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Graphene based composite grease for elastohydrodynamic lubricated point contact

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Abstract. This paper present tribological and dynamic evaluation of reduced graphene oxide (rGO) sheets as an additive in lithium grease. Highly dispersion mixing method is used to mix rGO in commercial lithium grease to prepare composite grease. Tribological contact under investigation is established by ball-on-disc configuration. Friction, noise and vibration responses are recorded for the point contact lubricated with composite grease and base lithium grease in rolling and sliding-induced-rolling conditions. Relative speed of disc with the speed of ball is varied in order to get sliding-induced-rolling contact. Observations are performed at different nornal loads and fixed speed in elastohydrodynamic lubrication (EHL) regime. Results show existence of an optimum concentration (0.4% w/w) of rGO in commercial lithium grease. Friction, noise and vibration are recorded minimum for concentration of 0.4% w/w of rGO in commercial lithium grease. Reduction in friction coefficient is recorded up to 30% and 20% for rolling contact and sliding-induced-rolling contact respectively at optimum concentration of rGO in lithium grease. The lamellar structure of rGO in base grease controls the lubricity of concentrated point contact.

1. Introduction

Every phenomenon in the mechanical system consists of relative motion between two surfaces in contact, involves friction and wear. Break through efforts are made to diminish/optimize the friction and wear through the application of lubricants, proper material selection, design modifications, etc. [1]. Modification in mechanical components design helps in reduction of friction of rubbing surfaces, one of such important mechanical component is rolling element bearings. Research has been carried out on the application and influence of lubricants, additives, and lubrication process on tribo concentrated contacts [2]. The role of lubricants is important in minimizing the friction between the rubbing surfaces. On the basis of their physical state they are classified as solid, semi-solid, liquid and gaseous. Grease is a semi-solid lubricant with base oil, thickener and additives as constituents. Grease are characterized by 3- dimensional thickener fibrous structure dispersed in base oil [3]. The base oil bleeds out on the application of mechanical stresses and further reabsorbed on removal of stresses. This tends the greases to behave more or less like a sponge [4]. Depending on applications greases have many advantages such as: better in squeeze film lubrication, sealing to contact avoiding contaminants etc. In spite of these advantages greases traps additives ranging from nano to few microns in their 3-dimensional fibrous structure. Greases forms the thin film with additives, which separates the interacting surfaces. Research on carbon based nanoparticles are being developing at relentless pace to study the application in wide range of technologies- from electronics to nano-fluids/lubricants [5–20]. Nano grease based on Carbon based nanoparticles are under study for not only its improved tribological property but also for enhanced heat transfer capacity and thermal stability in thermal greases; which are being used as thermal Interface material (TIM) in electronics devices including LED [7,12–16]. Use of optimum concentration of alkali fluorides (CaF2) nano-particles as additives to lithium grease exhibited anti-wear, friction reduction and extreme pressure properties [21]. Apart from anti-wear, friction reduction and enhanced load carrying capacity nano-calcium borate (NCB) as additives also shows significant changes in dropping point, unworked penetration and roll stability of lithium grease [22]. Titanium complex grease based on nano-titanium dioxide and nano-silicon dioxide shows improved tribological properties, while load-carrying capacity shows no significant changes [23]. Some literature also shows study of carbon nano-particles based grease.

Graphene is a prominent member of the carbon based nano-particles family. Graphene have excellent mechanical, electrical, thermal and optical properties [6,8,9,17,18]. Besides these properties, nano-spaced layered structure of multi-layered graphene makes it easy tangential shearing. In recent years, nano-particles including graphene based lubricants, oil as well as greases, have received greater attention of researchers [21–29]. Fan et al. [10] showed Multilayer Graphene (MLG) as better additive to bentone grease as compared to ionic liquid or graphite. MLG showed significant improvement in lubrication and physical properties along with thermal stability of base bentone grease. However, these researchers have studied tribological performance of nanoparticles added greases but little literature is available regarding noise and vibration study.

The aim of this work is to explore simultaneous study of friction, noise and vibration of rGO added commercial lithium grease in rolling and sliding-induced-rolling conditions at fixed speed and different loads. The present work focusses on establishing some co-relation between friction, noise and vibration (if exists) of concentrated point contact supplied with nano rGO grease at various concentrations in lithium grease.

