Tribologically Optimized Novel Lubricant with CuO Nanotubes and CuO Nanopowders as Additives

Nithin B R\textsuperscript{1} & Deepak B S\textsuperscript{2}

\textsuperscript{1}PG Student, Mechanical Engineering Department, Mar Baselios College of Engineering and Technology, Trivandrum, Kerala, India.
\textsuperscript{2}Assistant Professor, Mechanical Engineering Department, Mar Baselios College of Engineering and Technology, Trivandrum, Kerala, India.

Abstract: Many studies have been carried out on the application of nanoparticles in the field of lubrication. The reduction of friction and wear are dependent on the characteristics of nanoparticles such as size, shape and concentration. The characteristic study of lubricants with both nanotubes and nanopowders as additives, has not been done so far. This experiment is carried out in a pin-on-disc tribometer for friction and wear monitoring. The test is done by introducing different sample lubricants to the disc running against the pin at the rate of 15ml/min. The wear track radius was set at 120mm in the tribometer. A dead weight load of 5Kg was applied for all the tested samples. The Sample which constitute of 0.75\% nanotube and 0.25\% nanopowder mixed to SAE 30 grade oil shows the best tribological behaviour. It enhances the frictional force behaviour by 43\% and wear reduction by 38\% comparing to additive less lubricant.

Considering the cost effectiveness, every sample having additive is costly. However, the intermixed samples, that is, sample 3, sample 4 and sample 5, are less costly than sample 2, which is composed of nanotube additive alone. The exfoliation of nanotubes may be the possible reason for betterment in results while using nanotubes alone as additives (Sample 2). Since the nanopowders are so small in size, they can easily fill the asperity gaps and hence they prevent direct material contact. The best result provided by sample 4 may be due to the combined effect of these two mechanisms.

Index Terms: CuO Nanopowder, CuO Nanotube, Nanolubricant, Scanning Electron Microscopy, Tribometer.

1. Introduction

Conservation of materials and energy is becoming a very important global issue. The main cause of energy loss in a mechanical system is friction which can be reduced by lubrication. Selection of a good combination of a base-oil and proper additives is significant for effective lubrication. Many studies have been carried out on the application of nanoparticles in the field of lubrication. The reduction of friction and wear are dependent on the characteristics of nanoparticles such as size, shape and concentration. Many researchers report that the concentration of nanoparticles in the base-oils is an important parameter while formulating the nano-lubricants. The significant observation is that a low concentration of nanoparticles is sufficient to improve the desired property of the base-oils.

Cupric Oxide (CuO) can be synthesized in a large variety of forms: particles, nanotubes, multiwall nanotubes and also in the form of ropes, ribbons and thin microtubes several μm in diameter and millimeters in length. They can also be produced in batches of uniform diameter and electronic properties in contrast to carbon nanotubes, which are always grown as mixtures of metallic and semiconducting tubes with a huge dispersion of band-gap values. Before building functional and reliable electronic devices out of them, carbon nanotubes have to be sorted according to their electronic properties. Their richness in form, together with unique physical properties promises potential applications for CuO nanotubes going beyond those of carbon nanotubes. Copper is a Block D, Period 4 element, while oxygen is a Block P, Period 2 element. Copper oxide nanoparticles appear as a brownish-black powder. They can be reduced to metallic copper when exposed to hydrogen or carbon monoxide under high temperature. They are graded harmful to humans and as dangerous for the environment with adverse effect on aquatic life.
1.1. Structure of CuO nanopowder

The surface morphology of the CuO nanopowder was revealed through the SEM image shown in figure 1. It shows a homogeneous distribution of spherical particles of the prepared CuO nanopowder. Figure 2 shows the TEM image of CuO nanopowder. TEM images confirmed the connectivity between the spheres which observed in SEM pictures.

1.2. Structure of CuO Nanotube

Synthesis of CuO nanotubes are done by thermal oxidation method. It is one of the very simple and cost effective way among all the known synthesis methods to grow the CuO nanotubes. The procedure simply involved the thermal oxidation of these substrates in air and within the temperature range from 400 to 700 0C. Electron microscopic studies indicated that these nanowires had a controllable diameter in the range of 30–100 nm with lengths of up to 15 µm.

2. Experimental Work

2.1. Material Selection

2.1.1. Oil

The work is usually done on Polyalphaolefins (PAO) oils. There are different grades of SAE oils based on the requirements. For the current work, the lubricant chosen is Synthetic Oil of grade SAE 30. Synthetic oil is a lubricant consisting of chemical compounds that are artificially made (synthesized). Synthetic lubricants can be manufactured using chemically modified petroleum components rather than whole crude oil, but can also be synthesized from other raw materials. Synthetic oil is used as a substitute for lubricant refined from petroleum when operating in extremes of temperature, because, in general, it provides superior mechanical and chemical properties to those found in traditional mineral oils. Aircraft jet engines, for example, require the use of synthetic oils, whereas aircraft piston engines do not. Synthetic lubricants are also used in metal stamping to provide environmental and other benefits when compared to conventional petroleum and animal fat based products.

