplied.

## 2.1. Instrumentation Used for Characterization of the Synthesized Additives

The morphological characteristics of the additives were examined using scanning electron microscopy/high-resolution scanning electron microscopy (HR-SEM) with FEI-Nova Nano SEM 450 and transmission electron microscopy (TEM)/high-resolution transmission electron microscopy (HR-TEM) with FEI-Tecnai-G2 electron microscope. The Thermo Scientific Nicolet iS5 FTIR spectrometer was utilized to acquire FTIR spectra. The spectra were recorded by preparing pellets of the samples with KBr. The UV-visible spectra of the additives were obtained by recording them in the DMSO solution within the 200-1100 nm range using both a Shimadzu Pharma spec. UV-1700 model and a LAMDA 25 Spectrophotometer Perkin Elmer, Germany. Energy Dispersive X-ray Spectroscopy (EDX) analysis was performed using the ZEISS SUPRA 40, Oxford Instruments, to examine the estimated stoichiometric composition of nanomaterials. Structural characteristics of carbon-based nanomaterials were identified through Raman spectroscopic analysis. The Raman spectra were acquired using a Confocal Micro-Raman mapping system (UniRAM), which utilized a 785 nm laser as the excitation wavelength. The Rigaku Miniflex 600, XRD-System was utilized to conduct powder X-ray diffraction

(XRD) analyses, employing Cu-K$\alpha$1 radiation ($\lambda$=1.54Å), in order to determine the phase, purity, and crystalline material size. To ascertain the chemical composition of the synthesized materials and the tribofilm occurred on the worn steel surface, X-ray photoelectron spectra (XPS) were collected using the PHI 5000 Versa Probe II instrument by FEI, Inc.

## 2.2. Antiwear Testing

### 2.2.1. Base Oil

Without undergoing any additional purification, the neutral liquid paraffin oil (Qualigens Fine Chemicals, Mumbai, India), a lubricating base oil, was employed. It has a kinematic viscosity of 30 and 5.5 cSt at 40 °C and 100 °C respectively, viscosity index 122, specific gravity 0.82 at 25 °C, pour point -8 °C, cloud point -2 °C, flash point 180 °C, and fire point 200 °C.

### 2.2.2. Specification of Steel Ball Bearing

For tribological testing, balls with a diameter of 12.7 mm, composed of AISI 52100 steel alloy with a hardness range of 59-61 HRc, were employed. The balls were thoroughly rinsed with n-hexane and adequately air dried before and after each test.

### 2.2.3. Test Methodology

The prepared admixtures underwent one hour of sonication at room temperature. The synthesized additives underwent antiwear testing using a Four-Ball Lubricant Tester (Ducom Instruments Pvt. Ltd., Bangalore, India) in accordance with the standards set by the American Society for Testing and Materials (ASTM D4172). According to the Ducom four-ball tester manual, the test procedure evaluates the comparative ability of lubricating fluids to prevent wear in sliding contact, using specific conditions including an applied load of 392 N, sliding speed of 1200 rpm, duration 1 hour, and temperature 75 °C. The observed wear scar diameters of the three stationary balls were recorded, and their average value is denoted as MWD (Mean Wear Scar Diameter).

**Figure 2.1. Four-ball tester machine**

Initially, the ASTM D4172 standard was followed to conduct concentration optimization tests for the base oil, both with and without varying concentrations of the investigated additives. Under similar conditions, all tribological tests were performed at the optimized concentration. In accordance with ASTM D5183 guidelines, the step loading test was performed. After completing the running-in period under the specified test conditions (applied load: 392 N, sliding speed: 600 RPM, temperature: 75 °C, and optimized concentration (w/v)), increments of 98 N load were introduced every 10 minutes until the occurrence of tribo-surface seizure was observed. As a standard practice, the tribological testing was replicated three times for each case.

### 2.3. Tribological Parameters relevant to the present study

Tribological parameters are described in the following sections.

#### 2.3.1. Mean Wear Scar Diameter (MWD)

Equation (2.1) was used to determine the arithmetic mean of the diameter of each ball ($d_1$, $d_2$, and $d_3$) for every experiment. During the measurements, the three stationary balls remained undisturbed, and the image acquisition system was used to capture the wear scar diameter.

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

#### 2.3.2. Mean Wear Volume (MWV)

Using the Archard wear equation, the MWV data were derived from the MWD values.

- **Archard Wear Equation**

Wear volume, $$V = \frac{\Pi\, d_0^4}{64\, r}\{(\frac{d}{d_0})^4 - (\frac{d}{d_0})\} \qquad 2.2$$

Hertzian diameter, $$d_0 = 2(\frac{3\,\mathrm{Pr}}{4E})^{\frac{1}{3}} \qquad 2.3$$

Where, $$\frac{1}{r} = \frac{1}{r_1} + \frac{1}{r_2}$$

$$\frac{1}{E^*} = \frac{1 - v_1^2}{E_1} + \frac{1 - v_2^2}{E_2}$$

Where, $E^*$ = Resultant modulus of elasticity

$v$ = Poisson’s ratio

$r$ = Radius of steel ball

$E_1 = E_2 = 206$ GPa

$v_1 = v_2 = 0.3$

$d$ = mean wear scar diameter of steel ball (mm)