2. Materials and methods

2.1. Materials

A flattened chrome steel disc (AISI 52100) and super finished ball (AISI 52100) are used to establish tribological contact. The surface characterization of disc and ball on profilometer (Talysurf- by Taylor Hobson Ltd.) revealed surface roughness (R_a) of 0.01 µm and 0.02 µm respectively. Commercially purchased lithium grease is selected as base grease for the preparation of composite grease. The grease is mineral oil based with operating temperature range from -30^{0}C to 120^{0}C and dropping point above 180^{0}C. The additive rGO is provided by CSIR- Indian Institute of Petroleum, Dehradun.

2.2. Preparation of composite grease

Highly dispersion mixing method is followed for mixing rGO in lithium grease. Dispersion of rGO in toluene is carried by sonication for 1 hour. Sonication is performed for breakage of the agglomerates of rGO. The rGO-toluene dispersion is added drop-wise in hot lithium grease. Hot lithium grease is under heavy mechanical stirring and maintained at 110℃ to allow toluene to evaporate. Mechanical stirring is provided by magnetic stirrer for more than 45 minutes. The prepared hot mixture of rGO and lithium grease is allowed to cool up to room temperature under normal environmental conditions to obtain desired composite grease.

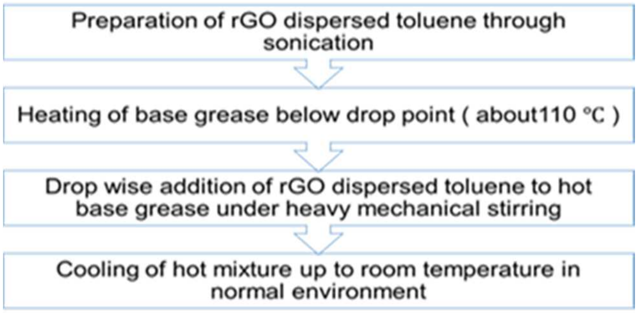

Figure 1. Method for the preparation of composite grease.

2.3. Characterization of rGO

Powder X- Ray diffractometer (Rigaku MiniFlex- 600, Japan) is used to identify the phase of rGO nanoparticles. The XRD powder diffraction pattern measurement is performed with Cu Kα radiation (λ = 1.5418 Å) at 40 kV and 15mA. The use of HRTEM to demonstrate micro fibrous structure of lithium base grease after the oil content was removed. Scanning electron microscopy reveals the layered structure of commercially obtained rGO, which is dispersed into toluene to get the nano-sheets of rGO. Dynamic light scattering (Particulate Systems NanoPlus from Micromatrics, USA) is used to characterize the dispersion in order to estimate the size distribution of rGO particles.

2.4. Tribological evaluation

Frictional performance of grease is evaluated with a fully automated EHD rig (PCS Instruments, London). Grease lubricated tribological contact is established with flattened disc on ball configuration (figure 2) in EHD rig. The experiments are performed at fixed ball speed 0.3 m/s and at different loads 10N, 20N, 30N, 40N and 45N (corresponding Hertz pressure are 0.6 GPa, 0.8 GPa, 0.9 GPa, 1.0 GPa and 1.2 GPa respectively) for rolling and sliding-induced-rolling conditions. The rolling and sliding-induced-rolling conditions are constrained of Slide-roll ratios (SRR) - 3% and 27% respectively. The parameter-SRR is the measure of sliding component in the rolling motion of ball-disc pair. Relative speed of the disc with ball is varied in response to get fixed SRR. For a particular SRR, the speed of ball remained fixed at input speed, while speed of disc varied according to the mathematical relation as SRR (%) = $\frac{\boldsymbol{u_1}-\boldsymbol{u_2}}{\frac{\boldsymbol{u_1}+\boldsymbol{u_2}}{2}}$ ×100, where, $\boldsymbol{u_1}$ = Speed of disc (mm/s) and $\boldsymbol{u_2}$ = Speed of ball (mm/s).

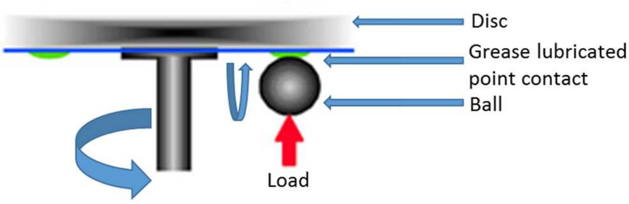

Figure 2. Schematic representation of ball-on-disc configuration in EHD rig.

