

6 Tribological evaluation of coconut oil-based greases with synergistic effect of additives

This chapter addresses the synergistic lubrication effects of two different types of nanomaterials (spherical particles and lamellar sheets) as additives to coconut oil-based grease formulation for providing the energy-efficient and sustainable alternatives to conventional grease. The spherical SiO₂ nanoparticles are separately blended with 2D lamellar sheets of MoS₂ and graphene oxide (GO) in variable ratio and probed their synergistic effects on physicochemical and lubrication properties. The combination of rolling friction and low shear properties using the synergistic blend of the spherical nanoparticles (SiO₂) and nanosheets (MoS₂ and GO) showed significant improvement in tribological properties.

6.1 Characterization of nanoadditives

Figure 6.1 shows the TEM images of SiO₂ nanoparticles. The SiO₂ nanoparticles exhibit the spherical shape with their diameter ranging from 20 to 35 nm. **Figure 6.2a** displays XRD patterns of SiO₂ nanoparticles. A broad diffraction peak at 2θ of 23.5° signified the 101 plane of SiO₂ as per the JCPDS card NO. 89–8951. The notably broader XRD peak suggests the amorphous nature of SiO₂ nanoparticles (Tzounis et al., 2014). **Figure 6.2b** depicts the FTIR vibrational spectrum of SiO₂ nanoparticles. A broad and strong vibrational peak at $\sim 1095\text{ cm}^{-1}$ is noted owing to an asymmetrical stretching of Si–O–Si (Rahman et al., 2007). Another sharp and intense peak is appearing because of the bending mode of Si–O–Si at 476 cm^{-1} (X. Wang et al., 2010). A small vibrational peak at $\sim 948\text{ cm}^{-1}$ is ascribed to the bending of Si–O–H (Vansant et al., 1995). These vibrational peaks confirmed the synthesis of SiO₂ nanoparticles. A broad peak is marked in the range between 3100 and

3600 cm^{-1} is assigned to the stretching mode of O–H (Rahman et al., 2007). The detailed characterizations of MoS₂ and GO nanosheets characterizations are already presented in the previous **Sections 4.1 and 4.2**.

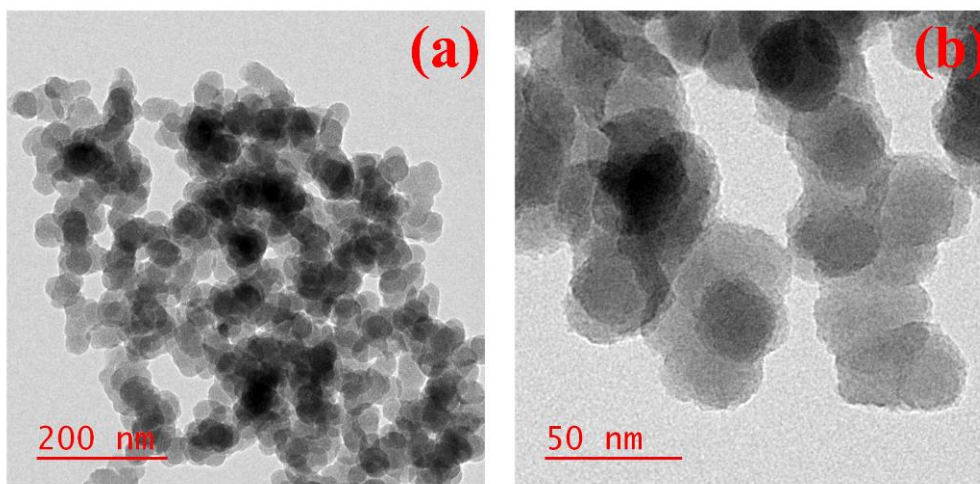


Figure 6.1: Low and high-resolution TEM images of (a–b) SiO₂ nanoparticles

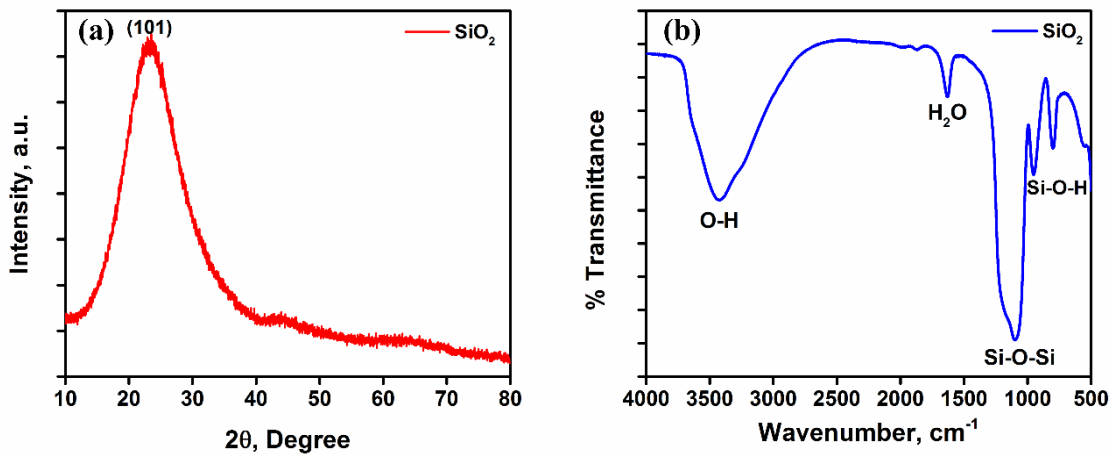


Figure 6.2: (a) XRD pattern (b) FTIR spectrum of SiO₂ nanoparticles

6.2 Physicochemical characterization of coconut greases

The coconut oil-based lithium grease without any nanoadditives is termed as *coconut grease*. The dosage of the nanoadditives mixture was fixed to 0.05 wt% for all formulations. The ratio of MoS₂ to SiO₂ and GO to SiO₂ were varied in five combinations, i.e., 100:0;

70:30; 50:50; 30:70; and 0:100 for the formulation of coconut grease samples. The coconut grease samples having thoroughly dispersed $\text{MoS}_2/\text{SiO}_2$ and GO/SiO_2 nanoadditives are termed as $\text{MoS}_2/\text{SiO}_2$ grease and GO/SiO_2 grease, respectively. Further, coconut grease was used as a benchmark to compare the physicochemical and tribological properties and emphasize the roles of nanoadditives.

