Tribological Behavior of Organic Anti-Wear and Friction Reducing Additive of ZDDP under Sliding Condition: Synergism and Antagonism Effect

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Tribological Behavior of Organic Anti-Wear and Friction Reducing Additive of ZDDP under Sliding Condition: Synergism and Antagonism Effect

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Abstract: The effect of eichhornia crassipes carbon nanotubes (EC-CNTs) as additives in both base mineral oil (MOO) and vegetable rapeseed oil (ROO) alone and also together with ZDDP additive under sliding condition was studied. The investigation shows that both on individual and in combine state enhanced tribological properties. The coefficient of friction reduction was 40 % and 37.5 % for EC-CNT inclusion against ROO and MOO respectively under 80 N. The study revealed that enough tribofilm were generated thereby separating the two surfaces leading to low COF. In the case of wear effect, combination of the two additives gives substantial reduction of 65.5 % and 70.2 % against MOO and ROO respectively. The study shows that more reduction was obtained with RO + EC-CNT + ZDDP than the other. The use of two additives in combine lubrication shows synergistic effect, however, observed antagonistic effect if MO + EC-CNT + ZDDP is used for long period of time. The study further revealed that EC-CNT does more of anti-wear service while ZDDP improves friction reducing effect as well as anti-wear.

Keywords: EC-CNTs and ZDDP additives, Lubrication, friction and wear, synergism and antagonism

1. Introduction

In industry, damages on the machine elements and loss of energy during lubrication, has sparked a lot of concern in maintaining smoother sliding and improving the energy quality of all machines during operation. As a result, a need to minimize wear and excessive friction in machine lubricated components attracts researcher's attention. The outstanding technique in solving these challenges caused by friction and wear is by incorporating base lubricants with adequate anti-wear additives [1–4]. Several additives have been tested on their anti-wear lubricating enhancement like silica nanoparticles [5], carbon nano-onion [6], ZDDP [7], MWCNTs [8], WS2 nanosheets [9,10], shows great results on sliding surface protection. Yu et al., [11] studied on nano-mechanical features of tribo-films from Cu anti-wear nanoparticles additives. The study observed that two mechanisms were responsible for the anti-wear film achievement. The operation was through chemical reactions by electrochemistry and electrostatic adhesion brought by frictional forces, thereby makes Cu nanoparticles settle on the valleys or worn surface of the sliding element [11].

The operation and performances from the above mentioned additives were partially different, though some tribological benefits could be achieved if blended together for lubrication [12,13]. Due to their molecular behaviors and some other properties, some of the additives if used with another could yield enhanced lubrication [14,15], otherwise referred to as synergism. More so, inability for two blended additives to contribute in reducing friction and wear due to interfering one another, poor compatibility among their molecular constituents are known as antagonism [16]. The negative effect from aminic dispersants and ZDDP anti-wear combination efficiency is an example of antagonism [17–19]. The negative result was because of complex solution formation thereby reduces adsorption or surface reaction by the ZDDP molecules [18)19)20]. However, two additives that do not interfere each other can synergistically yield outstanding performance, since single additive has different beneficial attribute, while sum provides greater effect compare one [21]. In the operation of radical inhibitor and peroxide decomposer antioxidants, much enhancement is expected, since they perform at different phase during oxidation cycle. Therefore, there is need to perform study on the behavior of two anti-wear additive when blended together. In this study, the combination effect of organic formulated Eichhornia Crassipes carbon nanotubes (EC-CNTs) and ZDDP anti-wears on their lubricating properties was investigated. Base mineral oil (MO) and vegetable rapeseed oil (RO) were employed as to fine their compatibility and performance.

2. Materials and Methods

The EC material were harvested in Nigeria, processed into EC-CNT by Tribological department of Universiti Teknologi Malaysia. The formulation was done through cyclic heating approach utilizing Ball mill machine, furnace, oven and other chemicals according [22) 23]. The developed sample was tested to know the nature of the CNT produced, and its functional group as well to ascertain the compatibility with base lubricant using Raman spectroscopy, particle size analyzer and FT-IR respectively.

