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Enhancing the tribological characteristics of Jatropha oil using graphene nanoflakes

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KEYWORDS

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Graphene
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ABSTRACT

Graphene is one of the contemporary nano-additives employed to boost the tribological characteristics of the lubricants. It has remarkable lubricating capabilities due to its peculiar properties and structure. This paper investigates the influence of graphene nanoflakes on the tribological properties of Jatropha oil (JO). Ball-on-flat tribological tests were conducted on a steel-steel tribo-pair with different concentrations (by wt.) of graphene mixed in the lubricant and the deviations with load and speed were noted. The wear scars were analyzed on Scanning Electron Microscope, Optical Microscope, and 3D Profilometer. The results indicated that the addition of graphene to JO substantially reduced the friction and wear rate between the tribo-pair. The best tribological performance was found at 0.5% graphene which reduced the coefficient of friction by 44% and wear rate by 43.7% as compared to base JO. This outcome was attributed to the formation of tribo-layer on the surface as characterized by Raman analysis which detected the presence of graphene nanoflakes in the wear scar. Finally, it was concluded that JO added with 0.5% graphene can be a promising lubricant option for industries in terms of energy-saving and environmental concerns. The graphene doped JO can be a viable substitute for depleting petroleum resources.

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NOMENCLATURE

JO	Jatropha Oil
Gr	Graphene
Cof	Coefficient of friction
SEM	Scanning Electron Microscopy
XRD	X-Ray Diffraction
rpm	Revolutions per minute

1.0 INTRODUCTION

The prime concerns of the world today are energy conservation and environment protection (Chan et al., 2018). The energy demands in the world are escalating more than ever due to the rapid exhaustion of fossil fuels. The energy scarcity could result in a spike in the oil prices and environmental issues. This has motivated the researchers in this field to explore the surrogate options of sources of energy which are inexhaustible (Singh et al., 2017a). Tribology has a very significant correlation with sustainability. It can increasingly contribute to energy savings, limiting pollution, and reducing friction and wear losses by developing environmentally friendly lubricants (Tzanakis et al., 2012). One of the most significant consumables in the industrial sector is lubricants. They can save energy, reduce operating costs, and increase the life of machine parts (Woma et al., 2019). A lubrication system is mandatory to restrict friction and wear, avoid the corrosion and to take the heat away (Shafi and Charoo, 2020c). Very low coefficient of friction (below 0.1) can be observed with liquid lubrication (Najar et al., 2017b) and nowadays some advance techniques are being used in the field of tribology and surface engineering in order to reduce the friction and wear to the minimum possible level (Najar et al., 2017a; Najar et al., 2019). Most of the lubricants in the market have been manufactured from petroleum-based oils. They have excellent lubricating abilities and are easily available (Yunus et al., 2020; Talib et al., 2019). However, the petroleum oil-based lubricants are toxic, non-renewable, and non-biodegradable. Bio-oils are a better option to replace mineral oils in the future (Zainal et al., 2018; Salleh et al., 2019). In view of the above reasons, the vegetable oil-based biolubricants are set be in enormous demand in the coming years. The percentage of biolubricants is expected to reach 15% to 30% in the next 15–20 years and this is going to be a very appealing field in the lubrication manufacturing industry (Panchal et al., 2017). Biolubricants are renewable, biodegradable, non-toxic, and environmentally friendly which makes them superior to mineral oils (Rao et al., 2018; Shafi and Charoo, 2020b). Because of their good properties such as high flash point, high viscosity, and viscosity index, they are desirable and offer great potential in the field of lubrication. However, they possess poor oxidation stability, high pour point, and low thermal stability which restricts their use. Suitable additives like antioxidants, anti-friction, and anti-wear need to be used to ameliorate their lubricating properties. Chemical modification also provides a good option. This can result in reducing frictional losses and saving of energy which in turn restricts the number of gases released in the atmosphere and reduces pollution (Syahir et al., 2017; McNutt and Sophia, 2016). Nanoparticles are one of the promising additives that can be used. They have a considerable effect on the tribological properties of vegetable oil-based lubricants (Darminesh et al., 2017). Nanoparticles have been widely used in improving the tribological properties of lubricants. The nanoparticles undergo physical interaction with the surrounding environment which greatly affects the frictional behavior, while as the anti-wear action mostly depends on the chemical interaction between the nanoparticles and the environment (Dai et al., 2016). The nanoparticles get placed in between the layers of the lubricant which increases its endurance (Shafi and Charoo, 2020a). Once the deficient properties of biolubricants are improved, they can be beneficial in almost every industrial and automotive application (Soni and Agarwal, 2014). Jatropha oil (JO) can be a good lubricating option and can be used to produce a biolubricant. It can be considered as a good competitor in the automobile industry as a lubricant (Menkiti et al., 2017; Zulkifli et al., 2014). The lubricating performance of JO in machining was enhanced by the addition of hexagonal boron nitride particles (Talib and Rahim, 2018). The base JO was mixed with graphite nanoparticles which drastically enhanced its tribological properties and reduced the wear rate (Begum et al., 2019). JO was blended with commercial engine oil by up to 50% and the testing was done on a pin-on-disc tribometer. The results revealed that the addition of JO to the engine oil reduced the friction and wear scar diameter and increased the mechanical efficiency (Singh et al., 2017b). Micro-sized MoS_2 particles were added to JO in different concentrations to improve its tribological performance. It was found that friction and wear got reduced, and the best results were obtained at 2% MoS_2 addition (Mushtaq and Hanief, 2021). Further it was reported that the MoS_2 nanoparticles considerably improved the lubricating capabilities of Jatropha oil (Hanief and Mushtaq, 2020). Graphene is the first existing 2D material discovered in the early 21st century. Graphene nanoparticles have been used as lubricant additives and have proved to be quite efficient in friction and wear minimization (Kumar and Wani, 2017). It is very impressive in mitigating the friction and wear as a solid lubricant, as a lubricant additive, and as an additive to composites and solvents (Berman et al., 2014). Graphene nanoparticles were added in different concentrations to the blend of jojoba oil and engine oil. The friction and wear were evaluated on a pin-on-disc tribometer. The graphene nanoparticles proved very effective in the reduction of both friction and wear (Thirumalai and Ramesh, 2017). The graphene nanoplatelets were added to neem oil and the testing was done on a 4-ball tester which resulted in decreasing Cof and wear scar diameter (Suresha et al., 2020).

