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ENHANCEMENT OF TRIBOLOGICAL PROPERTIES OF LUBRICATING OILS USING CUO AND NIO NANOMATERIALS

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ABSTRACT

This paper aims to understand the tribological properties of lubricant oils containing CuO and NiO nanoparticles. The synthesized CuO and NiO nanoparticles were characterized using XRD, SEM,EDAX and IR spectral technics. XRD results revealed that CuO and NiO nanoparticles exhibited in the monoclinic and cubic phase respectively and kinematic viscosity studies indicated that the base oils obtained by catalytic dewaxing with hydrocracking and hydro finishing iso-dewaxing technology is better than extraction with hydro dewaxing technology. The High-Frequency Reciprocating Rig and Four balls wear and scar analysis emphasized that the addition of CuO and NiO nanoparticles effectively reduced the wear scar properties of MB150 and MB500 base oil samples. This study is more useful for the lubricant manufacturer for its selection of base oils and their formulations.

Keywords: Antiwear, Viscosity, HFRR, Fourball wear tester, Base oils, Nano-additives

1. INTRODUCTION

Developing environmentally based lubricants is an important target for the lubricants industry. Chemically modified bio-based oils were used as an emerging alternative to mineral and synthetic base oils [1]. The addition of nanoparticles viz., Al₂O₃ SiO₂ and TiO₂ into lubricant oils, impact the thermophysical parameters particularly thermal conductivity and viscosity. It provides low kinematic viscosity and an increase in the viscosity index by 2%. Moreover, thermal conductivity was enhanced by 12-16%, compared with the case of engine oil without nanoparticles [2, 3]. Fe, Cu and Co nanoparticles and their combinations showed reduced friction coefficient and wear. The copper nanoparticles provide the most effective reduction in friction [4]. Lubricated friction and wear behaviours were examined with E52100 steel reciprocating Pin-on-Disc apparatus using TiO₂ as an additive. The friction and wear characteristics were examined with constant load and found that the increased coefficient of friction [5]. The tribological properties of paraffin have been studied with the addition of SiO₂[6].Addition of MoS₂andCuO nanoparticles in engine oil exhibits good friction reduction and anti-wear properties and also decreased the coefficient of friction as compared with standard engine oil without MoS₂ and CuO nanoparticles [7]. The silicon-based nanoparticles in lubricants shows reduce the real area of contact, therefore, decrease the frictional force. Also, the particle size and distributions are playing a key role in tribological nano lubricants [8].Copper oxide nanoparticles showed a reduction in sliding wear and friction in coconut oil and rapeseed oil[9,10]. Addition of SiO₂ in coconut oil which increases the viscosity but the addition of CuO nanoparticles in coconut oil leads to viscosity decreases. However, the addition of CuO and SiO2 in coconut oil enhanced the friction reduction properties [11-13]. The addition of nano-hybrid pour point depressant (NPPD) on flow property shown improvement in the viscosity of the base oils [14]. Fullerene nanoparticles mixed with base oils and found that the frictional coefficient was decreased by 90% in compared with raw oil [15]. Copper oxide nanoparticles as additives in lubricants reduced friction and show improved wear resistance [16-18]. Lubricant industries are using inorganic based additives for improving the tribological properties however; these additives are being used in 0.5 % to 2.5% which increases the input cost. Hence, there is a need to develop an additive which gives same performance but used in very small quantity. In this study, CuO and NiO

nanoparticles were used as surface-active materials to reduce the wear and scar tribological properties of the base oils and their observed results are discussed.

2. EXPERIMENTAL

2.1. Materials and methods of NiO and CuO nanoparticles

The chemicals and reagent such as Nickel chloride hexahydrate, Sodium hydroxide and Copper nitrate hexahydrate were procured from Sigma Aldrich and used without further purifications.

2.2. Synthesis of Nickel Oxide (NiO)nanoparticles

8g of nickel chloride hexahydrate (NiCl₂.6H₂O) was dissolved in 60 ml of de-ionized water and 6g of sodium hydroxide were dissolved in 40ml of de-ionized water then the mixture was stirred at 60°C for 4 hours followed by the formation of Ni(OH)₂. The synthesized Ni(OH)₂ were calcinated at 450°C for 2 h and 4 h in Muffle Furnace to obtain the NiO nanoparticles [19].