2.1 Experiment and Procedure

The tribological behavior of the selected additives (EC-CNTs and ZDDP) were conducted using high frequency reciprocating rig machine (tribo-meter). The machine consists of lower plate spacemen filled with oil, and upper ball that is connected to the load and adjusted it to be in contact with the plate before setting into action as shown in Fig. 1. Base mineral oil SN 650 and rapeseed oil (Rap.oil) were tested separately using different additives and in combination. The experiment was conducted under room temperature. The flat plate surface roughness (Ra), hardness and dimeter were 0.02 nm 810 Hv and 40*40mm respectively while the ball was 6-mm-diameter. Before running the test, different setup components were ultrasonically cleaned for 5 minutes with a heptane cleaning agent, followed by ethanol, and finally dried with hot air. This was done before and after each test. Note, the following abbreviation are used in this study like; mineral oil only (MOO), Rapeseed oil only (ROO).

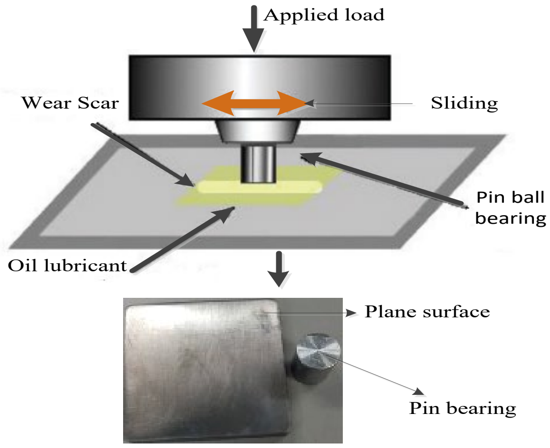

Fig. 1: Image of HFRR sliding components in operation

2.2 Friction and Wear Study

The base mineral and rap. oil was first tested before inclusion of various anti-wear additives, followed the combination of the two additives in the ratio of 1:1. The ratio of the additives on the individual used was 0.5 wt.% each. The volume of base lubricant used in the study was 50 ml. Applied load of 80 N, time duration of 15 min and temperature of 75 ^{0}C were used. Thereafter, load of 40 N, 60 N and 120 N were tested as to study their load carrying ability. The sliding speed of 0.3 m/s was chosen while the frictional force and frictional torque are recorded by the machine. The use of surface profiler and SEM machine incorporated with EDS were employed to analyze lubricated surface roughness, morphologies and element distributions.

3. Results and Discussion

Image of EC-CNTs is shown in Fig. 2 using SEM. The tube-like morphology was detected. The EDX analysis shows elements in EC-CNT with carbon (C) and oxygen (O) having the higher percentage as shown in Table 1. This detected element was also find in previous eichhornia crassipes elemental analysis [24)25)26]. The size analyzer shows that the particle was 98.4 nm.

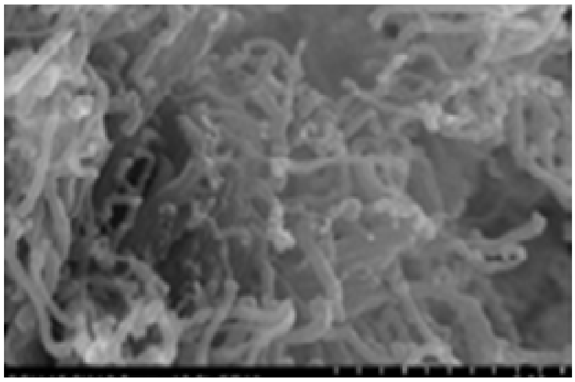

Fig. 2: SEM image of EC-CNT sample showing the tube-like structure.