2.5. Noise and vibration measurement

Noise and vibration of the contact is measured for dynamic evaluation of grease lubricated tribological contact. The experimental parameters were same as of tribological evaluation. The measurement required an external set-up to EHD rig. A sound level meter (CEL 500, from CASELLA CEL Ltd., UK) is used for the measurement of Sound Pressure Level (SPL) of the contact noise. The microphone of the sound level meter was placed near to disc-ball contact using a tripod. SPL was measured under 'A' weighting condition. A non-contact type condenser based transducer, positioned near to ball-disc contact is used to capture vibration signals. The transducer was connected to its amplifier (Accumeasure 9000, by MTI

Instruments Inc., USA) which passes signals to display and store in double channel FFT Analyser (by ONO SOKKI CO. Ltd., Japan).

3. Results

3.1. Reduced Graphene Oxide (rGO)
Figure 3a shows diffraction pattern obtained from XRD of rGO. The broad diffraction peak reveal a very small size of rGO particles. The JCPDS- Card no. 75-2078 indicates that phase of rGO is graphite. Figure 3b demonstrates the scanning electron microscopic image for the layered structure of rGO. Figure 4 shows the distribution of rGO into toluene. There are rGO clusters of different particle sizes which register difference in scattered light intensity. Further, figure 4 reveals that there are some nano-sized particle of rGO along with micron size clusters. The dispersion of these rGO into commercial lithium grease will results different composite greases.

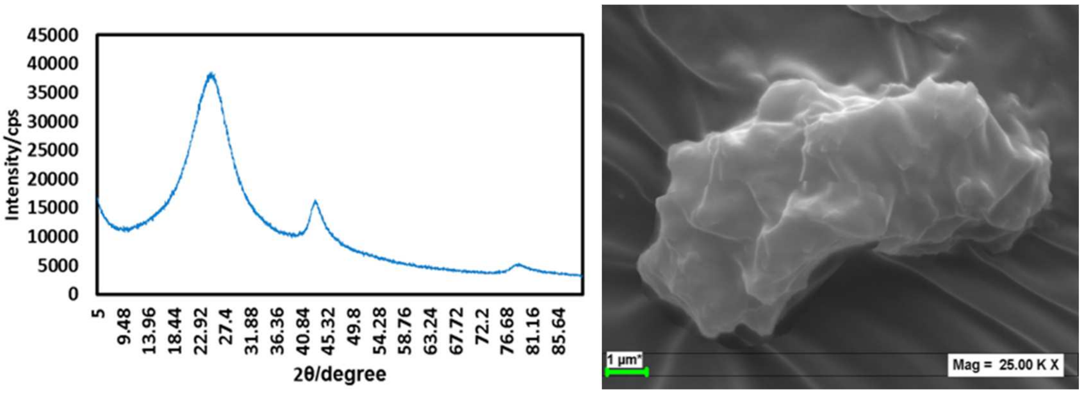

Figure 3. Characterization of rGO: (a) X-ray powder diffraction pattern and (b) SEM micrograph of rGO.

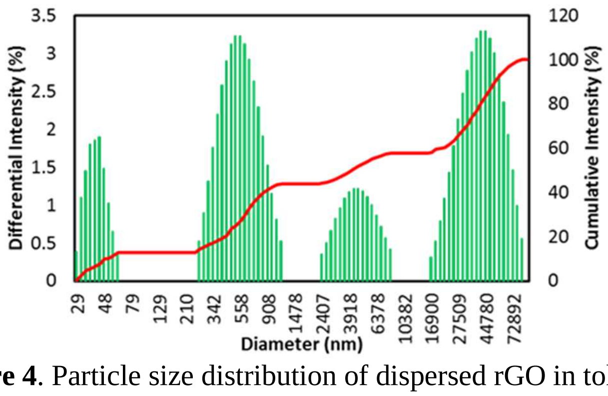

Figure 4. Particle size distribution of dispersed rGO in toluene.

3.2. Frictional response
Figure 5 gives frictional response of ball-disc tribological pair lubricated with varying concentration of rGO in base grease. A superficial look at figure 5a and 5b shows blending of rGO in base grease leads to significant improvement in frictional performance of base grease. Figure 5a shows frictional response for almost rolling contact (SRR 3%). Figure 5a shows level of friction is very low for the entire range of load. However, there is a general trend of increase in friction coefficient with load. However, when concentration of rGO increases beyond 0.40% (w/w) frictional performance of composite grease start degrading. It reveals that there exist an optimum concentration of 0.40% (w/w) of rGO in commercial lithium grease. While considering figure 5b, there is a significant increase in friction coefficient for the sliding-induced-rolling motion (SRR 27%) of disc-ball tribological pair. For both rolling conditions with respect to SRR

%, friction coefficient increases with load and composite greases show improved frictional performance as compared to base grease. Thus, friction level is minimum for optimum concentration 0.4 % (w/w) of rGO in base grease.