In light of the above cited literature, it is quite clear that several additives have received great attention as a means of improving the lubrication characteristics. But there are many additives that have not been studied in mixed form with biolubricants. This study is an attempt to investigate the tribological performance of jatropha oil by adding graphene nanoflakes in three different concentrations. The experiments were conducted on a ball-on-flat geometry while the tribo-pair was being lubricated with these mixtures. The optimum concentration of graphene for the best tribological result was determined and the impact of variations in load and speed on Coefficient of friction (Cof) and wear rate were also investigated.

2.0 MATERIALS AND METHODS

2.1 Preparation of Tribo-Pair and Nanolubricant

Jatropha oil (extracted from the seeds) was obtained from a reliable source in India and used as the base oil. The fatty acid combination and physicochemical properties of JO as provided by the supplier are given in Table 1 and Table 2 respectively. The Steel of EN-31 grade and chromium steel balls of 52100 grade (12.7 mm diameter) were used as a friction-pair in this study and their physical properties are presented in Table 3. The Steel slab was prepared by polishing its surface with different grit sizes of silicon carbide emery papers from 280 to 2000. Then the surface was made smooth by rubbing it on a very smooth velvet cloth. Graphene nanoflakes of thickness 5-10 nano-meters (nm) and length 5-10 micro-meters (μm) were acquired from NanoShel USA. The nanoflakes were characterized by Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD) analysis as shown in Figure 1. In the XRD analysis of Graphene, only one characteristic peak was observed at 2θ=26.4 degrees as shown in Figure 1. Rest of the peaks are very miniscule and can be ignored. This depicts the good quality of graphene nanoflakes with negligible contamination. The base oil was weighed on a highly accurate digital balance and stored in test tubes. The nanoflakes were added to the oil by weight percentages of 0.3%, 0.5%, and 0.7%. All the mixtures were ultrasonicated for at least 6 hours before going for testing to ensure uniform mixing and eluding any agglomeration of the nanoflakes.

2.2 Friction and Wear Tests

The tribological tests were executed on a ball-on-flat reciprocating tribometer as shown in Figure 2. The tribo-pairs were EN-31 steel and 52100 chromium steel balls (12.7mm diameter). The operating parameters are fed to the machine through the software. The speed is adjusted in terms of Revolutions per minute (Rpm) of the motor which is converted into reciprocating motion by the machine. Initially, the friction tests were conducted while lubricating the tribo-pair with pure JO, and JO containing different concentrations of graphene. All other parameters were kept constant and the optimum concentration of graphene in JO for best tribological performance was established. Then the tests were conducted at different loads (50N, 100N, 150N, and 200N) and different speeds (1000 rpm, 1100 rpm, 1200 rpm, and 1300 rpm). The tribo-pairs were lubricated with the same mixture containing the optimum percentage of graphene, to ascertain the dependence of Coefficient of friction (Cof) and wear rate on load and speed. The time for each test was 10 minutes and the stroke of the reciprocating motion was 2 mm. The Cof values were recorded by the computer connected to the tribometer. The wear scars on the steel slab were examined under the lens of 3D profilometer (shown in Figure 3) to calculate the wear volume (mm^3) for each scar and the wear rate (mm^3/Nm) was given by equation 1. The 3D images and texture graphs of the wear scars were also retrieved from the 3D profilometer. For each scar, the area at its maximum depth was selected to get the 3D image and texture graph as it undergoes maximum wear and is our main area of focus.

$$\text{Wear Rate} = \text{Wear Volume} / (\text{Sliding distance} \times \text{Load}) \quad (1)$$

Table 1: Fatty acid combination of jatropha oil.