$d_0$ = Hertzian diameter of circular contact supporting the load before wear (mm)

### 2.3.3. Wear rate

By analyzing the average wear volume data at various time intervals, the running-in and steady-state wear rates have been determined. For each experiment, the mean wear volumes at various time intervals (0.25, 0.5, 0.75, 1.0, 1.25, and 1.5 hours) were plotted against time. A linear regression model, including the origin, was then applied to the data points to determine the overall wear rate.

$$\frac{V}{l} = K\frac{P}{H} \qquad 2.4$$

V = mean wear volume

*l* = sliding distance (2πr.N)

K = wear coefficient

H = hardness of steel ball (59-61 HRc)

P = applied load (0.408×392N)

P = Actual load in Newton on each of the three horizontal balls that is 0.408 times of applied load.

### 2.3.4. Coefficient of Friction ($\mu$)

The friction coefficient of various antiwear additives is directly determined through equation (2.5) using the software of a four-ball tester.

$$F = \mu \times N \qquad 2.5$$

Where $\mu$ = Coefficient of friction, F = Frictional force, and N= Normal force

### 2.3.5. Analysis of Worn Surface

Surface magnified images of the wear scars on 12.7 mm diameter steel balls and elemental compositions of the tribofilm formed on the wear scar were obtained by scanning electron microscopy (SEM) and Energy-dispersive X-ray spectroscopy (EDX) by using a scanning electron microscope (ZEISS SUPRA 40 electron microscope). The roughness of worn surfaces was examined using a contact mode atomic force microscope (Nanosurf easyscan 2 Basic AFM, Model no. BT 02218) equipped with a $Si_3N_4$ cantilever (Nano sensor, CONTR type) having a spring constant of approximately 0.1 $Nm^{-1}$ and a tip radius above 10 nm. The chemical composition of the tribofilm that developed on the worn surface was investigated using X-ray photoelectron spectroscopy (specifically, K-alpha X-ray photoelectron spectrometer).

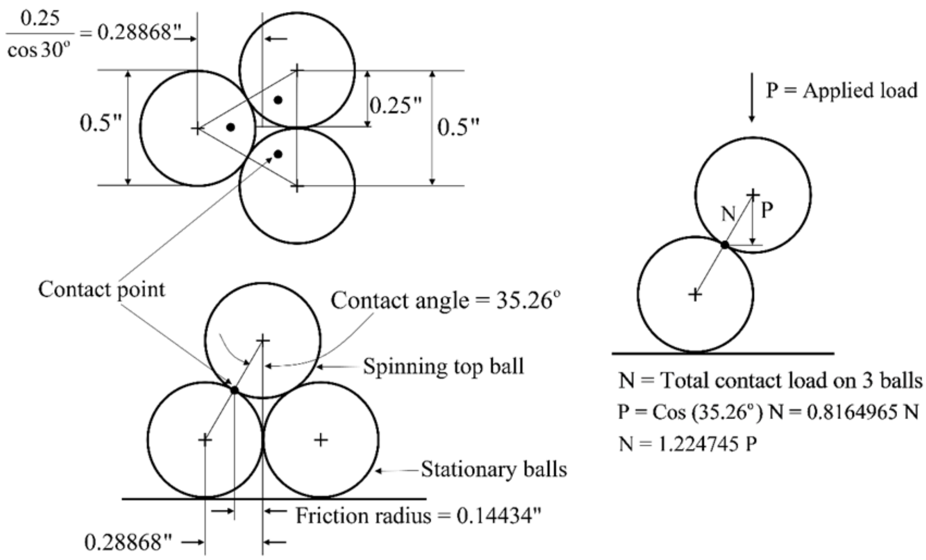
### 2.3.6. Frictional Power Loss (P)

Increased power consumption can be attributed to the dissipation of frictional heat. In order to decrease power consumption, it is necessary to lower the COF. The introduction of additive blends into paraffin oil has led to a notable reduction in the COF, resulting in decreased power consumption.

The given equation allows the determination of the frictional power loss.

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

The theory regarding the calculation for frictional power loss is being presented.




Using a four-ball tester, the antiwear test was conducted for one hour at 1200 RPM and under a load of 392 N, covering a total sliding distance of 1.656 km 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 a rotating steel ball by Frictional force)

Contact load on all three stationary steel balls (N) = $p/\cos\theta$

p = Applied load and $\theta$ = contact angle

For angular motion, Work done = $T.\theta$

T = Frictional torque (N.m) = F.r, Where, F is Frictional force = $\mu N$ ($\mu$ 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) = $3.662\times10^{-3}$ (m)

Since power consumed is given by work done per unit of time

Thus

$$P = T.\ \theta/\ t = T.\ \omega = \mu N \times r \times \omega \tag{2.6}$$

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

$\omega$ = angular velocity (rad/s) = $2\pi n/60$, where n = 1200 RPM,

Substituting all the values in equation (2.6), The frictional power loss

$$P = 221 \times \mu\ (W) \tag{2.7}$$

$$1kWh = 3.6\ MJ \tag{2.8}$$