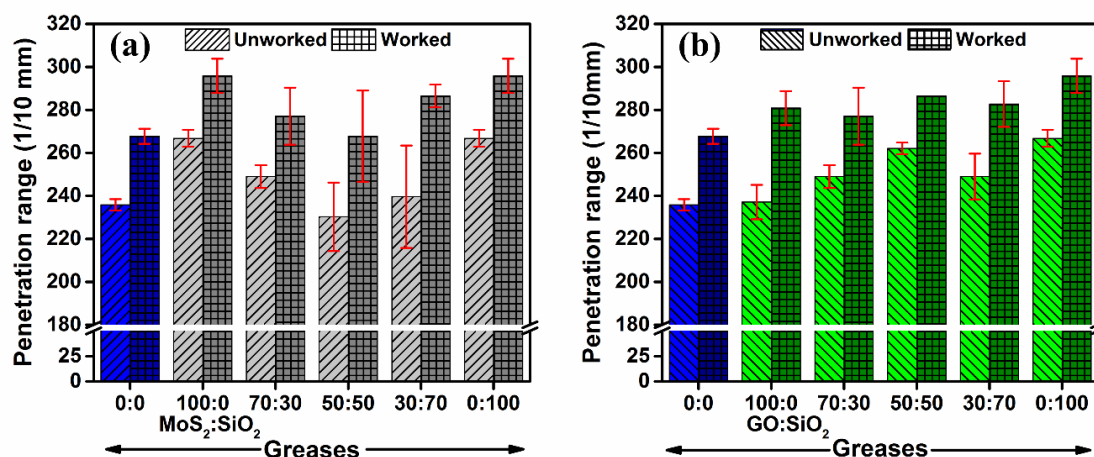


Figure 6.3: Unworked and worked penetration measurements of coconut grease, having 0.05 wt% nanoadditives. The combinations of (a) $\text{MoS}_2/\text{SiO}_2$, and (b) GO/SiO_2 in their variable ratios are used as nanoadditives to coconut grease

The spherical silica nanoparticles, combined with two-dimensional (2D) lamellar materials, i.e., MoS_2 and GO sheets in variable weight ratio, were thoroughly blended during the formulation of coconut grease. Two different combinations based on spherical particles and lamellar sheets are selected to explore their synergistic effects on the physicochemical and tribological properties of coconut grease. **Figure 6.3** shows the cone penetration results of coconut, $\text{MoS}_2/\text{SiO}_2$, and GO/SiO_2 nanoadditives blended samples of coconut grease. The unworked penetration depths revealed the NLGI grade 3 consistency for all samples of coconut grease. The grease samples were sheared and examined for the worked penetration depths, which indicated shear stability. After 60 double strokes, the coconut grease samples showed the NLGI grade 2 consistency. Notably, the blending of

nanoadditives in coconut grease couldn't make any measurable changes and maintained the NLGI grade 2 consistency, equivalent to conventional lithium greases. The variation in unworked and worked penetration indicates the poor shear stability of the formulated greases. The bio-greases prepared by trans-esterified Karanja oil and lithium soap too exhibited poor shear stability (Panchal et al., 2015).

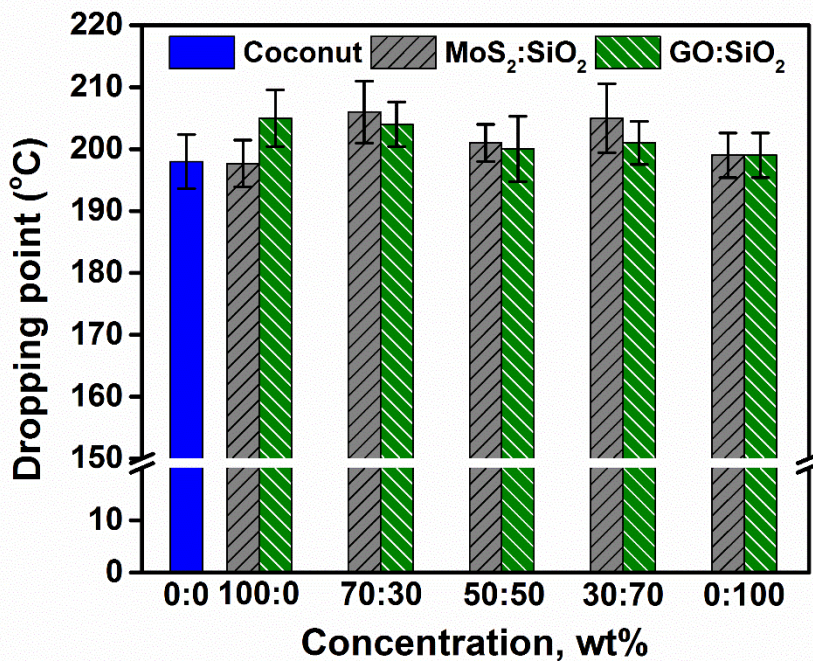


Figure 6.4: Dropping point measurement of coconut grease having 0.05 wt% of nanoadditives. The combinations of MoS₂/SiO₂, and (b) GO/SiO₂ in their variable ratios are used as nanoadditives to coconut grease

The semi-solid phase of grease changed into a liquid with a function of temperature. When oil starts to drop from the grease sample at a particular temperature, it is known as the dropping point, and it addressed the retainability of lube oil in the grease matrix. **Figure 6.4** shows the changes in dropping point when variable combinations of MoS₂/SiO₂ and GO/SiO₂ binary nanoadditives are incorporated in the coconut grease. The dropping point of coconut grease was found to be 198 °C, which increased to 205 and 203 °C in the presence of thoroughly blended MoS₂/SiO₂ and GO/SiO₂ nanoadditives, respectively. The

70:30 wt% ratio of both combinations ($\text{MoS}_2/\text{SiO}_2$ or GO/SiO_2) showed maximum enhancement in their dropping point.

6.3 Tribological performance of greases on four-ball tester

The engineering systems, such as gears, ball-bearing, cams, etc., require proper lubrication for smooth operation and extended life. The high friction leads to wastage of energy in the form of heat, which eventually facilitates the wearing of the materials from contact interfaces and deteriorates the life of engineering tools. The optimum lubrication between the interacting surfaces minimizes the friction, subsidizes the material loss due to wear, and dissipates the heat from contact interfaces. The tribological performance of coconut grease with and without nanoadditives in the combination of $\text{MoS}_2/\text{SiO}_2$ and GO/SiO_2 were assessed for steel tribo-pair under the boundary lubrication condition. **Figure 6.5** displays the changes in coefficient of friction (COF), wear scar diameter (WSD), and mean wear volume (MWV) of coconut grease in the presence of binary nanoadditives for steel tribo-pair. The coconut grease showed COF at 0.084, which reduced by ~3%, ~10%, and ~10% in the presence of MoS_2 , GO, and SiO_2 nanomaterials, respectively. The binary combinations of $\text{MoS}_2/\text{SiO}_2$ and GO/SiO_2 in variable wt% ratio as nanoadditives to coconut grease exhibited lower COF than individual materials. The $\text{MoS}_2/\text{SiO}_2$ combination in 30:70 ratio and GO/SiO_2 combination in 50:50 ratio decreased the COF by ~20% and ~19%, respectively. It could be attributed to the synergistic effect furnished by spherical SiO_2 nanoparticles and the 2D lamellar sheets of MoS_2 and GO. Likewise, the MWV of steel balls lubricated with $\text{MoS}_2/\text{SiO}_2$ (30:70) and GO/SiO_2 (50:50) blended coconut grease reduced to ~37% and ~25%, respectively. The coconut oil exhibit inherently low frictional properties; however, it lacks the antiwear (AW) characteristic, particularly under the boundary lubrication regime (Jayadas et al., 2007). Herein, the synergistic combination of

two different types of materials enhanced the friction–reducing properties of coconut grease and decreased wear by furnishing the AW properties.