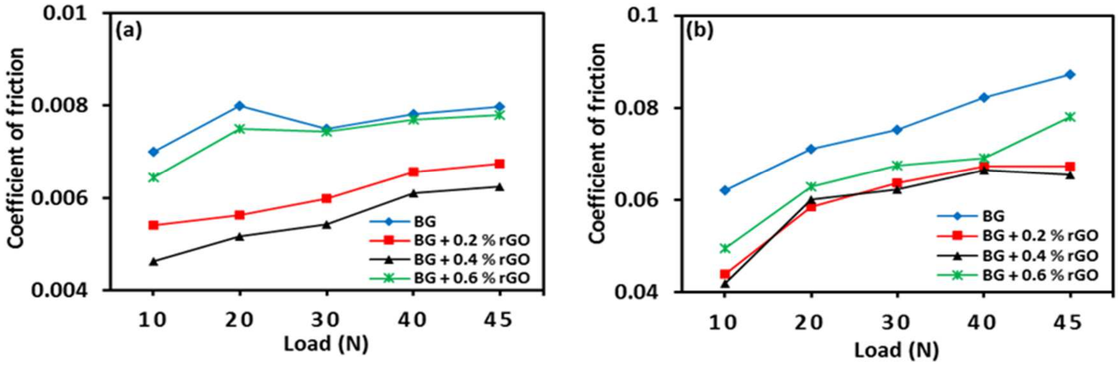

Figure 5. Variation of friction of coefficient with load for base grease and composite greases at different concentration of 0.20% rGO, 0.40% rGO and 0.60% rGO at different SRR of (a) 3% and (b) 27% respectively at fixed speed of 0.3 m/s.

3.3. Noise

The noise level is suppressed by the blending of rGO at different concentrations in commercial grease. Figures 6 and 7 shows the variation of noise level with load and concentration of rGO in grease at constant speed of 0.3 m/s during rolling contact (3% SRR) and rolling-sliding induced contact (27% SRR) respectively. The effect of loads (10N, 30N and 45N) is shown horizontally and effect of amount of rGO (0% w/w and 0.4% w/w) in base grease is shown vertically (figure 6 and 7). The variation of noise level with corresponding frequency are shown in spectra (figure 6 and 7) ranging from 20 Hz to 20,000 Hz.

The overall noise level (figure 8a), corresponding to fequency spectra (figure 6), shows the increase in noise level with increasing load. The minima in overall noise level is recorded when 0.4 % (w/w) of rGO is blended in commercial grease. Similarly, overall noise level (figure 8b) of frequency spectra at 27 % SRR (figure 7) also gives the minimun noise level for 0.4 % (w/w) of rGO in lithium grease. But the overall noise level is increased for 27 % SRR on comparing it with 3 % SRR (figure 8b).

