Investigation on the tribological performance of polyalphaolefins (PAOs) based lubricant with nanoadditives

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8. Conclusions and scope for future work

This chapter presents the key findings of the thesis work based on the tribological study of different grades of PAO oils and PAO 100-based grease with and without nanoadditives. Finally, the chapter sums up the scope for future work for further research work.

8.1. Conclusions

Polyalphaolefins (PAOs) have gained more attention due to their intrinsically excellent characteristics, such as high viscosity indices, wide operating temperature ranges, better oxidative and thermal stability, and lower volatility. Therefore, the present work is intended to develop PAOs oils (i.e., PAO 4, PAO 6, PAO 40, and PAO 100) based nanolubricants containing MWCNTs and LaF_3 nanoparticles as additives to provide an energy-efficient and sustainable alternative to conventional mineral oils. The major conclusions based on present experimental studies are summarized in subsequent sections.

8.1.1. Conclusions on the tribological performance of various grades of PAO with COOH-functionalized MWCNTs as an additive

8.1.1.1. Part-A: Conclusions on the tribological investigation of PAOs-based nanolubricants under fully flooded lubrication conditions using four-ball tribo-testing

In this part of thesis work, the COOH-functionalized multi-walled carbon nanotubes (MWCNTs) having an outer diameter of 20–30 nm and length 1−2 µm were dispersed in four different grades of polyalphaolefins (PAOs; i.e., PAO 4, PAO 6, PAO 40, and PAO 100) at various concentrations (0.025, 0.05, 0.075, 0.10, and 0.15 wt.%) to evaluate friction, anti-wear, and extreme pressure properties. The tribological test was conducted as per

ASTM standards using a four-ball tester. The comparative tribological properties of all PAO grades investigated and their salient observations are as follows:

- The tribological performance of all PAOs was improved with the addition of MWCNTs as an additive.
- PAO 6 with an optimum concentration of 0.075 wt.% MWCNTs exhibited the best friction-reducing ability (~ 31%) among all PAOs, while PAO 100 with optimum concentration revealed the worst antifriction performance. The viscosity played a vital role in the performance of COF in PAO 100. On the contrary, the concentration of 0.1 wt. % of MWCNTs in PAO 100 exhibited the best anti-wear properties. In contrast, in the case of PAO 4, PAO 6, 0.075 wt.% of MWCNTs and, for PAO 40, 0.025 wt.% was found to be an optimum concentration. PAO 100 exhibited the best anti-wear properties (i.e., minimum wear scar diameter (WSD), mean wear volume (MWV), and maximum reduction) than PAO 4, PAO 6, and PAO 40.
- MWCNTs was unable to make an influence on the extreme performance (EP) performance of PAO 4 and PAO 6 at all concentration level. The pre-seizure and weld load for both lubricants were noticed at 126 and 160 kgf, respectively, but remarkable improvement in the load-carrying capacity and EP characteristics have been observed with the MWCNTs in PAO 40 and PAO 100.
- It was concluded that optimum MWCNTs in all PAOs except PAO 4, the surface topography of worn surfaces improved significantly. The optimum concentration of MWCNTs in PAO 4 deteriorated the surface roughness of worn surfaces due to the hard and brittle nature of MWCNTs, which featured three-body abrasion between the mating interfaces.

- It has been recognized from the EDS pattern that COOH- functionalized MWCNTs have been deposited on the worn surface, which results in the formation of surface-protective tribo-film at the mating surface.

8.1.1.2. Part-B: Conclusions on the tribological investigation of PAOs-based and PPG based nanolubricants under starved lubrication conditions using SRV 5 tribo-test rig

In this section, three grades of PAOs (i.e., PAO 4, PAO 6, and PAO 100) and one of the grades of polypropylene glycol (PPG 2000) were chosen as base oil. The friction and wear studies were carried out as per ASTM D6425 by using SRV 5 tribometer with a "ball on disc" configuration. For a comprehensive comparison, this section has been divided into two parts, i.e., high viscosity base oils (i.e., PAO 100 and PPG 2000) as follows:

8.1.1.2.1. Conclusions on the tribological evaluation of high viscosity synthetic base oils (PAO 100 and PPG 2000) based nanolubricants

This work analysed the tribological performance of two high viscosity synthetic base oil, i.e., polyalphaolefin (PAO 100) and polypropylene glycol (PPG 2000). The following conclusions have been drawn:

- COOH-functionalized MWCNTs at different compositions (i.e., 0.025, 0.05, 0.075, 0.1, 0.15 wt.%) were dispersed in PAO 100 and PPG 2000. The tribological performance of both the base oils improved with the addition of MWCNTs as an additive. Overall, PAO 100 exhibited better tribological performance than PPG 2000.