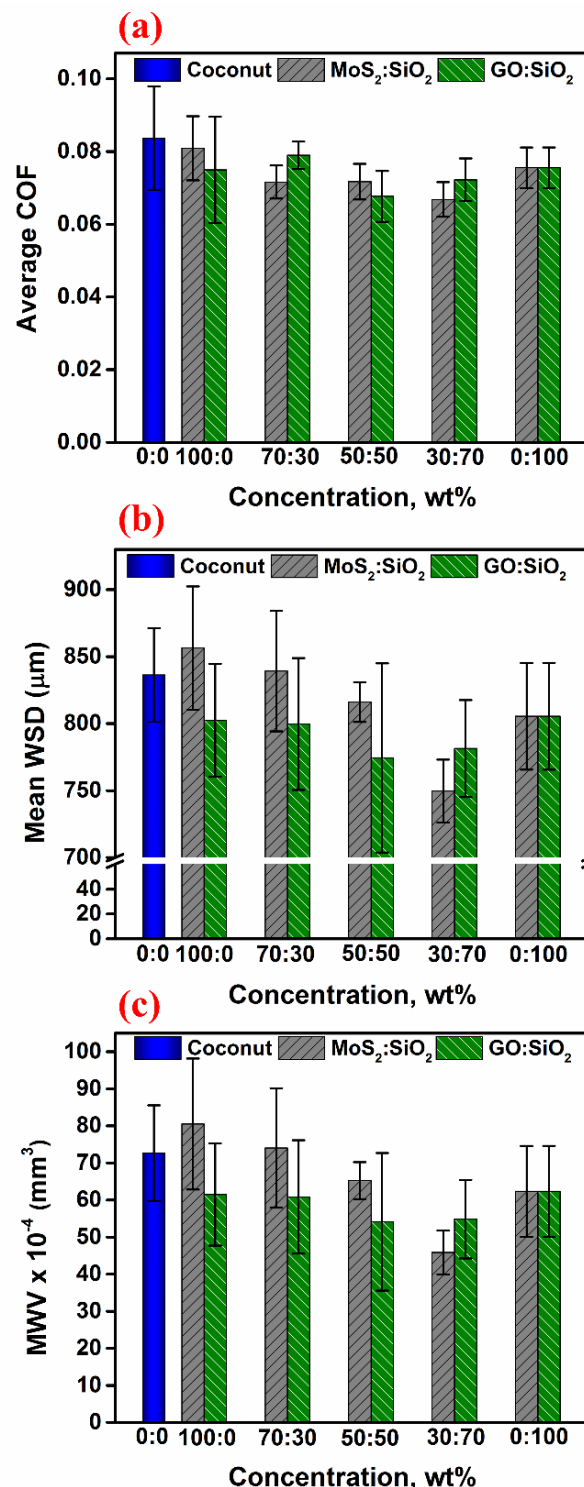


Figure 6.5: Changes in (a) COF, (b) WSD, and (c) MWV for steel balls using the coconut grease blended with binary combinations of MoS₂/SiO₂ and GO/SiO₂ as nanoadditives. The wt% ratio of individual nanomaterials in each combination is varied to obtain the optimized tribo–performance. (Applied load: 392 N, test duration: 60 min)

The extreme pressure (EP) behavior of coconut grease was estimated by measuring the weld and non-seizure loads, which were found to be 160 and 126 kgf, respectively. The load-bearing capacity of lithium grease, formulated by using paraffin oil, was found in the same range. These results suggest that coconut oil-based grease has an equivalent load-bearing capacity as paraffin oil-based grease, and it signified the potential of vegetable oil-based grease as an alternative to conventional greases. The incorporation of nanoadditives in variable combinations showed no measurable improvement in load-bearing capacity. It is further supported by recent findings using 2.0 wt% of polytetrafluoroethylene (PTFE) nanoadditives to titanium complex grease (Chen, 2010).

Table 6.1: Energy saving by use of binary combinations of MoS₂/SiO₂ and GO/SiO₂ as nanoadditives to coconut grease.

Wt% ratio of nanoadditives in a binary system	MoS ₂ /SiO ₂ blended grease		GO/SiO ₂ blended grease	
	Power consumption (MJ)	% Reduction in power consumption	Power consumption (MJ)	% Reduction in power consumption
Coconut	66.6	—	66.6	—
100:0	64.4	3	59.7	10
70:30	57.0	14	62.9	6
50:50	57.1	14	53.9	19
30:70	53.2	20	57.5	14
0:100	60.1	10	60.1	10

A significant fraction of energy (23%) is utilized to overcome the friction in elastohydrodynamic lubrication (EHL) rolling contacts in paper mills (Holmberg et al., 2013). Therefore, adequate lubrication practices can minimize friction losses. In the present work, the amount of energy consumed to overcome the friction during each test was assessed, and the percentage reduction in energy consumption by using the variable combinations of MoS₂/SiO₂ and GO/SiO₂ as nanoadditives to coconut grease is outlined in

Table 6.1. The coconut grease utilized a maximum amount of energy, i.e., 66.6 MJ, to overcome the friction during the tribo-test. The binary combinations of MoS₂/SiO₂ (30:70), and GO/SiO₂ (50:50) reduced the energy consumption by ~20%, and ~19%. However, individual SiO₂, MoS₂, and GO nanoadditives in the coconut grease have conserved the energy by ~10%, ~3%, ~10%, respectively. These results revealed that the synergistic effect of nanoadditives has could save energy almost double of their constituent materials.

6.4 Evaluation of worn surfaces

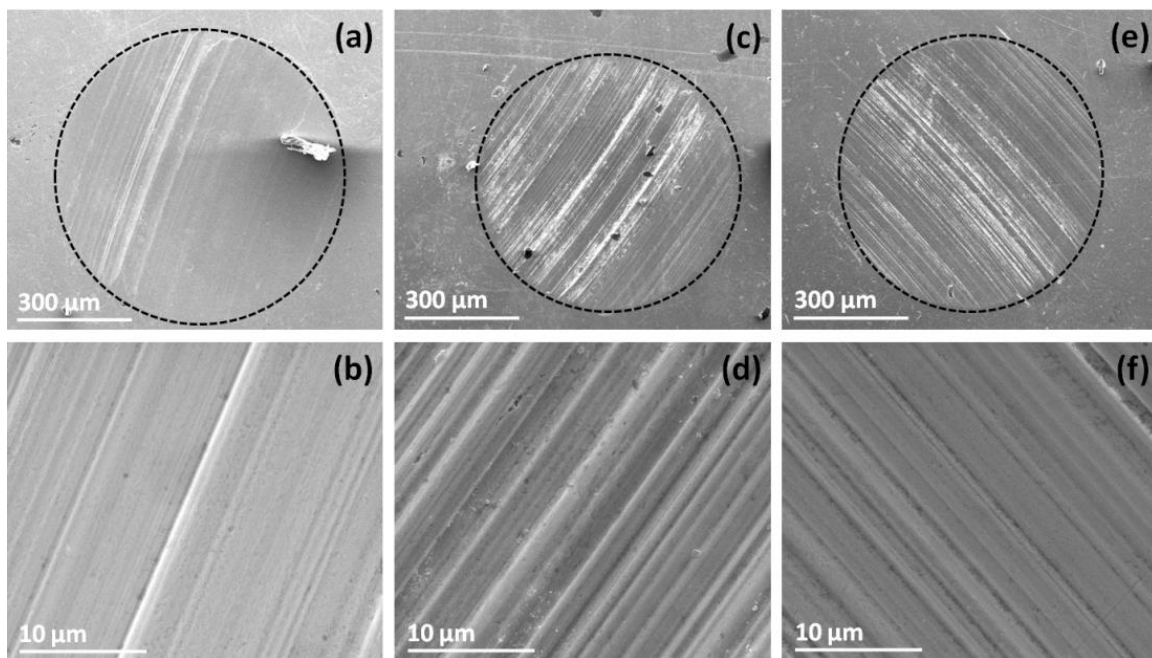


Figure 6.6: SEM images of worn steel balls lubricated with (a–b) coconut grease, (c–d) MoS₂/SiO₂ blended grease, and (e–f) GO/SiO₂ blended grease. (Applied load: 392 N, test duration: 60 min)