2.3. Synthesis of Copper Oxide (CuO) nanoparticles

For the synthesis of copper oxide nanoparticles, 2.9 g of copper nitrate is mixed with 1.2 g of sodium hydroxide and 100 ml of distilled water. Then the mixture was stirred at 80°C for 3 hours, a black precipitate was formed and the precipitate was centrifuged and dried at 500°C for two hours to get the copper oxide nanoparticles [20].

2.4. Characterization

Fourier transform infrared (FTIR) spectra were recorded on a Bruker tensor 27, under the wavelength of 4000 -400 cm⁻¹ range and the sample was prepared by KBr pellet method. The X-ray diffraction (XRD) patterns of the nanomaterials were measured with a Model D/ max-RC X-ray diffractometer using Cu-K α radiation source (k =1.5418 Å) and operating at 40 kV and 100 mA. Energy-dispersive X-ray spectroscopy (EDX) and SEM measurements were carried out on JSM-5610LV.

2.5. Base oil samples description

A pure sample of group II base oils was selected for this study namely MB150, MB500, HPC150 and HPC 500.MB150 and MB500 samples were manufactured through hydrocracking with iso-dewaxing and hydro finishing type of technology whereas HPC150 and HPC500 samples were made through solvent extraction with catalytic iso- dewaxing type of technology.

2.6. Physio-chemical tests

Kinematic viscosity with various temperatures, Compositional analysis and High-Frequency Reciprocating Rig analysis with the addition of nanomaterials (0.4%)was carried out using international standard methods viz. ASTM D 445, ASTM D 3238 and ASTM D6079 [21].

3. RESULTS AND DISCUSSION

3.1.Characterization of CuO and NiO nanomaterials

3.1.1.X-ray diffraction analysis

The XRD analysis determined the phase present in the nanoparticles and their results are shown in Fig 1.The XRD pattern was recorded in the fraction angle 2θ range 10° to 80° . The XRD patterns of the calcined samples exhibited sharper reflection peaks confirmed the good crystallinity and fine grain size.



Fig. 1: XRD pattern of the synthesized NiO and CuO nanoparticles

For CuO nanoparticles peaks appearing at $2\theta^{\circ} = 33.97$, 39.09, 41.38, 58.45, 61.98, 73.08, 75.25 and 75.45° can be readily correlated for (110), (200), (112), (202), (113), (222) and (204) crystal planes of the bulk CuO. By comparing these data with known standard data published by the Joint Committee on Powder Diffraction Standards (JCPDS Card No:05-0661) it is clear that the CuO nanoparticles exhibited with monoclinic phase. The observed XRD pattern of NiO at $2\theta^{\circ}=31.07$, 37.28, 43.79, 45.98, 57.03, 63.08, 75.93 and 80.08° can be readily responsible for (033), (012), (104), (110), (015), (021) and (006) planes. By comparing standard values (JCPDS Card No: 22-1189), it is clear that the NiO nanoparticles with cubic phase were formed and no characteristic peaks were observed other than NiO [22, 23].

The structural parameters are calculated such as crystallite size, microstrain and dislocation density of CuO and NiO nanoparticles by Debye–Scherer's formula given by the equation 1,2&3 and the values are tabulated in Table 1.

Crystalline size $(D) = \frac{0.9\lambda}{\beta \cos\theta}$ ----(1) Dislocation density (δ) = ----(2)

Macrostrain (ϵ)

Where, λ is the wavelength ($\lambda = 1.546^{\circ}$ A) (CuK α), β is the full width at half maximum (FWHM) of the line and θ is the diffraction angle.

The lattice parameters and volume have been calculated using the following equations 4 and 5 and the values are presented in Table 1.

$$a = d\sqrt{h^2} + k^2 + l^2 \dot{A}$$
 ----(4)
V=a³(m³) ----(5)

----(5)

----(3)

Samples	Average Crystallite size (nm)	Lattice Parameter (Å) & Volume (m³)	Dislocation Density $\delta \times 10^{15} (m^{-2})$	Micro strain $\epsilon \times 10^{-3}$
CuO	18.87	a =4.684,V=81.16	2.7510	2.2934
NiO	19.68	a =2.954,V=54.68	3.5043	1.9240

(SEM) 3.1.2. Scanning Electron Microscopy and Elemental Analysis (EDAX)

The surface morphology and composition of CuO and NiO nanoparticles were investigated by using a scanning electron microscope (Fig.2) equipped with EDAX analysis (Fig.3). This analysis shows that the NiO

nanoparticles were spherical in shape which is uniformly distributed along the surface. The SEM image of CuO revealed the CuO nanoparticles exhibited as nanoflakes. EDAX analysis (Fig.3) was used to interpret the elemental and chemical composition of the prepared nanoparticles [24].



