CHAPTER 2

EXPERIMENTAL SECTION

The current chapter provides a brief account of techniques used to characterize the structure of the synthesized antiwear additives. It also covers information regarding base oil characteristics, the specification of steel ball bearings, the experimental details of testing procedures used to assess the lubricating efficiency of the antiwear additives and methods used to investigate the morphology of worn steel surfaces as well as the tribochemistry of antiwear additives.

2.1. Techniques Employed for the Study of Lubricant Additives

A Thermo Scientific Nicolet iS5 FTIR spectrometer was used to record Fourier Transform Infrared spectroscopy (FTIR) data of the potassium bromide (KBr) pellets of synthesized additives in the range of 4000-500 cm^{-1}. The identification of organic material was performed by Nuclear Magnetic Resonance (NMR) spectroscopy which provides distinguishing signals for the same functional groups with various substituents in the vicinity. The NMR spectra for ^{1}H and ^{13}C were obtained at resonance frequencies 500MHz and 126MHz, respectively, in dimethyl sulfoxide-d_6 (DMSO-d_6) as solvent using Bruker Advance III 500 MHz spectrometer. The chemical shifts were reported as parts per million (ppm) downfield from the tetramethylsilane, Me_4Si (internal reference). Ultraviolet-visible spectra of lubricant additives were captured using a Shimadzu 1700 PharmaSpec UV-VIS Spectrophotometer. Transmission Electron Microscopy; TEM has been used to see the morphological characteristics of a material, such as size, shape, and crystallographic information at high resolution. The dispersion of powdered material in C_2H_5OH was put over the carbon-coated TEM grid and investigated using the FEI-Tecnai-G2 electron microscope. Morphological pictures of additives were recorded using Field Emission Scanning Electron Microscopy, FE-SEM (FEI- Nova Nano SEM 450). For phase identification, size of the

crystalline substances, and purity, X-ray powder diffraction, XRD studies were carried out by the Rigaku Miniflex 600 XRD-System using Cu-Kα1 radiation (λ= 0.154 nm). The chemical composition of the prepared samples and the tribofilms that had formed on the worn surface was examined by X-ray Photoelectron Spectroscopy (XPS) using a K-alpha X-ray photoelectron spectrometer. Scanning Electron Microscopy; SEM with Energy-Dispersive X-ray spectroscopy (EDX) was used to provide surface magnified pictures of the wear scar and the elemental compositions of tribofilm produced on the wear scar with the help of ZEISS SUPRA 40 electron microscope, Oxford Instruments. The roughness of the worn surfaces was examined using a contact mode atomic force microscope (AFM), model no. BT 02218, Nanosurf easy scan 2 Basic AFM, Switzerland, equipped with a Si_3N_4 cantilever (Nanosensor, CONTR type) with a spring constant of roughly 0.1 Nm^{-1} and a tip radius greater than 10 nm.

2.2. Parameters of tribology

2.2.1 Mean wear scar diameter

The wear scar diameter of the three stationary balls (d_1, d_2, and d_3) for each experiment was measured with the help of the image acquisition system, and their arithmetic mean value was revealed as the mean wear scar diameter (MWD) using the equation 2.1.

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

2.2.2 Frictional force (F)

The frictional force for different antiwear additives was calculated with the help of the equation 2.2.

$$\mathbf{F} = \boldsymbol{\mu} \times \mathbf{N} \qquad 2.2$$

Where μ = Coefficient of friction,

F = Frictional force, and

N= Normal force (Total load on the three balls)

2.2.3 Frictional power loss

The frictional power loss was calculated from the antiwear test as per equation 2.3.

$$P = T.\omega \quad (2.3)$$

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

T (Frictional torque) (N.m) = F.r,

F (Frictional force, μN), N = 1.22475p, p (actual applied load) = 392 (N)

r (friction radius) = 0.14434 (inch) = 3.662×10^{-3} (m)

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

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

$$P = 221 \times \mu \text{ (watt)} \quad (2.4)$$

1kWh = 3.6 MJ

2.2.4. Wear Rate

Overall, running-in and steady-state wear rates have been calculated based on observed mean wear volume data at different time intervals. Mean wear volumes at different times (0.25, 0.50, 0.75, 1, 1.25, and 1.5 h) for each experiment were plotted with time, and a linear regression model was fitted on the points including origin to find out the overall wear rate.

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

V = mean wear volume

K = wear coefficient

l = sliding distance (2πr.N)

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

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

2.2.5. Mean wear volume

Archard wear equation was used to compute the Mean wear volume**;** MWV from the MWD data [Kumar et al. (2002), Sethuramiah et al. (2015)].