- The test results showed that a maximum reduction in wear volume (WV) of around 87% was achieved in the case of PPG 2000 containing 0.025 wt.% of additive as compared to pure PPG 2000. However, PAO 100 at all concentrations of MWCNTs showed lower wear WV than PPG 2000-based nanolubricants. PAO 100 with 0.05 wt.% of additive offered the maximum reduction in the wear volume (~15%) compared to plain PAO 100.
- The introduction of 0.025 wt.% of MWCNTs in both oils exhibited a maximum reduction in friction coefficient. The test results revealed that PAO 100-based nanolubricants displayed the lowest friction coefficient compared to PPG 2000-based nanolubricants.
- The enhanced tribological performances of both base oils are attributed to good dispersion consistency of MWCNTs in the base oils and ingrained embedded stability of the additive between the mating surfaces, generation of nanolubrication effects such as rolling, mending, polishing effect, and also the formation of surface-protective tribo-film at the mating surface.
- The dispersion stability photographs of all nanolubricants demonstrated that no evidence of sedimentation was observed at all concentrations of MWCNTs even after four months

8.1.1.2.2. Conclusions on the tribological evaluation of low viscosity synthetic base oils (PAO 4 and PAO 6)

In this work, the tribological performances of two low viscosity grades of PAOs (i.e., PAO 4 and PAO 6) containing COOH-functionalized MWCNTs have been investigated by comparing their friction and wear reduction capacities under boundary lubrication regime. The following conclusion has been extracted from the present study:

- The inclusion of MWCNTs in both PAOs reduced friction coefficient, whereas PAO 6 at all additive concentrations showed better friction characteristics than PAO 4 based nanolubricants.
- The rheological results corroborated that PAO 6 offered a lower value of dynamic viscosity at a lower shear rate than PAO 4. Therefore, PAO 6 demonstrated better frictional characteristics in comparison to PAO 4.
- PAO 4 incorporating 0.05 wt.% of MWCNTs showed a maximum reduction in average friction coefficient by ~27%. In PAO 6, maximum attenuation (~7%) was acquired at a concentration of 0.075 wt.% of additive.
- The energy consumption at the mating surfaces was abated by ~27% and ~7% by adding 0.05 wt.% of MWCNTs in PAO 4 and 0.075 wt.% in PAO 6, respectively.
- The doping of 0.025 wt.% of additive in PAO 4 revealed the highest attenuation in wear volume by 88%. However, PAO 6 containing 0.05 wt.% showed the maximum reduction in wear volume (~27%).
- The appearance of higher atomic percent of carbon (C) and lower atomic percent of oxygen (O) in the EDS spectrum confirmed that MWCNTs were adsorbed on the interfaces of tribo-pairs to form a lubricating film (tribo-film), which served as an intermediator between the interacting surfaces to prevent the direct metal to metal contact.
- XPS results show that metal oxides and C bonds were formed on the worn surfaces of the ball and disc at all the lubricating conditions. Therefore, it can be deduced that the shape and size of MWCNTs play a significant role in the improvement of lubrication properties rather than tribo- chemical reactions.

The overall comparison in enhancement in antifriction (AF) and anti-wear (AW) performance of PAO grades under starved and fully flooded conditions are summarized in **Table 8.1**.

Table 8.1: Summary of overall comparative improvement in AF and AW performance of PAOs

Test rig	Base oil	Applied load (N)	AF performance				AW performance			
			Optimum dose of MWCNTs (wt.%)	COF		% Reduction in COF	Optimum dose of MWCNTs (wt.%)	MWV ($\times 10^{-4}$ mm^3)		% Reduction in MWV
				Plain	With optimum dose			Plain	With optimum dose	
Four ball tribo-tester (Fully flooded lubrication condition)	PAO 4	392	0.075	0.087	0.073	16	0.075	197	85	57
	PAO 6	392	0.075	0.051	0.036	31	0.075	60	38	36
	PAO 40	392	0.025	0.065	0.047	28	0.025	97	41	58
	PAO 100	392	0.075	0.074	0.058	22	0.1	69	27	61
SRV 5 tribometer (Starved lubrication Condition)	PAO 4	50	0.05	0.286	0.208	27	0.025	5	0.6	88
	PAO 6	200	0.075	0.183	0.168	8	0.05	3.7	2.7	27
	PAO 100	300	0.025	0.142	0.125	12	0.05	0.7	0.6	14
	PPG 2000	300	0.025	0.218	0.206	6	0.025	80	11	87

