Abstract

With the advancement of industrialization, the demand for high-performance lubricating oils increased rapidly. In many applications, high performance is necessary, requiring distinctive specifications that are not met by conventional petroleum-based lubricants. Synthetic lubricants can fulfil a broad range of requirements, and the intent range of specific characteristics can be achieved. Synthetic lubricants would not only conserve scarce petroleum products. Still, they would also increase the life and performance of equipment, i.e., reduced maintenance and waste, higher reliability and efficiency, lower emissions and pollution, etc. Polyalphaolefins (PAOs) are one of the most popular high-performance synthetic base oils. PAOs are saturated α-olefin oligomers usually termed synthetic hydrocarbons and exhibit several inherent excellent characteristics such as high viscosity indices, wide operating temperature ranges, better oxidative and thermal stability, and lower volatility. Nowadays, PAOs acquire rapid admissibility as high-performance base oils in many niche applications such as the automobile industry as engine lubricants and gear oil, turbine oil, compressor and pump oil, automatic transmission oil, hydraulic oil, and greases, etc.

PAOs are assigned to group IV by the American Petroleum Institute (API) and are generally categorized or graded based on their kinematic viscosity at 100℃. PAO having viscosities 2, 4, and 6 cSt at 100℃ referred to as low viscosity PAOs, and 8, 10, 40, 100 cSt are considered medium to high viscosity PAOs. It is well accepted that any petroleum-based lubricants, synthetic hydrocarbons, or vegetable oils in their purest form cannot meet all of the lubricating standards established by the machine equipment/engine manufacturers. Therefore, a variety of nanoadditives are introduced in base oils to enhance the tribo-performance of base oils. In recent years different types of nanomaterials have been used

to improve the tribological performance of base oils. The present work focused on developing PAOs-based nanolubricants that must be able to replace the petroleum-based stock. In this context, four different grades of PAO (i.e., PAO 4, PAO 6, PAO 40, and PAO 100) were selected as base oils for comparative tribological investigations. The nanosized additives demonstrated excellent anti-wear and load-bearing capacity than prevailing additives. Therefore, COOH functionalized multiwalled carbon nanotubes (MWCNTs), and oleic acid-treated lanthanum trifluoride (LaF_3) were used as nanoadditives in variable concentrations.

In the first segment of thesis work, comprehensive comparisons have been made by conducting tribological experiments in fully flooded and starved lubrication conditions. In this context, four grades of PAOs (PAO 4, PAO 6, PAO 40, and PAO 100) were selected as base oils, and variable doses of MWCNTs (0.025-0.15 wt.%) were used as an additive. Further, the tribological experiments under fully flooded lubrication conditions were conducted using a four-ball tribometer as per ASTM standard. Whereas three grades of PAOs (i.e., PAO 4, PAO 6, and PAO 100) and one of the grades of polypropylene glycol (PPG 2000) containing MWCNTs as an additive were used as nanolubricants to carry out friction and wear tests using SRV5 tribometer with "ball-on-disc" configuration under starved lubrication conditions. The test results showed that PAO 6-based nanolubricants exhibited the best anti-friction (AF) performance under fully flooded lubrication conditions. In contrast, the best anti-wear (AW) performance was obtained in the case of PAO 100-based nanolubricants. The tribological results of starved lubrication conditions indicated that PAO 100-based nanolubricants demonstrated the best AF and AW performance compared to other PAOs-based nanolubricants.

The next segment of thesis work addresses the optimization of various control parameters by using Taguchi's method to assess the tribological properties of PAOs-based nanolubricants. The concentrations of MWCNTs, applied load, sliding velocity, and kinematic viscosity of PAOs were selected as process parameters or control factors. The MWCNTs at a varying concentration (0.025-0.15 wt.%) were blended separately in PAOs to formulate the nanolubricants. Tribological experimentations were performed according to Taguchi's L_{18} mixed orthogonal array by using a "ball on disc" type tribometer. The analysis of variance (ANOVA) was adopted to estimate the most prominent factors influencing the tribological performance of nanolubricants. The statistical results showed that the applied load, followed by a concentration of MWCNTs, conferred the most significant impact on the frictional characteristic. In contrast, the kinematic viscosity of PAOs, followed by the concentration of MWCNTs, has been observed as the most significant influencing factor on the anti-wear properties of nanolubricants.

The other segment of thesis work focused on the extreme pressure (EP) characteristics of different grades of polyalphaolefins (i.e., PAO 4, PAO 6, PAO 40, and PAO 100) with LaF_3 nanoparticles as an additive. The LaF_3 nanoparticles were synthesized by the sol-gel method and modified with oleic acid. The characterization techniques viz. X-ray diffractometer (XRD), Fourier transform infrared spectroscopy (FTIR), and X-ray photoelectron spectroscopy (XPS) confirmed the synthesis of LaF_3 nanoparticles. The shape, size, and microstructural characterization of LaF_3 nanoparticles were analysed using high-resolution transmission electron microscopy (HR-TEM). Varying doses (0.025-0.15 wt.%) of synthesized nanoparticles were blended in all PAOs to prepare nanolubricants. The EP properties of nanolubricants were measured using a four-ball tester as per ASTM D2783 to find the last non-seizure load (LNSL), initial seizure load (ISL), just before weld load (JBWL), weld load (WL), and load wear index (LWI). The results revealed that the incorporation of LaF_3 nanoparticles efficiently enhanced the EP properties of all PAOs. However, 0.15 wt.% of additive was the optimum dose in all PAOs, demonstrating the best

EP behaviour, i.e., the highest LWI and lowest wear scar diameter (WSD) at ISL and JBWL. PAO 100-based nanolubricants showed superior EP characteristics, whereas inferior EP properties were observed for PAO 4-based nanolubricants.

PAO 100-based nanolubricants offered outstanding AW, EP properties, and moderate AF performance compared to other PAOs based nanolubricants. Therefore, in the final part of the thesis work, PAO 100 was selected as base oil and thickened with lithium soap for obtaining stable and consistent greases. The thickener concentration was fixed at 14 wt%. The variable doses (0.025-0.15 wt.%) of nanoadditives (MWCNTs and LaF_3) were blended in the PAO 100-based grease samples. The effect of nanoadditives on the physicochemical and tribological properties of the PAO 100 greases was evaluated as per ASTM standards. The results signified the improvement of tribological performance of PAO grease in the presence of nanoadditives. Moreover, LaF_3 blended grease demonstrated better tribo-performance compared to MWCNTs blended grease. The worn surfaces of steel balls lubricated with different grease samples were analysed using scanning electron microscopy (SEM), scanning probe microscope (SPM), energy-dispersive X-ray spectroscopy (EDS), and X-ray photoelectron microscopy (XPS) techniques to understand the role of nanoadditives. The results unveiled that the deposition of nanoadditives formed a tribo-film on interacting surfaces, which protected the tribo–interfaces against friction and wear.