Archard wear equation

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

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

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

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

E^* = Resultant modulus of elasticity, v = Poisson's ratio, r = Steel ball radius,

$E_1 = E_2 = 206$ G Pa, $v_1 = v_2 = 0.3$, P = Actual load in Newton on each of the three horizontal balls (0.408 times of applied load), d = Steel ball mean wear scar diameter (mm),

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

2.3. Tribological testing

2.3.1. Steel ball bearing specifications

For the testing, 12.7 mm diameter AISI 52100 alloy steel balls with a hardness of 59–61 HRc were used. Before and after each test, the balls were cleaned adequately with n-hexane and thoroughly air-dried.

2.3.2. Paraffin oil (PO)

Neutral liquid paraffin oil procured from Qualigens Fine Chemicals, Mumbai, India, was used

without further purification. It is a lubricating base oil with a specific gravity of 0.82 at 25 ^{o}C, kinematic viscosities of 30 and 5.5 cSt at 40 and 100 ^{o}C, respectively, a viscosity index of 122, pour point -8 ^{o}C, cloud point -2 ^{o}C, fire point 200 ^{o}C and flash point 180 ^{o}C.

2.3.3. Testing Procedures

The prepared blends were subjected to one-hour sonication at room temperature. The synthesized lubricant additives underwent antiwear testing by ASTM D4172 test using Four-Ball Lubricant Tester from Ducom Instruments Private Limited, Bangalore, India. The tribological testing was typically conducted three times for each case.

First of all, optimization of the concentration of the investigated additives was performed under ASTM D4172 conditions; 392 N (applied load), 1200 rpm (sliding speed), 75 ^{o}C (temperature), 60min (time), noting mean wear scar diameter (MWD), and coefficient of friction (COF) for PO with and without various concentrations of additives.

The load-carrying capacity of paraffin oil and its admixtures with lubricant additives were evaluated using ASTM D5183 test standards. At first, the running-in period was completed following the test conditions; 392 N load, 600 rpm, 75 ^{o}C temperature, and 60 min duration. Further, the steady-state test was continued with a 98 N load added every 10 min until the seizure load, which denotes the failure of lubricant additives to bear that load due to excessive frictional torque. Additionally, the antiwear test was carried out upto1.5 h with a time interval of 15 min at 392N load to determine the wear rate.

2.4. Wear Scar Surface Examination

Scanning Electron Microscopy; SEM with Energy-Dispersive X-ray spectroscopy; EDX was used to provide surface magnified pictures of the wear scar and the elemental compositions of tribofilm produced on the wear scar, respectively, with the help of ZEISS

SUPRA 40 Scanning Electron Microscopy; SEM with Energy-Dispersive X-ray spectroscopy; EDX was used to provide surface magnified pictures of the wear scar and the elemental compositions of tribofilm produced on the wear scar, respectively, with the help of ZEISS SUPRA 40 electron microscope, Oxford Instruments. The roughness of the worn surfaces was examined using a contact mode atomic force microscope (AFM), model no. BT 02218, Nanosurf easy scan 2 Basic AFM, Switzerland. The chemical states of the elements present in the prepared additives and the tribofilms formed on the worn surface were examined by X-ray Photoelectron Spectroscopy(XPS) using a K-alpha X-ray photoelectron spectrometer.

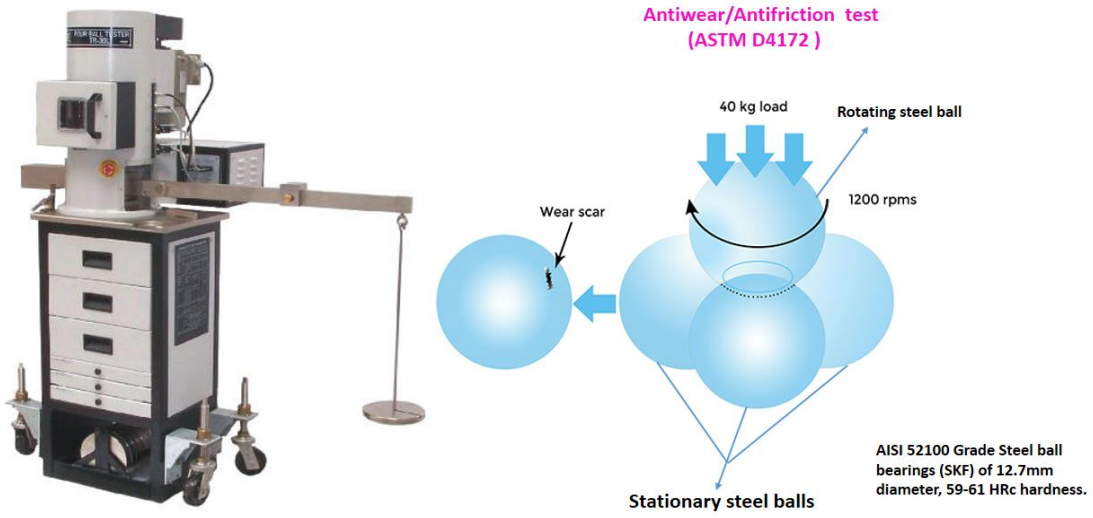

Fig. 2.1. Four ball tester machine