8.1.2. Conclusions on Taguchi's optimization of various parameters for tribological performance of polyalphaolefins-based nanolubricants

In this segment of thesis work, Taguchi's robust design method was used to analyse the influences of applied load, sliding velocity, the kinematic viscosity of PAOs, and concentration of MWCNTs on the tribological performance of nanolubricants. The most prominent factor and optimal combination of factors that regulate the tribological properties of nanolubricants are determined. Analysis of variance (ANOVA) is executed to assess the significance of four control factors. The friction and wear properties of nanolubricants have been evaluated with the assistance of "ball on disc" type tribo-test rig. Based on the present study, the following conclusions are drawn:

- The optimal combination of control factors that offer the minimum COF was found to be as A_3 (0.05 wt.% concentration of MWCNTs), B_3 (applied load of 100 N), C_3 (1.57 m/s sliding velocity), and D_2 (6×10^{-6} m^2/s kinematic viscosity, i.e., PAO 6). While for minimum specific wear rate, the best combination of control factors was obtained at A_4 B_2 C_3 D_3 (i.e., the concentration of MWCNTs=0.075 wt.%, applied load=80 N, sliding velocity=1.57 m/s, and kinematic viscosity=40×10^{-6} m^2/s (PAO 40)).
- The ANOVA analysis illustrated that the applied load with a percentage contribution of 47.1% was found to be the most prominent control factor that influenced the COF, followed by concentration of MWCNTs (39.3%) and kinematic viscosity (6.6%) and sliding velocity (3.7%). In contrast, kinematic viscosity (38.6%) was the factor that exhibited the most significant impact on

specific wear rate, followed by concentration of MWCNTs (28.1%) and applied load (26%), and sliding velocity (4.9%).

- The morphological and topographical analysis of the worn surfaces revealed that comparatively, a smoother surface with slender furrows was noticed when balls were lubricated with PAOs containing an optimal dose of MWCNTs.
- The appearance of C peak in the EDS spectra of worn surfaces demonstrated the formation of lubricating oil film containing the MWCNTs, which acts as a spacer between friction pairs to avoid the direct metal to metal contact.
- The stability analysis of nanolubricants showed no sign of sedimentation of additive even after 120 days, thus demonstrating MWCNTs are potential for commercialization as an additive.

8.1.3. Conclusions on the influence of oleic acid-treated LaF_3 nanoparticles as an additive on extreme pressure properties of various grades of polyalphaolefins

A comparative study on EP properties of different grades of PAOs with the addition of lanthanum trifluoride (LaF_3) nanoparticles as an additive is reported. The sol-gel method was adopted to synthesis the LaF_3 nanoparticles and modified with oleic acid (OA). Two low viscosity PAOs (i.e., PAO 4 and PAO 6) and two high viscosity PAOs grades (i.e., PAO 40 and PAO 100) were chosen as base oils. The synthesized nanoparticles at varying concentrations (0.025-0.15 wt.%) were dispersed in all PAOs to develop the nanolubricants. The extreme pressure properties of nanolubricants were obtained using a four-ball tester as per ASTM D2783. The following conclusions have been deduced from this investigation:

- The dispersion of LaF_3 nanoparticles in all PAOs led to enhancement in the EP properties, but 0.15 wt.% dose of LaF_3 nanoparticles in all grades of PAOs exhibited excellent EP behaviour.
- The inclusion of LaF_3 nanoparticles in PAO 4 and PAO 6 could not impact just before weld load (JBWL) and weld load (WL) at all additive doses. JBWL and WL for both PAOs at all concentration levels were 1236 N and 1570 N, respectively. Although, LaF_3 (0.025-0.15 wt.%) in PAO 4 and PAO 6 exhibited significant improvement in EP properties, i.e., higher load wear index (LWI), lower WSD at initial seizure load (ISL), and JBWL compared to plain PAO 4 and PAO 6.
- PAO 40 incorporating 0.15 wt.% LaF_3 offered the highest LWI and lowest WSD at ISL and JBWL than plain PAO 40 and exhibited the maximum augmentation in LWI, approximately 13%. In contrast, WL was increased to 2452.5 N when 0.15 wt. % dose of additive was dispersed in PAO 100.
- The morphological and topographical analysis of worn surfaces revealed the smallest wear scar and smooth worn surfaces even under higher loading conditions in PAO 100 containing 0.15 wt.% LaF_3.
- It has been recognized from the EDS pattern that OA-modified LaF_3 nanoparticles have been deposited on the worn surface, which results in the formation of surface-protective tribo-film at the mating surface.
- Overall, PAO 100-based nanolubricants demonstrated superior EP characteristics (i.e., higher LWI, higher WL, and minimum wear scar at ISL and JBWL). In contrast, inferior EP properties were observed in the case of PAO 4-based nanolubricants.