S. No.	Fatty acids	Composition of Jatropha oil (%)
1.	Oleic acid C18:1	40 - 45
2.	Linoleic acid C18:2	30 - 34
3.	Palmitic acid C16:0	15 - 16
4.	Stearic acid C18:0	8 - 10
5.	Myristic acid C14:0	1 - 1.8
6.	Arachidic acid C20:0	0.3 - 1

Table 2: Physicochemical properties of jatropha oil.

S. No.	Property	Jatropha oil
1.	Kinematic Viscosity at 40°C	47 - 54 cSt
2.	Kinematic Viscosity at 100°C	9 -10 cSt
3.	Viscosity Index (VI)	180 - 186
4.	Flash point	186°C
5.	Pour point	8°C
6.	Density (×10³kg/m³)	0.918

Table 3: Physical properties of the materials.

S. No	Physical Properties	EN-31 Steel	52100 Cr steel ball
1.	Hardness (HRC)	63	63 - 66
2.	Elastic Modulus (GPa)	210 - 215	190 - 210
3.	Density (g/cm³)	7.80	7.81
4.	Poisson's Ratio	0.28	0.29
5.	Melting Point (°C)	1540	1424
6.	Tensile Strength (MPa)	750	2240
7.	Yield Strength (MPa)	450	2030

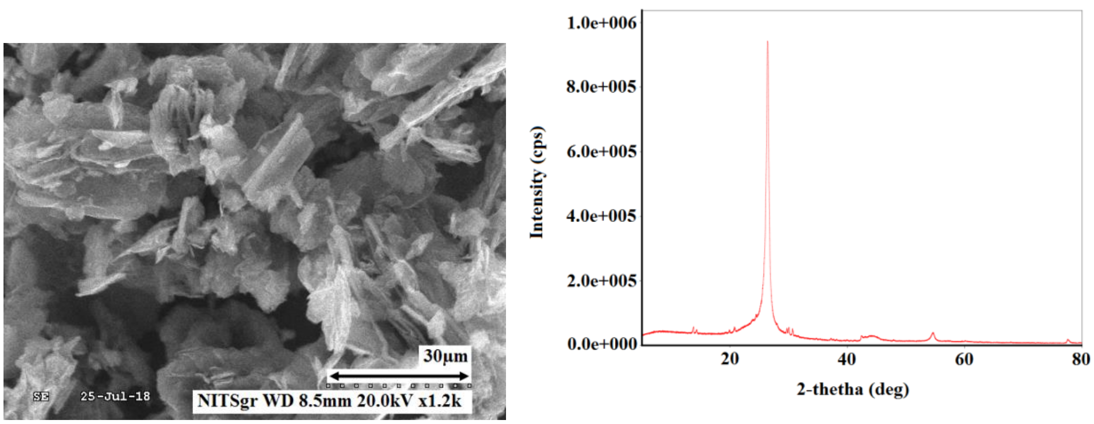

Figure 1: SEM image and XRD analysis of graphene nanoflakes.

Figure 2: Reciprocating tribometer.

Figure 3: 3D profilometer.

3.0 RESULTS AND DISCUSSION

3.1 Variation of Cof and Wear Rate with Graphene Concentration

Figure 4 shows the variation of Cof (with time) and wear rate with the concentration of graphene in JO at a constant load of 50N and 1000 rpm. The statistical data at different Graphene (Gr) concentrations in JO is presented in Table 4. It can be observed that the Cof with pure JO as a lubricant is high and decreases after the addition of graphene nanoflakes. The minimum Cof was recorded at 0.5% of graphene addition as given in Table 4. Hence, with the addition of graphene nanoflakes to JO, the tribological performance was observed to improve. The Cof was reduced by 43%, 44%, and 29% at 0.3%, 0.5%, and 0.7% concentrations respectively. The wear rate was observed on the higher side with pure JO as a lubricant. However, as the addition of graphene nanoflakes was initiated, the wear rate started dropping. The wear rate decreased by 40.5%, 43.7%, and 38.4%, at the concentrations of 0.3%, 0.5%, and 0.7% respectively. This decrement in the Cof and wear rate can be associated to the deposition of graphene nanoflakes on the steel surface. The nanoflakes react with the oil and environment to form a protective layer between the sliding surfaces thereby evading the metal-to-metal contact (Singh et al., 2018). However, at 0.7% concentration of graphene, both the Cof and the wear rate increased marginally as compared to 0.3% and 0.5% concentrations. This result can be explained due to the presence of an exorbitant quantity of nanoflakes which results in their agglomeration and creates a polishing effect. This increases the Cof and promotes the abrasive wear of the sliding surface (Wang et al., 2018). But the Cof and wear rate at 0.7% were still low as compared to the values received at base oil lubrication. Generally, it can be observed that after adding graphene nanoflakes to JO, its lubricating capabilities were considerably enriched. Friction as well as wear got reduced as compared to base oil lubrication at constant parameters. Both the minimum Cof (0.04514) and minimum wear rate (6.9×10^{-6} mm^3/Nm) were recorded as 0.5% of graphene addition and it can be considered as the optimum concentration for best tribological results. Hence, from the results obtained, it can be anticipated that the Graphene nanoflakes offer a great potential as lubricant additives to reduce Cof and wear rate between the tribo-pair. This blend can be very significant for the lubricant industry in the future to save energy and increase the life of the machine components.