As a result, the coconut grease lubricated the contact interfaces of steel balls and furnished the low friction ($\mu = 0.084$). The steel balls lubricated with coconut grease developed wear scar of 835 μm diameter (**Figure 6.6a**), significantly lower than the paraffin grease-lubricated steel balls. The formation of fatty-acids derived protective thin film of low shear strength is accountable for the lubrication effect by coconut grease. The blending of binary

combinations of GO/SiO₂ and MoS₂/SiO₂ nanoadditives further improved the AW properties; thus, their presence between the tribo–interfaces led to a significant reduction in WSD (**Figure 6.6c,e**). The steel balls lubricated with MoS₂/SiO₂ blended coconut grease developed the smallest wear among all samples. It could be attributed to significantly low shearing properties due to weak van der Waals interaction between the MoS₂ lamellae. In addition, the presence of oxygen functionalities in the basal plane of GO might compromise the low shearing properties. Therefore, GO/SiO₂ sheets exhibited a higher COF than the MoS₂/SiO₂ (30:70) binary system.

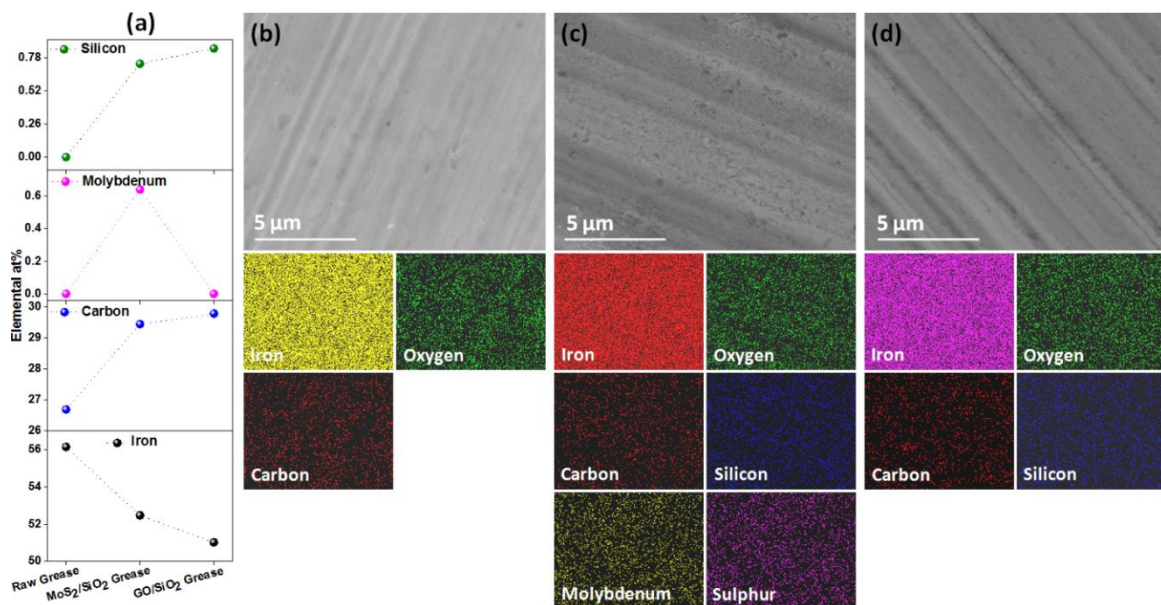


Figure 6.7: (a) Quantitative estimation of different elements in the tribo–film deposited over the worn scars lubricated with coconut grease and nanoadditives blended grease samples. Microscopic images along with the corresponding area elemental distribution of worn scars of steel balls lubricated with (b) coconut grease, (c) MoS₂/SiO₂ blended grease, (d) GO/SiO₂ blended grease. (Applied load: 392 N, test duration: 60 min)

The at% of contributory elements in the tribo–film over the worn scar based on the EDS measurements explicitly expressed the role of fatty acids of coconut grease and nanoadditives. As shown in **Figure 6.7a**, the significant presence of carbon (26 to 32 at%) signified the role of coconut oil–based fatty acids. Furthermore, the thorough distribution

of carbon across the worn scars of steel balls lubricated with coconut and nanoadditives blended grease samples suggest uniform tribo-film formation. The distribution of Mo, S, and Si over the worn scar (**Figure 6.7c**) lubricated with MoS₂/SiO₂ blended grease confirmed the roles of these nanomaterials in tribo-film formation. Moreover, XPS analysis of the worn surface of steel balls is carried out to further understand the tribo-chemical thin films.