2.1.2. Nanotubes and nanopowders

Cupric Oxide an inorganic compound having the formula CuO. Copper has 2 stable oxides and CuO is one among that. It is a black solid that is extracted from copper ore. Copper(II) oxide belongs to the monoclinic crystal system. The copper atom is coordinated by 4 oxygen atoms in an approximately square planar configuration. It is produced on a large scale by pyrometallurgy used to extract copper from ores. The ores are treated with an aqueous mixture of ammonium carbonate, ammonia, and oxygen to give copper(I) and copper(II) ammine complexes, which are extracted from the solids. These complexes are decomposed with steam to give CuO. It is widely used as a solid lubricant because of its low friction properties and robustness. CuO with particle sizes in the range of 1–100 µm is a common dry lubricant [5]. Few alternatives exist that confer high lubricity.
and stability at up to 350 °C in oxidizing environments.

2.1.3. Pin

The pin is manufactured by CNC machining. CNC machine was available at Industrial Estate, Trivandrum. The pin is made of Mild Steel. The pin is having a diameter of 10mm and length 33mm. The tribometer can be worked with 5 different diameter pins, via. 2mm, 5mm, 8mm, 10mm and 12mm. These are the ASTM G40 standard dimensions for measuring wear and erosion. A ball rigidly held is often used as pin specimen. Here, the tip of pin is made hemispherical. A total of 6 pins are needed for the experimentation of selected samples.

2.2. Test Samples

The thorough examination of literatures shows that the amount of nanomaterials to be added is 1% by weight, for observing the optimum result [2] [3]. The experiment will be done by mixing the tubes and powders under various concentrations. The optimum value of coefficient of friction will be determined hence. The samples to be tested are,

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Percentage of Lubricant</th>
<th>Percentage of Nanopowder</th>
<th>Percentage of Nanotube</th>
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</thead>
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<tr>
<td>1</td>
<td>100%</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>99%</td>
<td>1%</td>
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</tr>
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<td>99%</td>
<td>0</td>
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</tr>
<tr>
<td>4</td>
<td>99%</td>
<td>0.75%</td>
<td>0.25%</td>
</tr>
<tr>
<td>5</td>
<td>99%</td>
<td>0.25%</td>
<td>0.75%</td>
</tr>
<tr>
<td>6</td>
<td>99%</td>
<td>0.5%</td>
<td>0.5%</td>
</tr>
</tbody>
</table>

3. Results

The results are directly obtained from the readings provided by Pin-on-disc tribometer. The wear track radius was set at 120mm in the tribometer. A dead weight load of 5Kg was applied for all the tested samples. The sliding velocity was set to 3.14m/s and the test was carried out for a time period of 300 seconds which gives an effective sliding distance of 950m. The computer connected with the tribometer gives the plots for wear rate and frictional force with respect to time. A Scanning Electron Microscope is used to visualise and analyse the wear surface at higher magnification.

3.1. Sample 1: Lubricant Without Additives

The first sample was selected for the validation test. The sample was composed of 100% SAE 30 graded oil alone. It shows uniformly increasing wear loss for a period of 180 seconds. After 180 seconds the wear touches its maximum value of 899micrometers. The figure 6 shows the plot for wear. Figure 7 shows the plot for frictional force. It
is observed that the frictional force also reaches a constant value of 5.1N after a time period of 180 seconds. The scanning electron microscopic image of the wear surface of pin is shown by the figure 8. It is clearly seen in the figure that the surface is peeled away due to friction. The SEM image also spots out that a couple of layers of the metal surface is chipped away, which infers the high amount of wear loss.

3.2. Sample 2: Lubricant Containing 1% Nanotube

The second sample was made of 99% SAE 30 oil and 1% nanotube by weight. The presence of nanotubes reduces the wear considerably. The wear rate reaches its maximum value of 602 micrometers after a running time of 115 seconds as shown in figure 9. The frictional force (figure 10) also reaches a stable value of 3.2N after 115 seconds. The frictional force is considerably reduced from sample 1. This can be further conformed by analysing the SEM image (figure 11). The SEM image shows blackish spots, which means the surface is softly removed and apart from figure 6 bulk material loss is not noticed.
3.3. Sample 3: Lubricant Containing 1% Nanopowder

In sample 3, 1% of CuO nanopowder by weight is mixed with 99% of SAE 30 graded oil. The result of wear loss is obtained to be uniform after 160 seconds of running time and its value is 645 micrometers. The coefficient of friction is high comparing sample 2. The frictional force also reached its optimum value after 160 seconds of running time and valued to 4.2N. The SEM analysis proves that there is considerable more wear than sample 2. In the SEM analysis shown in figure 14 shows more chipped away regions than figure 11, which clearly supports the higher wear loss.