Table 4: The statistical data at different graphene (Gr) concentrations in JO.

	Avg. Cof	**SD**	**Median**	**Maximum**	**Minimum**
Pure JO	0.08063	0.01579	0.07966	0.13854	0.05298
0.3% Gr	0.04603	0.01278	0.04385	0.10921	0.02626
0.5% Gr	0.04514	0.01240	0.04273	0.10113	0.02472
0.7% Gr	0.05745	0.00956	0.05659	0.09040	0.04002

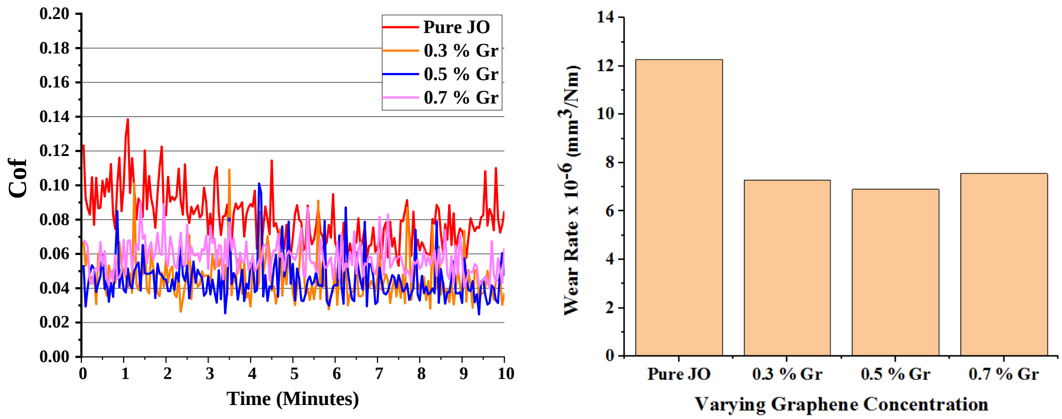

Figure 4: Variation of Cof and wear rate with graphene (Gr) concentration.

3.2 Variation of Cof and Wear Rate with Load

Figure 5 displays Avg. Cof and wear rate when the load was varied from 50N to 200N while keeping the other parameters constant. It can be observed that the Cof generally decreases with the increment in load. The Avg. Cof values were reduced by 8.3%, 21.8%, and 11.4% at 100N, 150N, and 200N respectively as compared to the value at 50N. This behavior can be explained by the phenomenon of excellent distribution of the nanoflakes on the surface and their rapid reaction with the oil and environment due to high heat generation at higher loads. This action accelerates the formation of the safeguarding tribo-layer (Xie et al., 2016). The layer formed protects the contacting surfaces and limits the friction. However, at 200N, a marginal increment was recorded in Cof which might be due to the possibility of a partial breakdown of the tribo-film due to very heavy load. The film gets thin and concedes more metal-to-metal intercourse. Nevertheless, the Cof received was well within the limits and still below the Cof received at 50N. The minimum (0.03531) and maximum (0.04514) Avg. Cof was recorded at 150N and 50N respectively. From Figure 5, it is clear that the wear rate increased with load. It increased by 12%, 27%, and 61.5% at 100N, 150N, and 200N respectively as compared to wear rate at 50N. The minimum (6.9×10^{-6} mm^3/Nm) and maximum (11.14×10^{-6} mm^3/Nm) wear rate was recorded at 50N and 200N respectively. The spike in wear rate with increments in load can again be attributed to the partial breakdown of tribo film at higher loads allowing more asperity contacts as explained above. From the above data, we can deduce that the Cof was found indirectly proportional and the wear rate directly proportional to load. These results indicate that the mixture of Jatropha oil and Graphene nanoflakes can be used to reduce Cof at higher loads. However, the wear rate increases and has to be checked at the higher loads. The minimum Cof and wear rate can be obtained at medium load which is of optimum value. Hence, we can conclude that this blend can be used to lubricate the tribo-pairs under moderate loads for best tribological performance.