Table 1: Elemental composition of EC-CNT Sample

Element	Composition quantity (wt.%)
C	70.4
O	15.4
Si	5.3
K	1.8
Al	1.6
Cl	1.5
Cu	1.3
Ca	1.3
Mo	0.5
Mg	0.4
Co	0.3
Ni	0.2

3.1 Raman spectroscopy/FT-IR study of EC-CNT

Fig. 3 shows Raman spectroscopy result of the formulated EC-CNT, compared to untreated EC nanoparticles (EC-NPs). Two peaks were detected in the graphs at 1375.31 and 1601.37 cm^{-1} representing D-band and G-band respectively. The location of those peaks is because of the vibration of carbon atom graphene layer. The modified EC-CNT candidate show pronouncement of the G-band and D-band compared to EC-NPs. The clear peaks on EC-CNT was as a result of the cyclic heating and purification using diluted HCL which was not done on EC-NPs [27)]. The purification caused the sp^3 carbon accessibility and hybridization [28)]. The result is similar to the result analysis carried out by present presentation [28)]. The result of G-band higher intensity than D-band revealed carbon nanotubes and graphene sheet defect [28)].

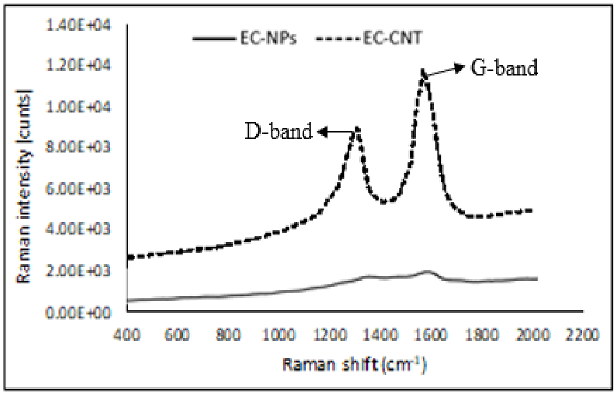

Fig. 3: Images of EC-CNTs and EC-NPs Raman spectroscopy studied indicating D and G bands

Images of FT-IR analysis of EC-CNTs, base oil and oil blended EC-CNT for their functional groups and compatibility are shown in Fig. 4. The results show absorption peaks at 2925 and 2848 cm^{-1} for all the samples, with little change in their transmittance. This effect is from the asymmetric and symmetric stretch of CH_2 present in the samples [29)]. Another peaks found at 1662 and 1638 cm^{-1} caused by COO- group vibration shift within EC-CNTs and base oil resulting in symmetric and asymmetric stretching [30)]. Pronounced shift was found in EC-CNT at 1589 cm^{-1}, caused by poor concentration of COO-group, leading to absorption peak at 1168 cm^{-1} with N-H bond stretching [31)]. Effect from C-O-C stretching behavior at 1012 cm-1 relates to –OH bending from cellulose, hemicellulose and lignin, while 783 cm^{-1} peak comes from aromatic C-H out of plane bends [31)32)]. The blending sample (oil and EC-CNTs) shows shift on the intensity band with a value of 1756 cm^{-1}, attributed to ester cleavage and presence of carboxyl group [32)].

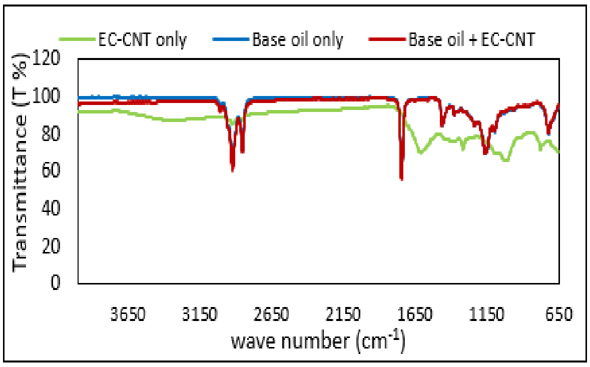

Fig. 4: FT-IR spectra study of base oil, EC-CNT and EC-CNT blended

3.2 Friction Characteristics

Fig. 5 shows the average coefficient of friction (COF) reduction against time under various lubricants (ROO, MOO, RO+ EC-CNTs, MO +EC-CNT, RO +ZDDP, MO +ZDDP, RO + EC-CNT + ZDDP and MO + EC-CNTs + ZDDP) under load of 80 N, time duration of 15 min, frequency of 5 Hz and stroke of 10 mm. From the recorded results, apart from MOO, ROO and MO + EC-CNTs, the lubrication shows full boundary lubrication condition according to stribeck curve [33)]. This indicated that inclusion of both EC-CNTs and ZDDP substantially reduce the operation COF. Under MO + EC-CNTs, shows some high effect at the start of the test but later decreases and stabilize around 190 s till the end of the operation. The values of COF with the use of MO + EC-CNT was 0.1005 against 0.1677 and 0.1608 for ROO and MOO respectively. This demonstrated good COF reduction compared to ROO and MOO respectively as shown in Fig.5.