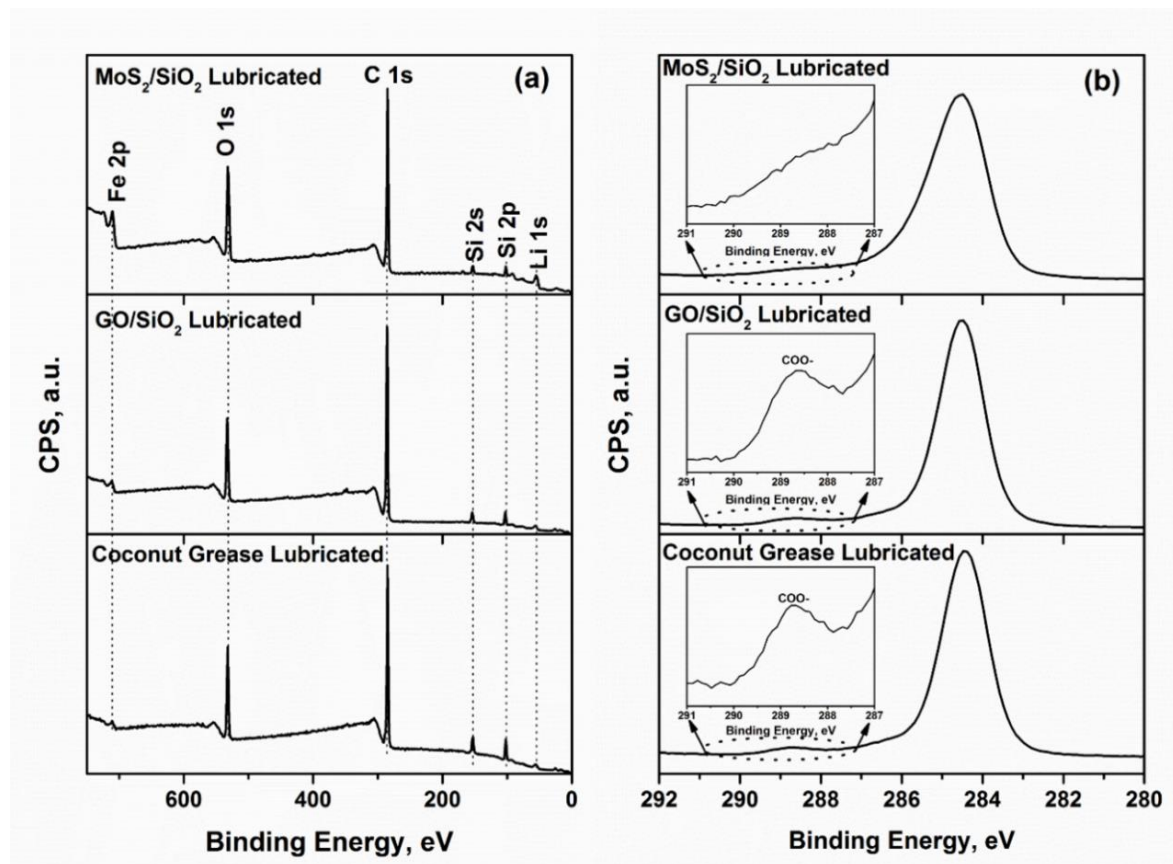


Figure 6.8: XPS (a) survey and (b) C 1s spectra of worn surfaces of steel balls lubricated with coconut grease, GO/SiO₂ blended grease, and MoS₂/SiO₂ blended grease. The binding energy range of 287–291 eV is emphasized as Inset graphs of each C 1s spectrum to reveal the role of carboxylic groups in thin film formation

The XPS spectra of worn surfaces of steel balls lubricated with coconut grease, GO/SiO₂ blended grease, and MoS₂/SiO₂ blended grease is depicted in **Figure 6.8**. The Fe 2p, O 1s, C 1s, Si 2p, and Li 1s peaks in survey spectra of all samples (**Figure 6.8a**) suggested the preparation of coconut grease-based lubricating thin film over the steel substrate. High-

resolution C 1s spectrum (**Figure 6.8b**) of coconut grease–lubricated steel ball with a peak maximum at 284.5 eV is attributed to deposition of coconut oil and thickener over the worn scar, facilitated by tribo–stress. The presence of shoulder at higher binder energy in the in C 1s spectra of worn steel balls lubricated with coconut grease and GO/SiO₂ blended grease, suggested the participation of fatty acids–based molecules and GO for tribo–chemical thin film formation (Ji et al., 2011; Paul et al., 2019). The presence of the carboxyl group is attributed to fatty acids constituent of coconut oil and GO nanosheets. Moreover, the lack of carboxyl group peak in worn steel ball lubricated with MoS₂/SiO₂ blended grease indicated that MoS₂ nanosheets play a significant role in forming tribo–chemical thin film. The O 1s peak, along with Fe 2p feature in survey spectra of all samples, suggested the formation of an iron oxide–based product over the worn surfaces of steel balls (Liu et al., 2012; Niu and Qu, 2018). However, the at% of these elements is noted to be significantly lower than the carbon–based products. The Li 1s feature at 54.7 eV is attributed to lithium soap, a prime constitutes to grease. The presence of carboxyl group and lithium indicate that the worn surfaces were lubricated with a combined effect of thickener and base oil. Further, nanoadditives boost the tribological performance of the greases.

Figure 6.9 shows high–resolution Mo 3d and S 2p XPS spectra of worn surfaces of steel balls lubricated with coconut grease, GO/SiO₂ blended grease, and MoS₂/SiO₂ blended grease. The lack of distinct peak features due to Mo 3d and S 2p on worn steel balls lubricated with coconut grease and GO/SiO₂ blended grease rules out the participation of MoS₂ nanosheets for the tribo–chemical thin film formation. However, the residual contents of these elements could be attributed to the presence of Mo and S in the steel balls. The steel ball lubricated with MoS₂/SiO₂ blended grease displayed strong signatures of Mo 3d and S 2p at 232.4 and 168.0 eV, respectively (Chouhan et al., 2020b). The broader peaks

due to Mo 3d and S 2p are attributed to their spin-orbit splitting features, i.e., Mo 3d_{3/2}, 3d_{5/2} and 3p_{1/2}, 2p_{3/2}, respectively. These results suggest the participation of MoS₂ based nanosheets in tribo-chemical thin film formation over the lubricated surfaces of steel balls under the tribo-stress. The position of S 2p peak at 168.0 eV (**Figure 6.9b**) on the worn surface of steel ball lubricated with MoS₂/SiO₂ blended grease indicated the formation of FeSO₄ (Hu et al., 2017). The signature of Mo and S along with carbon over the worn steel balls confirmed the role of MoS₂ and coconut oil for establishment of a lubricious tribo-film.