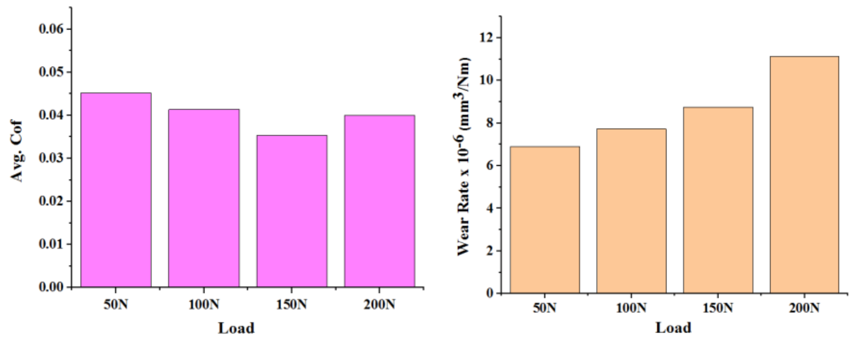

Figure 5: Variation of Cof and wear rate with load.

3.3 Variation of Cof and Wear Rate with Speed

Figure 6 compares the Avg. Cof and wear rate when the load was kept constant at 50N and the speed was changed from 1000 to 1300 rpm. The Cof decreased continuously with the increasing speed and the minimum Cof (0.02919) was recorded at the speed of 1300 rpm. The Avg. Cof reduced by 22%, 27%, and 35.3% at 1100 rpm, 1200 rpm, and 1300 rpm respectively. The wear rate continuously declined with the gain in speed and the minimum wear rate (4.3×10^{-6} mm^3/Nm) was recorded at 1300 rpm as well. The wear rate reduced by 13.7%, 26.3% and 37.7% at 1100 rpm, 1200 rpm, and 1300 rpm respectively. Hence, we can say that both the Cof and wear rate were found to be inversely proportional to speed. This result can be imputed to the fact that at lower speeds the thickness of the lubricating oil film is lower which allows more intercourse between asperities and high friction and wear values are received. As the speed is increased, the film gets slightly thickened which restricts the asperity contacts and reduces the Cof and wear rate (Shafi *et al.,* 2018). These results show that the blend of Jatropha oil and Graphene nanoflakes can be used to reduce the Cof and wear rate between the tribo-pair. Also, it is revealed that the higher the speeds, the lower are the Cof and wear rate. Hence this blend can be used as a lubricant at higher speeds to get the better tribological performance.

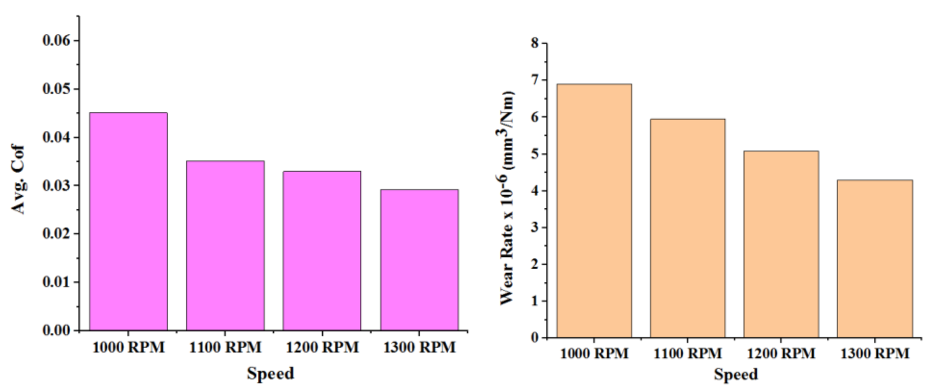

Figure 6: Variation of Cof and wear rate with speed.

3.4 Surface Analysis of Wear Scars

Figure 7 corresponds to the Scanning Electron Microscopy (SEM), Optical Microscopy, and 3D profilometer images of the wear scar at 50N load, 1000 rpm speed, and lubricated with pure JO. As seen from Figure 7a (SEM image), a lot of furrows, and some ploughing marks are visible on the surface depicting abrasive wear. Also, some small pits can be visualized where metal is removed, and they represent adhesive wear. Some deep furrows can also be seen from the optical image in Figure 7b. It depicts that the wear mechanism here is a combination of adhesive and abrasive wear as both types of wears are visible on the surface. Figure 7c shows the 3D image of the wear scar from the 3D profilometer and the bumps on its surface can be easily seen. Figure 7d shows the texture pattern of the wear scar and several small bumps can be seen in the image, one of which is very deep representing a large amount of wear. Figure 8 displays the SEM, Optical, and 3D profilometer images of wear scar which was lubricated with JO + 0.5% Graphene mixture. From Figure 8a, we can deduce that the SEM image is displaying a few shallow grooves. The ploughing marks and adhesion pits are almost absent. Figure 8b from the optical microscope shows one deep groove and few tiny scratches with no adhesive marks. From these images, we can draw that the wear severity has certainly reduced and in this case, abrasive wear is mostly the predominant mechanism. The 3D image and the texture graph of this scar are presented in Figures 8c & 8d respectively. As seen from the images, the bumps received are very small and the texture pattern is fairly smooth as compared to Figure 7, where pure JO was used as a lubricant. Hence, these images vividly indicate a change in wear mechanism and also improvement in the antiwear capabilities of JO by adding 0.5% of graphene nanoflakes. As explained in section 3.1, this improvement can be credited to the ability of graphene nanoflakes to react with the oil and the environment and form a defensive tribo-layer on the steel surface. This layer prevents the surface contact between tribo-pair reducing both friction and wear.