Fig. 2: SEM images of NiO and CuO nanoparticles



Fig. 3: EDAX spectrum of NiO and CuO nanoparticles

3.1.3. FT-IR spectra of CuO and NiO

The FT-IR spectras of the synthesized CuO and NiO nanoparticles (Fig.4) were recorded in the range of 400-4000 cm⁻¹ which identifies the chemical bonds as well as a functional group of the compounds. The broadband

appeared at 3440cm⁻¹ is ascribed to the O-H stretching vibration of the adsorbed water molecule on the surface of the nanomaterials. The strong band appeared around 465 and 484cm⁻¹ are assigned to metal-oxygen stretching mode (M-O-M) of the CuO and NiO nanoparticles [25].



Fig. 4: FT-IR spectrum of CuO and NiO nanoparticles

3.2. Analysis of Base oil Samples

Base oil samples namely MB150, MB500, HPC150and HPC500were selected for this antiwear, kinematic viscosity studies and compositional analysis. The compositional analysis showed that all the selected base oils were meeting the characteristic features of group II base oils. Further, these base oils were added with copper-based nano antiwear additive (CuO;0.4%) and nickel-based antiwear additive (NiO;0.4%) and the observed results are discussed in this research paper.

3.2.1. Compositional analysis

The stability of base oils is based on its paraffinic and naphthalenic carbons. The presence of more paraffinic leads to more stability similarly, less of naphthenic showed more stability of base oil [26]. The paraffin present in MB150 was more than HPC150 and naphthene in MB150 was less than HPC150. Similarly, paraffin of MB500 is more than HPC500 and naphthene of MM500 less than HPC500 (Table 2).

				Base oils +CuO	Ca	Ср	Cn	Base oils	Ca	Ср	Cn
Base oil	Ca	Ср	Cn					+NiO			
MB150	0	73.64	26.36	MB150	0	75.31	24.69	MB150	0	74.93	25.07
HPC150	7.16	61.32	31.52	HPC150	0	73.37	26.63	HPC150	0	73.27	26.73
MB500	0	75.06	24.94	MB500	7.34	63.01	29.65	MB500	7.46	63.26	29.28
HPC500	7.46	62.94	29.6	HPC500	6.91	60.88	32.21	HPC500	6.97	60.98	32.05

Table.2: Composition of Base oils with and without additives

(Ca-Aromatic carbons; Cp-Paraffinic carbons and Cn-Naphthenic carbons)

After the addition of CuO and NiO (0.4%), the paraffin of MB150 more than HPC150 and naphthene of MB150 was less than HPC150. Similarly, the paraffin of MB500

was more than HPC500 and naphthene of MB500 less than HPC500. The percentage changes of paraffinic and naphthenic carbons of MB150 & MB500 were less than HPC150 & HPC500. Based on the above observations MB150 & MB500 were more stable than HPC150 & HPC500 even after the addition of nanomaterials. This reveals that MB150 & MB500 were the best base oils than HPC150 & HPC500.

3.2.2. Kinematic viscosity study

The kinematic viscosity of CuO and NiO nanoparticles added MB150, MB500, HPC150 and HPC500 were studied in different temperatures and the results are presented in Tables 3-5. The percentage change in viscosity with temperature shows MB150, MB500 is lesser than the HPC150 and HPC500 respectively. This shows that MB150 and MB500 did not undergo changes in viscosity and it is not showing thickening like HPC 150 and HPC 500 base oils. Similarly, copper-based nano additive in MB500 and MB150 the percentage change in viscosity with temperature shows lesser than the HP150 and HP500 respectively where the synergy between MB base oils are showing better compared with HPC base oils. Further, the percentage change in viscosity with the temperature of nickel-based nano additives added MB500 and MB150 are showing lesser than HP500 and HP150. In conclusion, Nickel-based additive added with MB500 and MB150 shows better viscosity property than copperbased additive added with MB500 and MB150. Similarly, nickel-based additive added with HPC500 and HPC150 shows better viscosity property than copper-based additive added with HPC 500 and HPC150. This concludes that Catalytic dewaxing with hydrocracking and hydro finishing iso-dewaxing technology is better than extraction with hydro dewaxing technology [27].