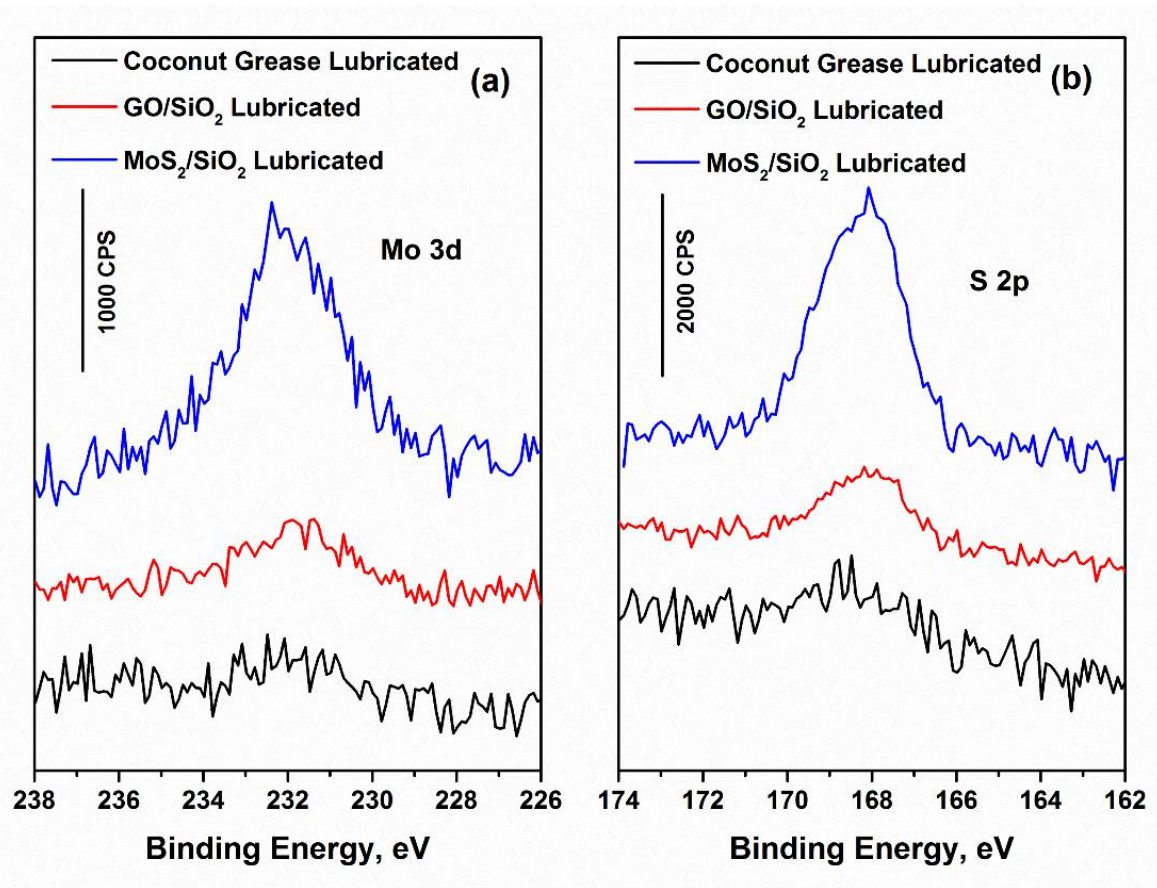


Figure 6.9: (a) Mo 3d and (b) S 2p spectra of worn steel balls lubricated with coconut grease, GO/SiO₂ blended grease, and MoS₂/SiO₂ blended grease

The surface roughness parameters (S_a , S_q , S_{sk} , S_{ku} , S_{pk} , S_k , S_{vk} , S_{r1} , S_{r2}) are important factors for engineering surfaces (Gadelmawla et al., 2002). The influence of surface roughness

parameters on the friction behavior of greases is assessed consciously. The three-dimensional (3D) and two-dimensional (2D) topographic images, surface roughness profile, and bearing area ratio curve (BAC) of the worn scars lubricated under the boundary lubrication condition are presented in **Figure 6.10**. The 3D morphology of the worn surface of steel ball lubricated with coconut grease (**Figure 6.10a**) showed the stick-slip phenomenon as the main cause of adhesive wear. The corrugated topographic features appeared mostly on the worn surface of steel balls lubricated with coconut grease compared to MoS₂/SiO₂ and GO/SiO₂ blended coconut grease. The steel ball lubricated with MoS₂/SiO₂ blended coconut grease exhibited deep valleys, which are also unambiguously seen in the SEM micrographs (**Figure 6.10c–d**). The GO/SiO₂ nanoadditives blended coconut grease attained the superior surface smoothness among all samples (**Figure 6.10g–i**). The various surface roughness parameters of worn scars were evaluated through the BAC and summarized in **Table 6.2**. The skewness (S_{sk}) indicates the symmetry of the surface profile with reference to the mean line. It is very susceptible to possible high peaks and deep valleys. The surface profiles mainly distributed with peaks show *positive skewness*, whereas the surface profiles predominantly covered with deep valleys bear *negative skewness*. Therefore, a surface with positive skewness means that the surface comprises more peaks whose heights are nearby to the mean as contrasted with the Gaussian distribution. The surface profiles of worn steel balls lubricated with coconut grease show positive skewness, while MoS₂/SiO₂ and GO/SiO₂ blended greases demonstrated negative skewness. The negative skewed surfaces have more lubricant retention characteristics as compared to positive skewed surfaces. The kurtosis (S_{ku}) provides information on the sharpness of the spikes concerning the mean line. The lower value of the distribution curve ($S_{ku} < 3$) is referred to as *platykurtic*, which indicates a bumpy surface. The higher value of the distribution curve ($S_{ku} > 3$) is termed as *leptokurtic*,

which designates a spiky surface (Gadelmawla et al., 2002). Herein, the worn surfaces of all samples classified as leptokurtic surface since they have $S_{ku} > 3$.

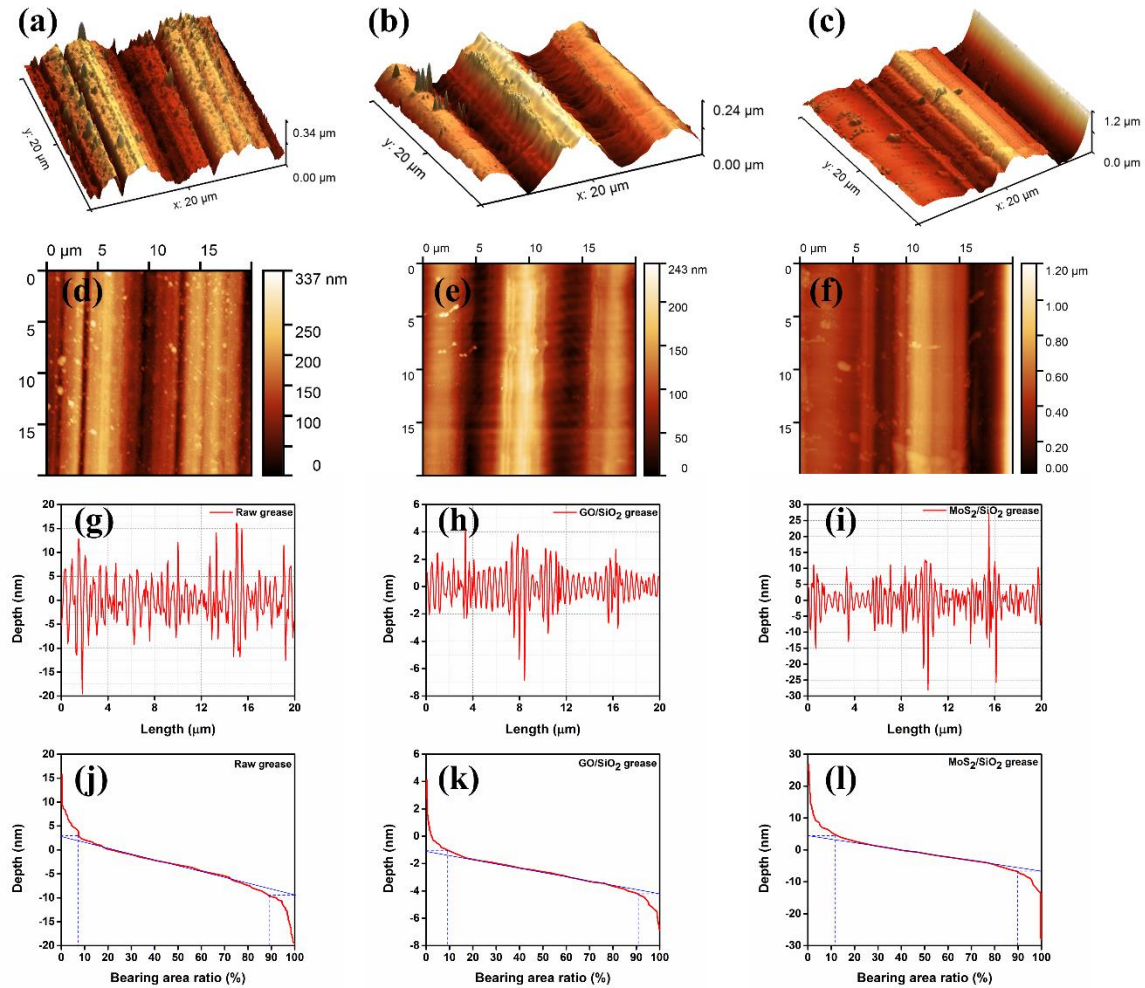


Figure 6.10: (a–c) 3D and (d–f) 2D topographic images, (g–i) roughness profiles, and (j–l) bearing area ratio curve of worn steel balls lubricated with coconut grease, GO/SiO₂ blended greases, and MoS₂/SiO₂ blended grease. (Applied load: 392 N, test duration: 60 min)