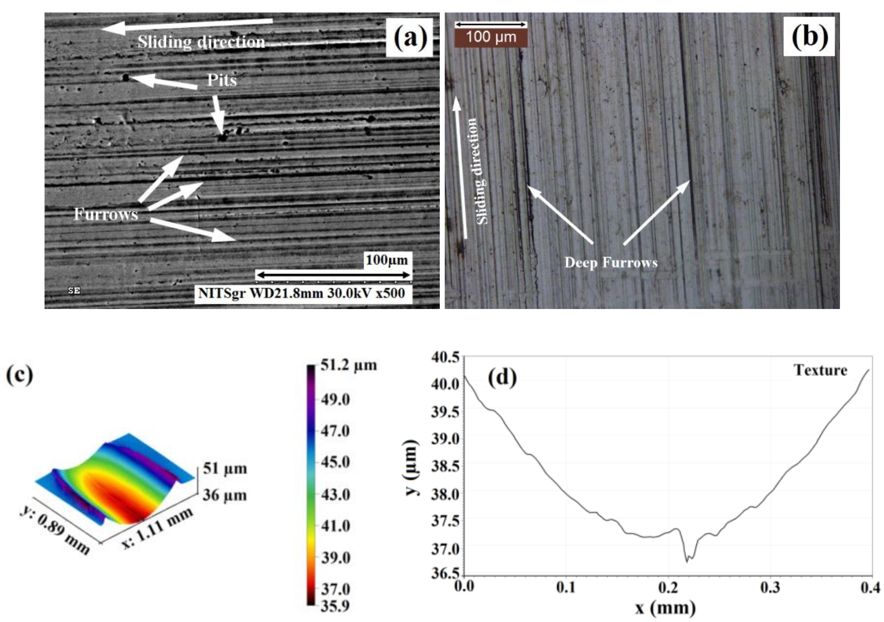

Figure 7: Surface topography of wear scar at 50N load and 1000 rpm lubricated with pure JO.

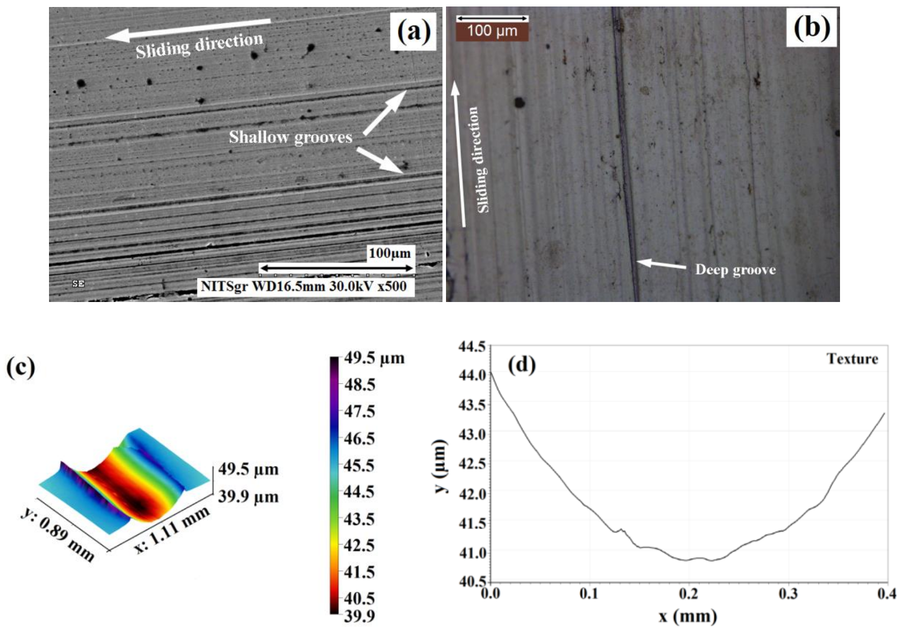

Figure 8: Surface analysis of wear scar at 50N load and 1000 rpm lubricated with JO added with 0.5% graphene.

The surface analysis of wear scar with 200N load is shown in Figure 9. As seen from the SEM image (Figure 9a), the surface is very rough as compared to the surface at 50N shown in Figure 8. Several deep furrows can be visualized which indicates the severe abrasive ploughing during sliding. Also, from the optical image (Figure 9b), some deep grooves and marks can be seen. There are also black spots in Figure 9b that reflect the smearing of graphene nanoflakes inside and around the deep furrows due to high load. The 3D image and texture pattern (Figures 9c & 9d) also indicate that there are more bumps and deep asperities recorded at 200N load. Hence, we can say that by increasing the load to 200N, the wear has increased as compared to 50N. This deterioration in wear can be due to the thinning of the tribo-film at higher loads allowing more asperity contacts.