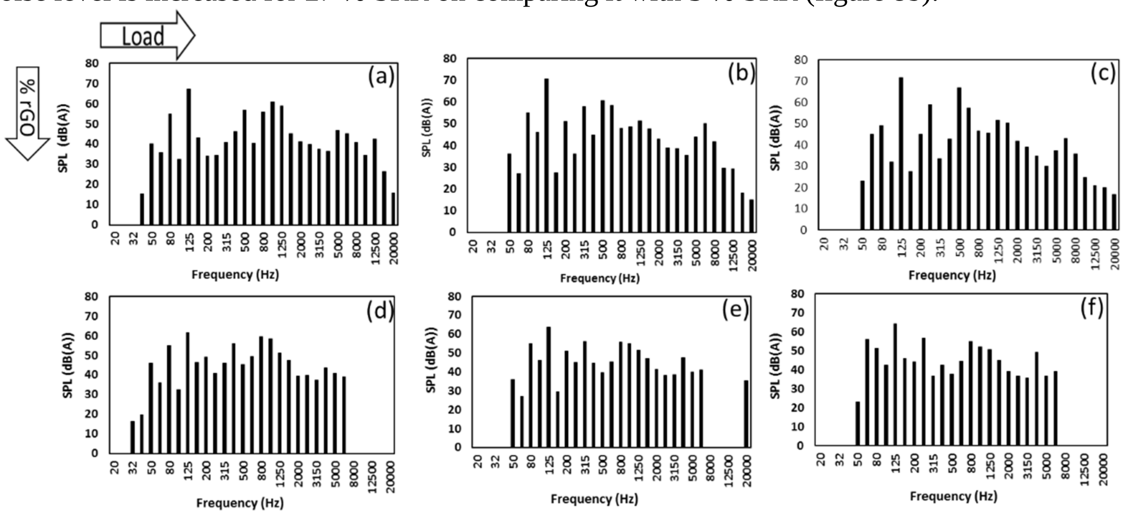

Figure 6. Variation of noise level with load and concentration of rGO at constant speed of 0.3 m/s during rolling contact (3% SRR). The effect of loads (10 N, 30 N and 45 N) is shown horizontally and effect of amount of rGO (0% w/w and 0.4% w/w) in base grease is shown vertically.

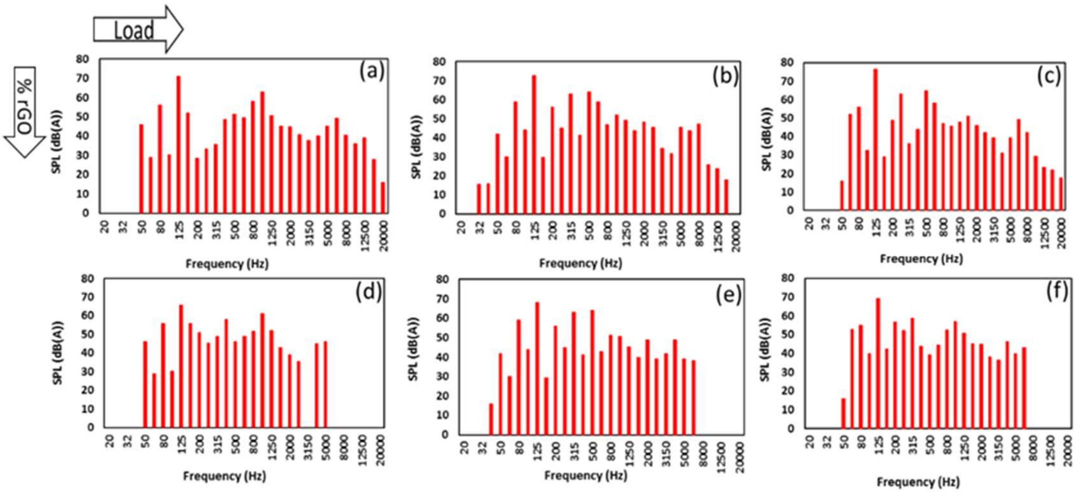

Figure 7. Variation of noise level with load and concentration of rGO at constant speed of 0.3 m/s during sliding-induced-rolling contact (27% SRR). The effect of loads (10 N, 30 N and 45 N) is shown horizontally and effect of amount of rGO (0% w/w and 0.4% w/w) in base grease is shown vertically.

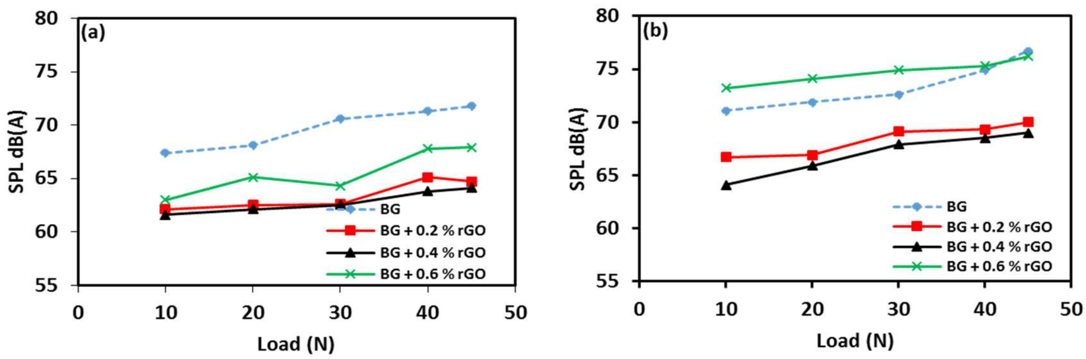

Figure 8. Variation of overall noise level in SPL dB(A) with load and SRR % for base lithium grease and composite greases with different percent concentrations (w/w) of rGO at constant speed of 0.3 m/s: (a) 3 % SRR (b) 27 % SRR.