Table 6.2: Surface roughness parameters of worn surfaces of steel balls lubricated with coconut grease, MoS₂/SiO₂ blended grease, and GO/SiO₂ blended grease

Greases	S_a (nm)	S_q (nm)	S_{pk} (nm)	S_k (nm)	S_{vk} (nm)	$S_{r1}\%$	$S_{r2}\%$	S_{sk}	S_{ku}
Coconut	51	58	13	12	10	7.28	89.27	0.09	3.97
GO/SiO ₂	41	48	5	3	3	9.25	90.84	-0.42	5.46
MoS ₂ /SiO ₂	148	197	22	11	21	11.61	89.85	-0.59	7.45

The BAC comprises all statistics of the surface profile. The top segment of the curve indicates the reduced peak height (S_{pk}), which is scuffed away during the running-in period. The middle part of the curve denotes the core roughness depth (S_k), and it indicates a functional depth of the core profile. After completion of the running-in period, the surface bears the load and influences performance and life. The lower segment of the curve stands for reduced valley depth (S_{vk}), which preserves the lubricant. The peak material component (S_{r1}) represents the percentage of BAC that intersects with the upper limit of the S_k . The valley material component (S_{r2}) denotes the percentage of BAC that intersects with the lower limit of the S_k . The assessment of BAC shows that the worn surface of steel balls lubricated with MoS₂/SiO₂ blended coconut grease has the highest S_{vk} , and it signified the good retention capacity of lubricating grease. The retained grease in the valleys helpful for the lubrication process and maintains a stable tribo-film. The worn surface lubricated with MoS₂/SiO₂ blended coconut grease has the highest negative skewness. The synergistic effect of S_{vk} and S_{sk} nourishes the interacting surfaces effectively.

6.5 Discussion

The maximum contact pressure of ~3.4 GPa was developed between the steel balls under the applied load of 392 N. The ~3.4 GPa Hertzian contact stress is significantly high to squeeze and shear the grease structure between the contact interfaces of steel balls. Moreover, the shear-thinning characteristics of grease increased the shear rate. The tribo-experiments were performed at 75 °C and the heat generated at the contact zone because of frictional force further increase the temperature, which led to the softening of the grease. The high temperature and shear rate compromised the strengthening of the fibrous network of coconut grease, which resulted in bleeding of the trapped coconut base oil. The thoroughly dispersed nanoadditives are also released and lubricated the tribo-interfaces.

The excellent wettability of coconut oil (**Figure 3.1c**) has excellent potential to lubricate the tribo-surfaces effectively by forming a tribo-film.

The morphology of nanoadditives plays a crucial role in the enhancement of tribological properties. The SiO₂ nanoparticles (**Figure 6.1a–b**) are spherical and impersonate as nano-bearing between the tribo-pairs, which converted the sliding friction into a combined effect of sliding and rolling friction, consequently lowering the friction. The lubrication mechanism and the role of nanomaterials are governed by their shape, size, and composition. The recent findings have demonstrated that nanoslices and nanosheets of MoS₂ as additives to liquid paraffin and PAO oils, significantly improved the tribological properties and exhibited better tribo-performance than that of MoS₂ nanoballs and nanoflowers-based additives under identical tribo-conditions (Hu et al., 2010; Vattikuti and Byon, 2015). The MoS₂ and GO shows the sheet-like lamellar structure (**Figure 4.3a–b and Figure 4.6a–b**). The Mo atoms are interconnected between the two-dangling sulfur atoms in a trigonal prismatic sandwich-like model. The dangling sulfur atoms are susceptible to interact with metallic surfaces and gradually formed a protective thin film over the tribo-interfaces, driven by high Hertzian stress (Kumari et al., 2016). The weak van der Waals interaction between molecular lamellae of MoS₂ facilitated the shearing and decreased the friction between the tribo-pair. The presence of Mo, S, and Si signature (**Figure 6.7c**) confirmed their participation in thin-film formation, while steel balls were rubbed with a binary combination of MoS₂/SiO₂ blended coconut grease. Moreover, XPS analysis (**Figure 6.8**) corroborates the formation of protective film over rubbed surface lubricated with MoS₂/SiO₂ blended coconut grease. Likewise, the laminates of GO are also connected with each other. However, because of the hydrogen-bonding network made by ample oxygen functionalities in the basal plane, it compromises the low-shearing properties to some extent, yet it furnished good lubrication properties (Berman et al., 2015;

Mungse and Khatri, 2014). The low interlayer shear strength of two different types of lamellar materials under the boundary lubrication regime is believed to shear and develop a protective tribo-film over the worn surfaces through their tribo-induced adsorption/interaction (Fan et al., 2014). The XPS analysis (**Figure 6.8**) validates the adsorption of tribo-chemical thin film over rubbed surface lubricated with GO/SiO₂ blended coconut grease.