Using ROO and MOO with ZDDP individually, the result yielded better result than MOO + EC-CNT as listed in Tab. 2. The effect from combining the two anti-wear additive were little compared single used, though still reduce COF. However, testing with RO + EC-CNT +

ZDDP yielded the best result compared to MO + EC-CNT + ZDDP. The reduction was 70.2 % and 65.5 % on ROO + EC-CNT + ZDDP and MOO + EC-CNT+ZDDP against ROO and MOO, respectively. The results variation in the two blended additives might be related to the effect recorded from the use of MOO + EC-CNTs. This result is similar to that of previous study [16]. Though, the outcome shows synergistic effect but the result of MO + EC-CNT + ZDDP if continued for a long period of time will lead to antagonistic effect as shown in Fig. 5. This may be attributed to incompatibility between MO and EC-CNT. The good performance recorded from the use of the additive are attributed to their excellent formation of tribo-film leading to low friction generation with reduced COF. Since the performance of lubricant cannot define with the COF alone, the use of surface roughness (Ra) and wear scar diameter (WSD) was employed [34–36]. Fig. 6 shows the SEM images of the lubricated surfaces with the Ra listed in Table 3.

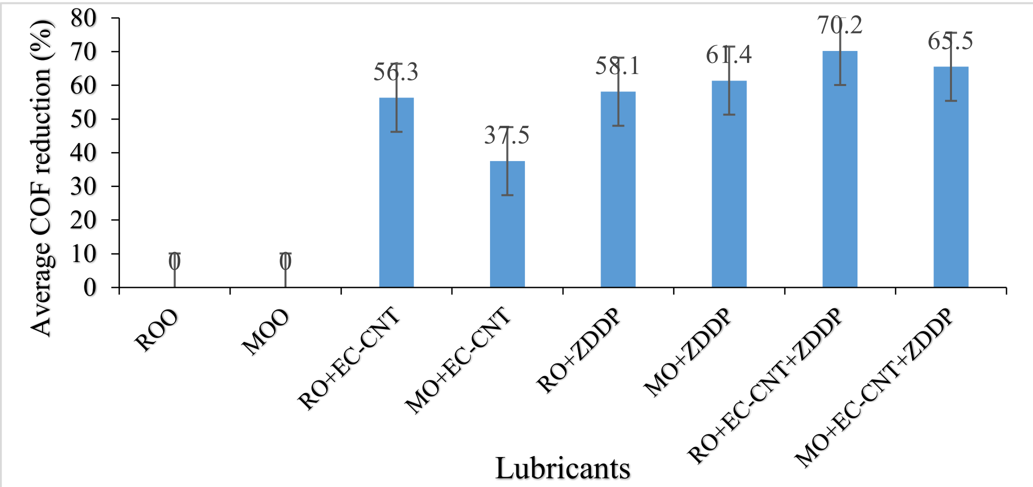

Fig. 5: Average COF reduction on the various lubricants (80 N, 5 Hz, 15 min, 10 mm stroke), against base lubricant