The respective SEM, Optical, and 3D profilometer images for JO + 0.5% Graphene at 50N and 1300 rpm are shown in Figure 10(a to d). Figures 10a and 10b confirm that the smoothest surface is received in this sliding condition. Apart from a few ploughing marks, the surface looks perfectly smooth with minimum wear received. This is following Figure 6 where the minimum wear rate was received at 1300 rpm. The 3D image and texture graph also confirms the smoothness of the surface of this scar giving a clear image and almost straight line with minimum bumps or asperities. Hence, we can conclude that the analysis of the surface of wear scars has yielded harmonious results to that of wear rate data recorded above and both are agreeing with each other. The smoothest surface with minimum damage was received at the sliding condition of 50N and 1300 rpm. It establishes the fact that the low load and high-speed condition is favorable for minimum wear between the steel-steel tribo-pair lubricated with JO + 0.5% G mixture. It may be helpful to the industries where the machinery is operating under the same conditions. They can utilize this mixture to obtain the best tribological performance and impart longevity to the machines.

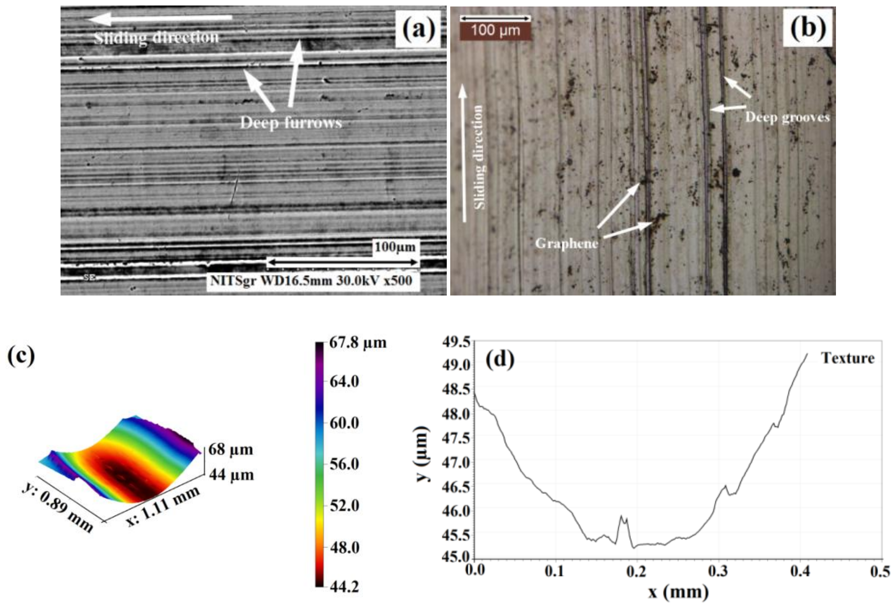

Figure 9: Surface topography of wear scar at 200N load and 1000 rpm lubricated with JO added with 0.5% graphene.

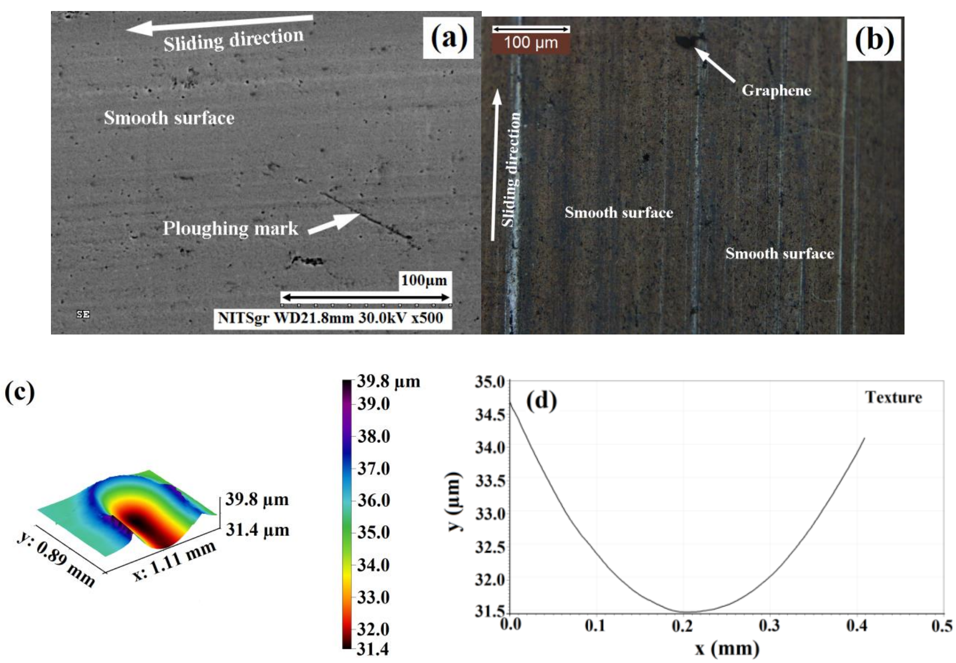

Figure 10: Surface topography of wear scar at 50N load and 1300 rpm lubricated with JO added with 0.5% graphene.