8.1.4. Conclusions on tribological evaluation of PAO 100 oil-based grease

The comparative tribological study of PAO 100 oil-based lithium grease incorporating variable concentrations of COOH-functionalized MWCNTs and OA-modified LaF_3 nanoparticles as additives are addressed. The concentration of nanoadditives was ranged from 0.025 to 0.15 wt.%. PAO 100 oil-based grease was used as a reference for comparing the various physicochemical and tribological properties of PAO grease containing nanoadditives. The four-ball tester was used to assess the lubrication behaviour of all grease samples as per ASTM standards. The following observations are made:

- The consistency of PAO grease deteriorated in the presence of MWCNTs and LaF_3 nanoadditive.
- The incorporation of nanoadditive exhibited the marginal improvement in the drop point of PAO grease.
- The optimized dose of MWCNTs (0.05 wt.%) and LaF_3 nanoparticles (0.075 wt.%) revealed a significant reduction in COF, WSD, and MWV.
- The presence of an optimized dose of MWCNTs and LaF_3 in PAO grease conserved the energy by ~ 14% and ~ 44%, respectively.
- The lower concentration of both nanoadditive did not contribute to enhancing the EP properties of PAO grease. However, a remarkable augmentation in EP performance of PAO grease was obtained at the higher dose of both nanoadditive.

8.2. Overall general conclusions according to the problem definition

The overall conclusions of the thesis work are as follows:

- The dispersion stability analysis of both nanoadditive in PAOs depends on the viscosity.
- The lower concentration of MWCNTs and LaF_3 did not show good dispersion stability in low viscosity PAOs (PAO 4 and PAO 6).
- PAO 40 and PAO 100 exhibited good suspension stability at all concentrations of MWCNTs (~ 120 days) and LaF_3 nanoadditive (~15 days) due to higher viscous characteristics than PAO 4 and PAO 6.
- PAO 4-based nanolubricants revealed the poorest dispersion stability and poor tribological performance than other PAOs based nanolubricants.
- PAO 6-based nanolubricants demonstrated the best anti-friction characteristics, whereas PAO 100-based nanolubricants revealed the best anti-wear performance.
- A lower concentration of nanoadditives (optimum concentration) enhanced the antifriction and antiwear properties of all PAOs. In contrast, the higher concentration of both nanoadditive led to improvement in the EP characteristics of high viscosity PAOs (i.e., PAO 40 and PAO 100).
- It was also observed that MWCNTs exhibited excellent anti-wear properties than friction properties under both lubrication conditions (i.e., fully-flooded and starved).
- However, the minute dosage of nanoadditives in all PAOs grades and PAO grease has shown remarkable improvements in AF and AW properties.
- The tribological results of PAO grease with nanoadditive demonstrated that smaller size and spherical shape nanoadditive (i.e., LaF_3) exhibited comparatively better

antifriction and anti-wear properties compared to a relatively larger size and tubular shape nanoadditive (i.e., MWCNTs)

- The promising physicochemical and tribological results of PAO grease suggest that PAO 100-based greases can be suitable alternatives to conventional mineral oil-based greases.

8.3. Scope for the future work

Although the present work provides detailed insight into the potential of PAOs based nanolubricants for tribological contact situations, a few more research areas could be carried out forward:

- Since the low viscosity PAOs exhibited better anti-friction performance while higher viscosity PAOs showed superior anti-wear behaviour. Therefore, the tribo-performance of the blend of two or more PAOs grades can be explored.
- In addition, the effect of temperature and load variation on tribo-performance of PAOs blended with/without nanoadditive can be examined.
- The synthesis of various hybrid nanoadditives of different morphology and their dispersion stability and influence on the tribo-performance of PAOs or PAOs blend needs to be investigated.
- PAOs blend-based greases with and without nanoadditives can be developed for semi-solid lubrication conditions, and their tribo-performance can be explored under various operating conditions.