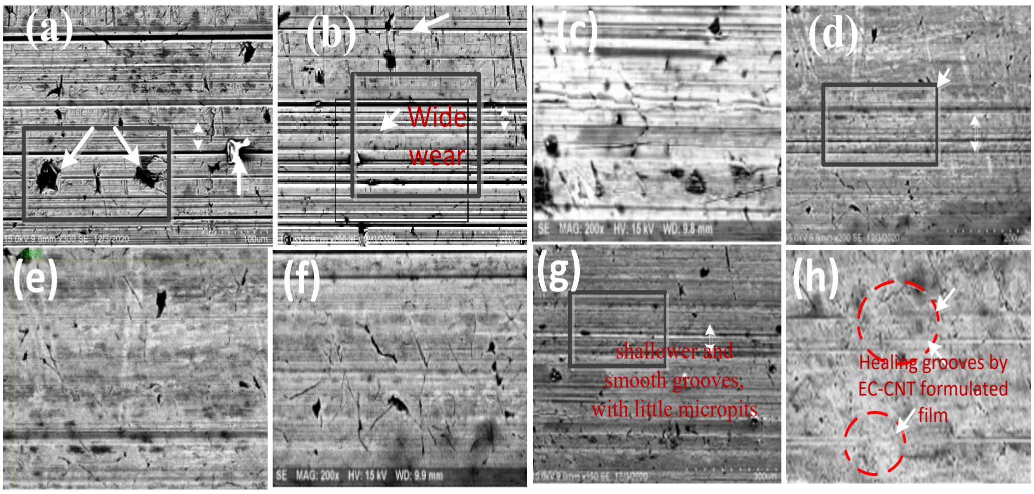

Fig. 6: VEPSEM images of the various lubricated surfaces; a) MOO; b) ROO; c) RO + ZDDP; d) RO + EC-CNT; e) MO + EC-CNT; f) MO + ZDDP; g) MO + ZDDP+ EC-CNT; h) RO + EC-CNT + ZDDP

Table 2: Average mean COF for various lubricants

Lubricant samples	Average mean COF
ROO	0.1677
MOO	0.1608
RO + EC-CNT	0.0733
MO + EC-CNT	0.1005
RO + ZDDP	0.0703
MO + ZDDP	0.0620
RO + EC-CNT + ZDDP	0.0499
MO + EC-CNT + ZDDP	0.0554

Table 3: Surface roughness from various lubricated surfaces

Lubricants	Value (nm)
ROO	0.790
MOO	0.735
RO + EC-CNT	0.481
MO + EC-CNT	0.583
RO + ZDDP	0.411
MO + ZDDP	0.309
RO + EC-CNT + ZDDP	0.289
MO + EC-CNT + ZDDP	0.295

Fig. 7 shows the WSD from the various lubricated surfaces. The tribological results generated from the combination of ZDDP and EC-CNT in both mineral and rap. oil samples are much better than those of only EC-CNTs and ZDDP. However, the lubricating performances both in COF and SWD observed to be close to those of single additives application. During the operation, ZDDP provides some service of friction modifier, while EC-CNT nanoparticles mostly serve as anti-wear [17]. The same effect from ZDDP was reported in previous work [37]. Under single additive of EC-CNT with RO yielded better WSD than MO + EC-CNT, while ZDDP with MO shows smaller value of WSD. Using the two additives, blended surface with RO + EC-CNT + ZDDP gives the best performance both in COF and SWD as shown in Fig. 5. The WSD reduction by MO + EC-CNT + ZDDP and RO + EC-CNT + ZDDP were 4.8 (*10 -3) mm3 and 5.1 (*10 -3) mm3 respectively. This is 57.9 % and 57.1 % reduction compared to MOO and ROO respectively The mechanism observed on the surface during operation shows that the additives provides services of healing, rolling and film formation [17]