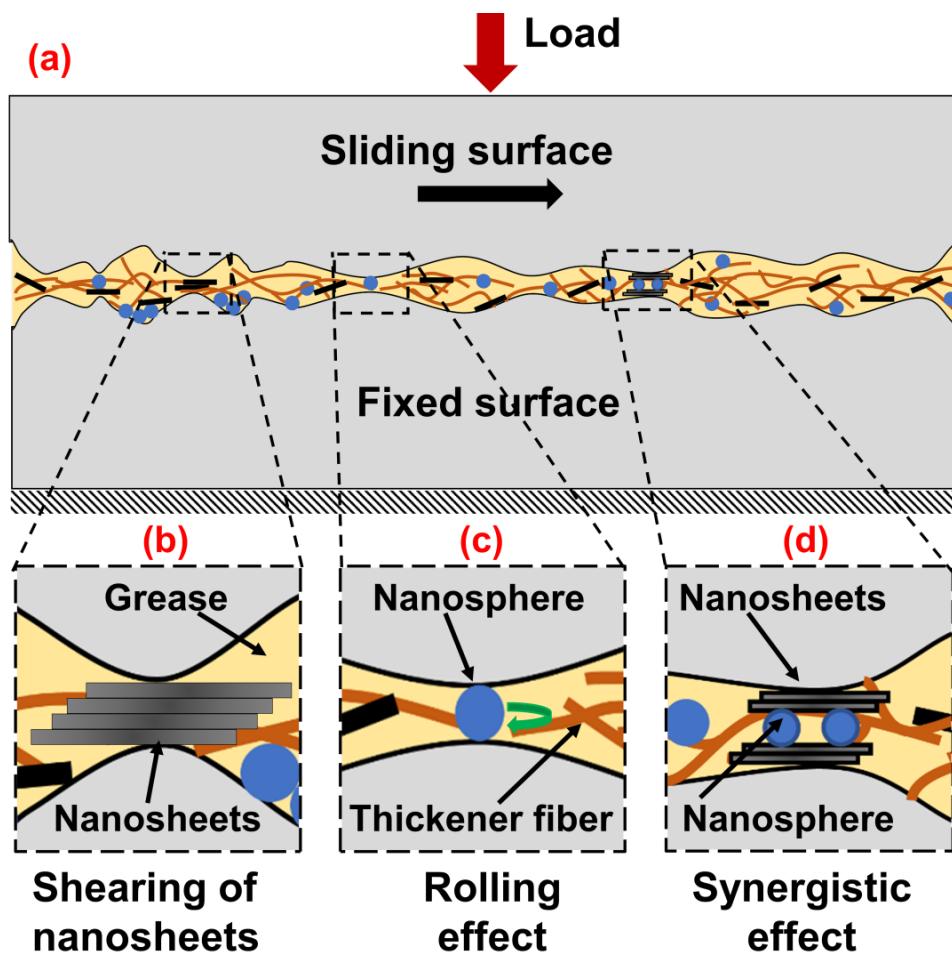


Figure 6.11: (a) Schematic representation of the plausible synergistic effect of nanoadditives in coconut grease. (b) Low-shearing property by nanosheets of MoS₂ and GO, (c) rolling effect by spherical SiO₂ nanoparticles, (d) synergistic effects by SiO₂ nanoparticles along with MoS₂ or GO nanosheets

The steel balls lubricated with only SiO₂ nanoparticles-blended coconut grease decreased the COF and MWV by ~10% and ~14% compared to coconut grease-lubricated steel balls.

The use of 2D lamellar sheets of MoS₂ and GO along with spherical SiO₂ nanoparticles led to significant improvement in tribological properties, far superior to individual materials as additives to coconut grease. The reductions in COF and MWV of steel balls by using optimized binary combinations of MoS₂/SiO₂, and GO/SiO₂ nanoadditives to coconut grease were noted to be almost double of their individual materials as additives to coconut grease. The different morphologies of the nanoadditives show a synergistic effect under the tribo-stress, which is believed to significantly improved the friction-reducing and AW properties. **Figure 6.11** presents schematic illustrations demonstrating the plausible effect of blended nanomaterials in the coconut grease for enhancement of tribological performance. The spherical SiO₂ nanoparticles between the sliding interfaces of steel balls are believed to transform the sliding motion into partially rolling motion (**Figure 6.11c**). The lamellar sheets of MoS₂ and GO extended the low-shearing properties because of their lamellar structure. The binary combinations of spherical SiO₂ nanoparticles and 2D layers of MoS₂ or GO as additives to grease, effectively furnished the significantly improved tribo-performance by extending the rolling friction of SiO₂ nanoparticles between the two separate sheets of either MoS₂ or GO as demonstrated in **Figure 6.11d**. The plausible synergistic effect, where both rolling and low shear strength effects together by MoS₂/SiO₂, and GO/SiO₂ combinations, led to superior tribo-performance. Furthermore, the ultralow thickness of MoS₂ and GO sheets and nano-sized SiO₂ particles easily accommodated between the asperities of the sliding pairs and heal the surfaces by mending effect (Gupta and Harsha, 2018b).

Each tribo-test was carried out for 60 min, and the top ball was slid with a relative velocity of 0.39 m/s over the stationary balls. During the test, the asperities were sheared, and new surface features were developed. The newly generated surfaces are chemically active to adsorb/react with atmospheric conditions. A thick oxide film (10 to 100 nm) is formed

instantaneously on the metallic surfaces under the contact (Stachowiak and Batchelor, 2013). The trapped water molecules in between the interlamellar spacing of nanosheets and the fibrous matrix of the grease led to catalyze the oxidation events of freshly worn steel surfaces. Therefore, the signature of oxygen peaks appeared on each worn surface. Further, the presence of nanoadditives constituents was confirmed by the EDS and XPS analyses (**Figure 6.7 and Figure 6.8a**), which signified the accumulation/deposition of nanoadditives over the worn surfaces. The combined outcome of base oil, thickener, and nanoadditives formed a tribo-film over the contact surfaces of steel balls and improved the lubrication properties.

The surface roughness parameters have a significant influence on friction and wear characteristics. The surfaces having negative skewness are excellent bearing surfaces and retain the lubricating oil. Herein, the SPM test result (**Table 6.2**) indicates that the worn steel balls lubricated with nanoadditives-blended greases exhibited negative skewness. Therefore, nanoadditives blended greases demonstrated excellent tribological characteristics compared to coconut grease. These results also suggest that nanoadditives not only protected the interacting surfaces but also improved surface textures. Zhu and Huang (2017) have investigated the effect of roughness parameters on tribological performance based on BAC and reported that under the lubricating condition, the average COF has a negative correlation with BAC. The highest S_k value has attained a large average COF. Herein, the surface lubricated with coconut grease demonstrated the highest S_k value (**Table 6.2**) compared to worn surfaces with nanoadditives-blended greases, and the tribological results (**Figure 6.5a**) corroborates the highest average COF for coconut grease. Further, high S_{vk} value indicated that the surface has excellent bearing capacity. The BAC of $\text{MoS}_2/\text{SiO}_2$ blended grease has the highest S_{vk} value (**Table 6.2**), revealing that it has superior oil retention characteristics. Therefore, its tribological performance was superior

among all the samples. The retained oil behaves as a reservoir and compensates for the insufficiency of the lube oil during lubrication and results in a sustainable tribo-film.

6.6 Summary of the chapter

Energy-efficient and environmentally-sustainable grease formulated from colloidal dispersion of three different nanomaterials in coconut oil and then demonstrated potential alternative to conventional grease. In this context, coconut oil as a lube base stock and lithium soap as a thickener were used for grease formulation. The spherical SiO_2 nanoparticles, MoS_2 , and GO lamellar sheets, prepared by sol-gel, hydrothermal reduction, and Hummers' method, respectively, were blended (0.05 wt%) in two different combinations (spherical nanoparticles and 2D sheets) in coconut oil. Their colloidal dispersion in coconut oil was used for the grease formulation. The presence of these nanomaterials increased the dropping point of coconut grease to 205 °C. The optimized blend of $\text{MoS}_2/\text{SiO}_2$ (30:70) and GO/SiO_2 (50:50) showed a significant reduction in COF, WSD, and MWV, driven by the synergistic effect of constituted nanomaterials. The spherical SiO_2 nanoparticles extended the low friction because of the micro ball-bearing effect, whereas the low shear strength of 2D lamellar sheets (MoS_2 and GO) decreased the friction under the tribo-stress. The synergistic combination of spherical nanoparticles and 2D sheets in an optimized ratio under the tribo-stress (**Figure 6.11d**) improved the tribological properties because of the combined effect of rolling friction and low shear strength. The $\text{MoS}_2/\text{SiO}_2$ (30:70), and GO/SiO_2 (50:50) blends in coconut grease reduced the energy consumption by ~20% and ~19%, respectively, making the engineering system energy efficient.