Table 3: Kinematic Viscosit	у ((KV)	changes o	of Base	oils	with	tem	perature)
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KV @Temp	MB150	MB150	HPC150	HPC150	MB500	MB500	HPC500	HPC 500
		%Change in KV		% Change in KV		% Change in KV		% Change in KV
100°	6.13		5.27		11.07		10.73	
80°	9.40	53.34	8.28	57.12	18.69	68.83	18.34	70.92
60°	16.93	80.11	14.70	77.54	36.18	93.58	37.16	102.62
50°	23.81	40.64	20.82	41.63	53.85	48.84	56.94	53.23
40°	35.50	49.10	31.24	50.05	84.04	56.06	92.37	62.22
0°	321.90	806.76	323.40	935.21	1374.00	1534.94	1517.00	1542.31

Table4: Kinematic Viscositychanges of Base Oils with 0.4%Copper oxide nanoparticles

KV @Temp	MB150	MB150	HPC150	HPC150	MB500	MB500	HPC500	HPC 500
		%Change in KV		% Change in KV		% Change in KV		% Change in KV
100°	11.28		10.63		6.05		5.20	
80°	19.05	68.88	18.45	73.57	9.48	56.69	8.14	56.54
60°	36.79	93.12	37.42	102.82	16.67	75.84	14.41	77.03
50°	54.93	49.31	57.27	53.05	23.37	40.19	20.42	41.71
40°	85.97	56.51	92.72	61.90	34.47	47.50	30.40	48.87
0°	1384.00	1509.86	1524.00	1543.66	321.50	832.70	312.90	929.28

Table5: Kinematic Viscosity changes of base oils with0.4% Nickel oxide nanoparticles

KV @Temp	MB150	MB150	HPC150	HPC150	MB500	MB500	HPC500	HPC 500
		%Change in KV		% Change in KV		% Change in KV		% Change in KV
100°	11.27		10.60		6.06		6.14	
80°	18.98	68.41	18.43	73.87	9.49	56.60	9.74	58.63
60°	36.88	94.31	37.28	102.28	16.64	75.34	16.41	68.48
50°	54.99	49.11	57.10	53.17	23.39	40.56	23.42	42.72
40°	85.88	56.17	92.43	61.87	34.59	47.88	35.40	51.15
0°	1344.00	1464.97	1514.00	1538.00	321.90	830.62	338.9	857.34

3.2.3. High-Frequency Reciprocating Rig Test

In this analysis, 2 ml sample was taken in the test reservoir and the temperature was adjusted nearer to 60°C. Once the temperature is stabilized a vibrator arm holding a non- rotating steel ball loaded with a 200g mass is lowered until it contacts a test disk completely submerged in the sample. The ball is caused to rub against the disk with 1-mm stroke at a frequency of 50Hz for 75 minutes. The ball is removed from the vibrator arm and cleaned. The dimensions of the wear scar were measured and recorded (Table 6 and 7). Copper-based nano additive (CuO; 0.4%) is added with MB150 and MB500 as well as HPC 150 and HPC 500 base oils and found that MB150 and HPC 150 base oils not showing any reduction in wear scar values. However, MB500 and

HPC 500 showed a reduction in wear scar values. This may be due to thicker base oils has synergy with these additives and hence it shows a reduction in wear scar. Similarly, the nickel-based nano additive (NiO;0.4%) was added with MB150 and MB 500 as well as HPC 150 and HPC 500 shows the reduction in wear for all samples. This may due to nickel-based additive is working very well with these base oil whereby reduction in wear. It has also been noticed that nickel-based additive has synergy between MB 150 and MB500 base oils thereby tremendous reduction compared with HPC150 and HPC500 base oils. This may due to more of saturated paraffinic hydrocarbon present in MB150 and MB500 base oils.