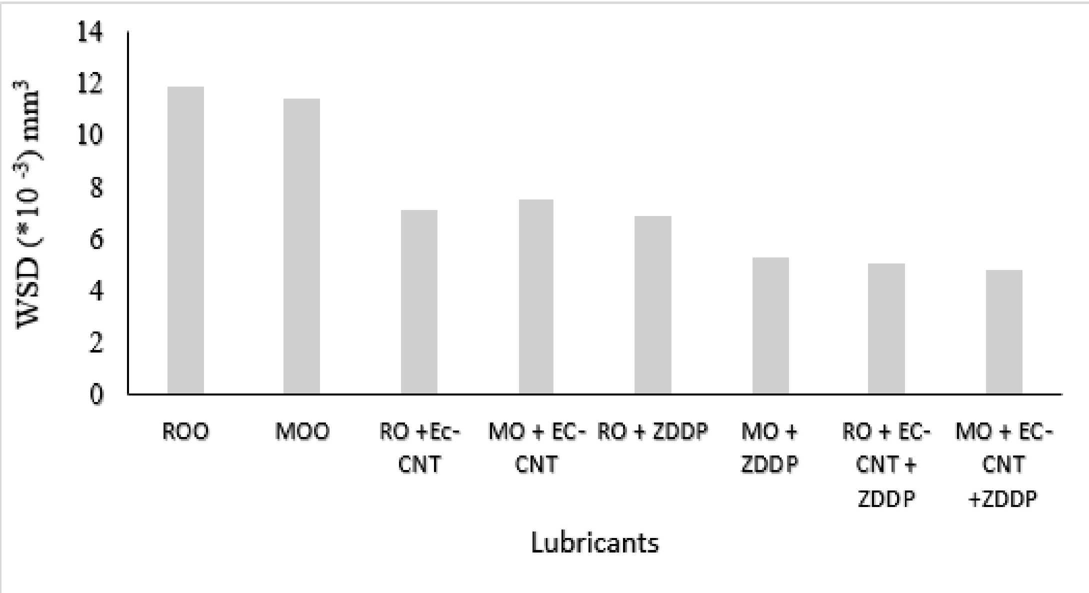

Fig. 7: WSD versus various lubricants under 80 N, 5 Hz, 10 mm stroke and 15 min

Tab. 4. presents the EDX result of VEPSEM analysis conducted on the lubricated surfaces of MOO, ROO, RO + ZDDP, RO + EC+ CNT and RO + EC-CNT + ZDDP respectively. Under MOO and ROO, represent lubricated surfaces of base mineral and rap. oil respectively. The result indicated that the surfaces lubricated with base mineral and rap. oil has more of iron (Fe) elements and little impurities like Cobalt (Co), silicon while oxygen was much on lubricated surface with only rap. oil. This indicates lack of tribo-chemical reaction on the surface contact of the sliding elements. In the base oil testing, no film was developed due to no anti-wear additives in the operation. In the lubricated ZDDP and EC-CNT as in RO + ZDDP, the table shows the presence of Zn, P form ZDDP additive while RO + EC-CNTs, many elements (C, Ca, Si, Mo, Cl, Mo, Al and Na were seen on the graph of the EC-

CNT lubricated surface. The presence of the elements on the graph of lubricated surface by ZDDP and EC-CNT indicated the formation of tribo-film showing occurrence of tribo-chemical reaction during the sliding operation [33]. However, combination of ZDDP and EC-CNT, exhibited similar feature with many element display when tested RO + EC-CNTs, while the Zn indicated the main element of ZDDP. Because of the formation of ZDDP and EC-CNT film between the sliding contact, the surface lubricated with ZDDP + EC-CNT yielded smooth surface compared others while that lubricated with only rap. oil exhibited the worst result.

Table 4. EDX for elemental composition in EC-CNT and EC-CMC Samples

Sample/Element (wt.%)	C	O	Fe	Si	Zn	p	Al	Cl	Co	Ca	Mo	Ca	Na	Total (%)
MOO (base oil)	12.3	3.2	83.8	-	-	-	-	-	0.3	-	-	-	-	100
ROO (base oil)	13.6	7.6	78.6	-	-	-	-	-	0.2	-	-	-	-	100
RO + ZDDP	7.5	6.5	58.6	-	27.4	-	-	-	-	-	-	-	-	100
RO + EC-CNT	24.2	5.4	60.3	1.8	-	1.7	1.3	1.4	1.3	1.2	0.6	0.4	0.5	100
RO + EC-CNT + ZDDP	19.3	5.2	50.7	1.8	19.4	0.3	0.2	0.1	0.6	1.1	1.2	0.1	-	100