3.4. Vibration

Figure 9 show vibration level at the contact lubricated with varying concentration of rGO in base grease at fixed speed and different loads. The vibrational response in rolling condition (SRR 3%) is shown in figure 9a. There is a general trend of increase in vibration level with load for a particular type of grease. Although the variation in magnitude is very low. Considering the response for composite greases, vibration level decreases with increase in concentration of rGO. Decrease in vibration level is subjected to an optimum concentration of rGO (0.40% w/w). Further increase in concentration level of rGO leads to significant rise in vibration. The vibration level for sliding-induced-rolling condition is shown in figure 9b. The trend for vibration level is increasing with load and decreasing with increase in concentration of rGO in composite greases. The reduction in vibration level is subjected to an optimum concentration of 0.4 % (w/w) of rGO in greases as like in rolling condition (3 % SRR).

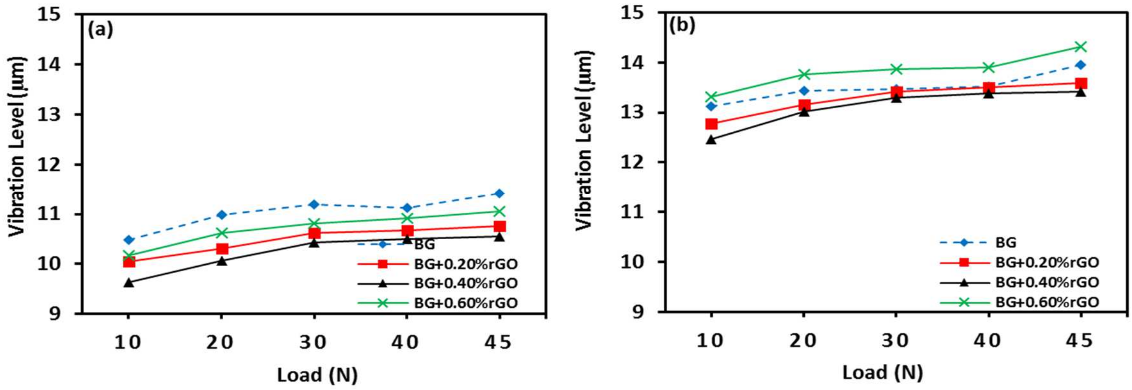

Figure 9. Variation of vibration level with load for base grease and composite greases at different concentration of 0.20% rGO, 0.40% rGO and 0.60% rGO at different SRR of (a) 3% and (b) 27% respectively at fixed speed of 0.3 m/s.

4. Discussion

Considering the condition monitoring, reduction in friction usually causes improvement in life of concentrated contact. Composite greases find wide application in reducing the frictional losses of concentrated contacts resulting in reduced noise and vibration. Thus, due to enhanced properties of composite greases compared to conventional grease, tends to improve the operating life of concentrated contacts in wide variety of machine and mechanisms. The increase in friction due to induced sliding may be refer to increase in rubbing at contact. The general trend of increase in friction coefficient with load may be attributed to the argument that the tribo-pair is working under mixed lubrication regime. The improved frictional behavior of composite grease may be attributed to extremely thin layered structure of rGO which form a protective film to separate the steel surfaces [10,30]. The layered structure of graphene offer very low shear resistance [30] which leads to low friction between pair. The parameters friction, noise and vibrations are seen to be inter related in the present study. With the rise in friction coefficient of tribo pair with variation in load, noise and vibration level also increases. But optimality in concentration of rGO (0.4% w/w) in commercial grease reduces these parameters to the minimum.

5. Conclusion

The rGO is able to modify the lubricating performance of commercial lithium grease. The improvement in tribological and dynamic characteristics of contact lubricated with composite grease, is subjected to an optimum concentration of rGO in lithium grease. The optimum concentration of rGO is 0.4% (w/w) in base lithium grease for both rolling point contact (3% SRR) and sliding-induced-rolling contact (27% SRR). At optimum concentration the lubricated contact is characterized with minimum friction, noise and vibration. Friction coefficient decreased up to 30% and 20% for rolling and sliding-induced-rolling contact for optimum concentration of rGO as compared to base lithium grease. The composite grease provide a boundary film of lubricant between rubbing contact. The improved performance of composite grease may be attributed to layered structure of rGO. The layers of rGO facilitates easy shearing between contacts. The increase in SRR leads to increase in friction, noise and vibration of tribological point contact.

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