Table 6: HFRR Test results of Base Oils with 0.4% of Copper-based nano additive

Sample	MB150	MB150+CuO	MB500	MB500+CuO	HPC150	HPC150+CuO	HPC500	HPC150+CuO
Scardiater(Mm)	567	608	695	399	260	347	243.5	241

 Table 7: HFRR Test results of Base Oils with 0.4% of Nickel-based nano additive

Sample	MB150	MB150+NiO	MB500	MB500+NiO	HPC150	HPC150+NiO	HPC500	HPC500+NiO
cardiater(Mm)	567	354	695	319	260	241	243.5	238

3.2.4. Four-Ball Wear Scar Test

Three steel balls are clamped together and immersed with lubricant or test oil and fourth steel ball is pressed with force 40kgf into the cavity formed by three balls for three-point contact. The temperature was maintained at 75°C and kept the top ball rotate in the speed of 1200rpm for 60 minutes. Once the test was completed all three lower balls were removed and measured the wear scar and the values were tabulated in Table 8 and 9.

Table 8: Wear scar test results of the Base oil samples

Sample	Wear scar test 1	Wear scar test 2	Wear scar test 3	Wear scar Average
MB500	900.30	707.58	906.31	838.06
MB150	864.17	831.05	806.96	834.06
HPC 500	719.64	707.59	689.53	705.59
HPC150	885.24	909.33	894.28	896.28
MB500 +CuO	731.68	653.43	746.74	710.62
MB150+ CuO	728.67	596.18	719.64	681.50
HP 500+ CuO	812.98	828.04	853.13	831.38
HPC150+ CuO	1038.8	1077.9	975.58	1030.76
MB500 + NiO	644.36	662.43	752.76	686.52
MB150+ NiO	725.12	613.12	671.12	669.79
HPC500 + NiO	858.15	822.01	846.01	842.06
HPC150+ NiO	906.32	945.47	1047.80	966.53

MB 150 and MB500 base oils were shown less wear scar than HPC 150 and HPC 500 base oils. A similar trend also appeared with a copper-based additive(CuO;0.4%) added with MB150 and MB500 and the wear scar is reduced from 834.06mm to 681.38mm and 838.06mm

to 710.62mm respectively whereas inHPC150and HPC 500 the wear scar values increased from 896.28mm to 1030.76mm and 705.59mm to 831.38mm respectively. Nickel-based additive(NiO;0.4%) added with MB150 and MB500 shown less wear and the wearscar magnitude

is efficiently reduced from 834.06mm to 669.79mm and 838.06mm to 686.52mm respectively. But this nickelbased additive was not had synergy with HPC 500 and wear scar value increased from 705.59 mm to 842.06mm. This result concluded that addition of CuO and NiO nanomaterials could effectively improve the performance of MB150 and MB500 base oils due to the tribe-sintering of nanoparticles on the wear surfaces and reducing the metal-to-metal contact and thereby reducing the friction and wear behaviours [28].

Base Oils	Wear scar(mm) Without additive	Wear scar(mm) with CuO	Wear scar(mm) with NiO
MB500	838.06	710.62	686.52
MB150	834.06	681.50	669.79
HPC500	705.59	831.38	842.06
HPC150	896.28	1030.76	966.53

Table 9: Average wear	scar test r	esults of	Base oils	with nan	o additives
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4. CONCLUSION

The physiochemical test results of all samples (MB150, MB500, HPC 150&HPC 500) showed that all are group II base oils meeting the sulphur percentage less than 0.03, saturates were more than 90 and Viscosity Index ranged between 80 and 120.Paraffin of MB150was higher than HPC 150 and naphthenes of MB150 were lower than HPC 150 which showed that MB150is superior oil than HPC150. Similarly, Paraffin of MB500 is higher than HPC 500 and naphthenes of MB500 is lower than in HPC 500 which shows that MB500 is superior oil than HPC 500.High-Frequency Reciprocating Rig test results concluded that addition of 0.4% CuO and NiO nanomaterials to the base oils effectively reduced the wear scar diameter in the MB500 and HP500 base oils. The Four Ball wear scar analysis indicated that the addition of 0.4% CuO and NiO nanomaterials to the base oils highly influenced to reduce the wear scar values of MB150 and MB500 base oils. To conclude, nickel based additive shows a better reduction in wear scar values than the copper based nano additives indicated that NiO nanoparticles may used as a best surface modifier which could significantly improved the anti-frictional and antiwear properties of MB150 and MB500 base oils.

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