3.2 Load Carrying Capacity

Fig. 8 depicts the effect of different loads on the strength of applied lubricants. The addition of the various ant-wear additives shows substantial enhancement on the performance of the lubricant. The result show that lubricated surfaces with only ROO and MOO exhibited similar behavior as shown in the graph (Fig. 8). The two base lubricants (ROO and MOO) show steady increase on the COF with load increment. However, with inclusion of both additives (EC-CNT and ZDDP), showcase good decrease on COF. The COF generated by MOO and ROO were 0.1608 and 0.1677 on 40 N while 0.1485 and 0.1688 on 100 N respectively. The use of EC-CNT with RO gives COF reduction from 0.0839 on 40 N to 0.0689 on 100 N. This is attributed to EC-CNT thermal stability and similar to previous report on carbon nanotube [33] [35]. Similar result was generated from the use of ZDDP on MO and RO. However, the result from blended EC-CNT with MO shows little variation from blended EC-CNT with RO, owing to poor lubrication, caused by delay on tribo-film formation which was not observed from blended EC-CNT with RO. Application of RO + EC-CNT + ZDDP and MO + EC-CNT and ZDDP yielded the lowest COF from 0.0598 and 0.0601 under 40 N to 0.0540 and 0.0501 on 100 N respectively. This implies that under addition of the two additives, the more the load, the better the performance with lower COF [17]. This can be attributed to the synergetic behavior between the two additive, where ZDDP acting as friction modifier and anti-wear together with EC-CNTs. This is similar with previous presentation [20].

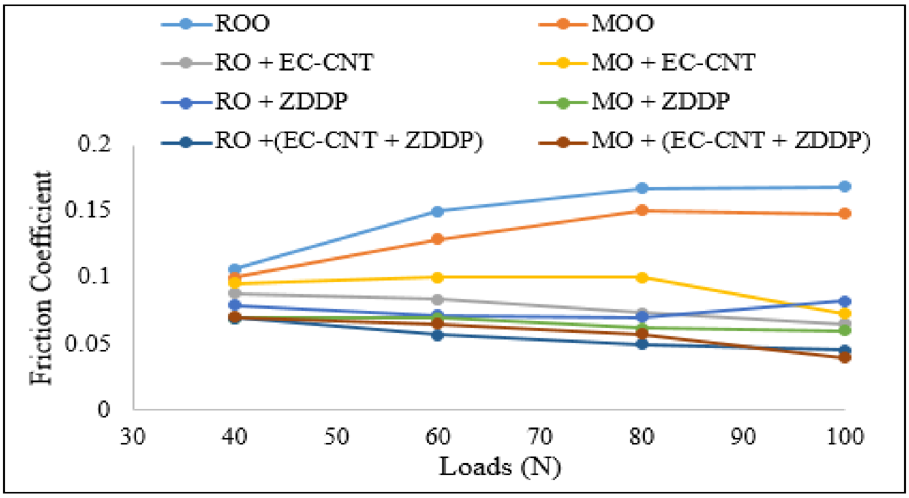

Fig. 8: Coefficient of friction against load on various lubricants

4.0 Conclusion

The tribological behavior of two anti-wear additives performance both on individual and combine form under sliding lubrication condition were investigated in this study. First, the base lubricants were test alone before inclusion of the additives. The outcome indicated that friction and wear were reduced significantly. Under COF, inclusion of individual additives was reduced by approximately 40 % and 37.5 % for RO + EC-CNT against ROO and MOO, but further reduced by 68.9 % and 70.2 % under MO + EC-CNT + ZDDP and RO + EC-CNT + ZDDP against MOO and ROO respectively. The reduction effect revealed that tribofilm were formed during lubrication. Under wear analysis, shows that combination of EC-CNT and ZDDP can provide adequate surface protection. The surface roughness was reduced as well as the wear. The reduction recorded from the two additives in the two different base lubricants were very close. This demonstrated that observed nano particles from EC-CNT and tribo-film are capable of providing good lubrication and prevents direct contact between the sliding surfaces. Developed synergistic effect among the used anti wear additives is responsible for the low COF and smooth surfaces during the operation.

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Nomenclature

COF	Coefficient of friction
EC-CNT	Eichhornia Crassipes Carbon Nanotubes
MOO	Mineral oil only
ROO	Rapeseed oil only
MO	mineral oil
RO	Rapeseed oil
WSD	Wear scar diameter

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