3.5 Raman Spectroscopy

Raman spectroscopic analysis was carried out on the wear scar surfaces. The results confirmed the presence of graphene nanoflakes on the wear scar imperative to form the protective tribo-layer on the surface. Figure 11(a) plots the Raman spectra obtained for pure graphene nanoflakes where D and G bands are formed at 1355 cm^{-1} and 1589 cm^{-1} respectively. Fig 11(b) shows the Raman spectra of the wear scar lubricated with JO + 0.5% Graphene. The D and G bands can be observed at 1325 cm^{-1} and 1588 cm^{-1} respectively which confirm the formation of tribo oxide-layer on the scar zone.

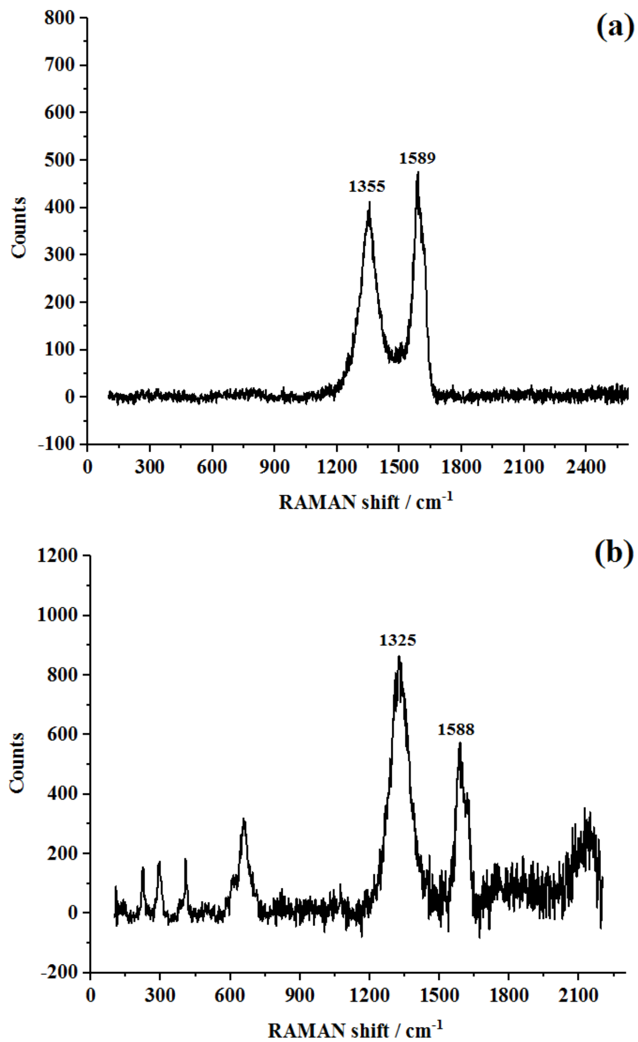

Figure 11: Raman spectrum of (a) graphene nanoflakes (b) wear scar on the steel surface.

4.0 CONCLUSIONS

The friction and wear behavior of Jatropha oil (JO) as a biolubricant was assessed and modified by the addition of graphene nanoflakes. The following conclusions can be drawn from this experimental analysis.

(a) Addition of graphene nanoflakes to the JO substantially improved its lubricating potentials by reducing the Coefficient of friction (Cof) and wear rate. It was attributed to the formation of tribo-layer on the surface which impedes the metal-to-metal contact as characterized by Raman analysis.

(b) The optimum concentration of graphene in JO for the prime tribological performance was recorded at 0.5% addition. By increasing the concentration to 0.7%, the lubricating performance started to aggravate mildly due to the presence of its exorbitant amount giving rise to a polishing effect on the surface and increasing the friction and wear.

(c) The Cof reduced, while as the wear rate increased with increasing load. However, with the increments in speed provided, both the Cof and wear rate lowered.

(d) The blend of JO and 0.5% graphene nanoflakes can primarily be advantageous in sliding tribological conditions, especially involving higher speeds and moderate loads.

Hence, overall, it can be concluded that JO has splendid tribological properties which can be further encouraged by the addition of graphene nanoflakes. This blend can be a good substitute to fill the shoes of mineral oils in the worldwide lubricating industry in the future, be it the industrial or the automobile industry. It can alleviate the dependency on petroleum-based oils, help in controlling environmental pollution, and save energy

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