3.4. Sample 4: Lubricant Containing 0.75% Nanotube and 0.25% Nanopowder

Sample 4 is combination of nanopowders and nanotubes. The sample constitute of 99% SAE 30 grade oil with 0.75% nanotube and 0.25% nanopowder by weight. This sample provides the least wear loss among other samples. After a running time of 110 seconds the wear loss becomes a constant value of 555 micrometers and frictional force touches a constant value of 2.9N, which are the best among the samples (figure 15 and figure 16). This can be very well justified by the SEM image of the sample’s wear surface (figure 17). The figure 17 points out very less chipped away regions and the
major portion is seen as blackish or whitish smooth shades. These are the clear indications of minimal wear. The smooth shades are formed because the abrasive wear was low due to the influence of nano additive lubricant.

3.5. Sample 5: Lubricant Containing 0.25% Nanotube and 0.75% Nanopowder

Sample 5 contains 0.25% of nanotubes and 0.75% nanopowders mixed to 99% of SAE 30 grade oil by weight (figure 18 and figure 19). The wear and frictional force becomes stable after a running time of 120 seconds. The maximum wear is noted as 591 micrometers which is higher than sample 4. This may be possibly due to the lesser volume of nanotubes in the sample. Also the frictional force is also higher than sample 4 and is recorded to be 4.2N after being stable. From the SEM image shown in figure 20 shows some chipped away region as well as some smoothly removed regions. The chipped away region is little more than seen in figure 17 which clearly justifies the rise in wear loss.

3.6. Sample 6: Lubricant Containing 0.5% Nanotube and 0.5% Nanopowder

The sample 6 is composed of equally added nanotubes and nanopowders, that is, 99% of SAE grade 30 oil is mixed to 0.5% nanotube and 0.5% nanopowder by weight. Figure 21 shows the wear loss plot to time. The wear loss is higher than sample 4 and sample 5. Maximum wear loss is obtained after 115 seconds of running time and is valued to 597 micrometers. The frictional force gets stable after 115 seconds and is noted to be 3.5N which is seen from figure 22 which is a plot between frictional force over time. The SEM image of wear surface is shown in figure 23. The image indicates some chipped away regions and also some smoothly removed regions. This is the clear evidence for the increased wear loss comparing sample 4 and sample 5.
3.7. Comparison of Wear Loss, Frictional Force and Cost

Figure 24 shows the comparison of tribological parameters of all the samples. Lubricant sample 1, which does not contain any nano additive is showing the highest wear and frictional force values. Sample 4 which is composed of 0.75% nanotube and 0.25% nanopowder shows the best result among the samples. Sample 4 yields much better result than sample 2 which is composed of nanotube additives alone.

The exfoliation of nanotubes may be the possible reason for betterment in results while using nanotubes alone as additives (Sample 2) [1]. Since the nanopowders are so small in size, they can easily fill the asperity gaps and hence they prevent direct material contact [11]. The best result provided by sample 4 may be due to the combined effect of these two mechanisms. There may be enough nanotubes to exfoliate and act as thin sheet like coatings and the small ratio of nanopowders could easily fill the asperities of the contact surface. This combined effect could enhance the lubrication mechanism, thereby providing a better lubricant.

The cost for preparation of lubricant is minimal for sample 1 since it doesn’t contain any additives. However, nanotubes can act as excellent lubricant additives but they are very much costly. 100ml of lubricant with nanotubes alone as additives are costlier more than 8,000 INR, which is way too costly for a lubricant. Even if nanopowders are costly, they are less economic than nanotubes. The results show that the intermixing of nanotubes and nanopowders can not only improves the tribological behaviour, but also reduce the cost comparing sample 2. After a closer analysis it can be noted that sample 5 is the least costly one among the intermixed samples and also it provides wear loss closer to sample 2.
Figure 26 and figure 27 readily compares all samples to sample 1. The thorough examination shows that sample 4 is 38% more efficient in wear reduction than using SAE oil without additives. Also sample 4 has 43% more enhanced frictional coefficient comparing sample 1.

4. Conclusion

This work aims at making of a novel lubricant which could yield better tribologically and economically, by the usage of CuO nanotubes and CuO nanopowders as additives. Six different samples with SAE 30 grade oil as the base medium is prepared for testing. The tests are done using a pin-on-disc tribometer. Tribological parameters are obtained from the friction and wear monitor attached to the tribometer.

The Sample 4 which constitute of 0.75% nanotube and 0.25% nanopowder mixed to SAE 30 grade oil shows the best tribological behaviour. It enhances the frictional force behaviour by 43% and wear reduction by 38% comparing to sample 1, which is additive less lubricant. Considering the cost effectiveness, every sample having additive is costly.

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6. References