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Studies on thermo-physical and tribological properties of nano-lubricants formulated with surfactant-modified CeO_2 nanoparticles in 500n base oil – A response surface methodology approach

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ABSTRACT – Thermo-physical and tribological properties of nano-lubricants formulated from 500N base-oil and cerium oxide (CeO_2) nanoparticles are studied using a customized pin-on-disc tribometer. CeO_2 nanoparticles are prepared by precipitation method and characterization is carried out using FESEM and EDS. Experimental data are used to formulate a response surface methodology (RSM) model in ANOVA using box behnken design (BBD). Optimum concentration of CeO_2 nanoparticles in 500N base-oil for minimum friction and wear is estimated to be 0.56% weight. Effect of temperature on viscosities and flash and fire-points of the lubricants are studied for different nanoparticle concentrations. Stability analyses by dynamic light scattering (DLS) have been carried. FESEM imaging coupled with EDS analyses helps to identify the possible mechanisms of lubrication.

1. INTRODUCTION

Nano-lubricant in the present paper represents a lubricant obtained by the addition of nanoparticles in base-oils. Many investigators have reported that the addition of nanoparticles to the base lubricants is an effective method to reduce friction and wear [1-2]. The friction-reduction and anti-wear behaviours of the nano-lubricants were shown to be dependent on the characteristics of nanoparticles such as size, shape and concentration. This work aim to synthesis CeO_2 nanoparticles using precipitation method which requires less energy for production. Another important challenge faced by lubricant formulators is the optimization of concentration of additives in lubricating oils. This work aims to demonstrate a comprehensive optimization procedure for the nanoparticle additive used based on experimental results and with the help of a powerful statistical tool based on design of experiments (DOE).

2. EXPERIMENTAL METHODOLOGY

CeO_2 nanoparticles are prepared by precipitation method and the characterization of the nanoparticles are carried out using Field Emission Scanning Electron Microscopy (FESEM) and Energy Dispersive Spectroscopy (EDS). RSM is used to examine the influence of factors such as load, speed, temperature and concentration of nanoparticles on the tribological behaviour of nano-lubricants formulated using paraffin-based 500N base-oil by the addition of CeO_2 nanoparticles on weight percentage basis. Tribological properties are evaluated using a modified pin-on-disc tribometer along with a specially designed attachment for heating the lubricant. Coefficient of friction (COF) and specific wear rate (SWR) are used as the response factors. Design Expert® 7.0.0 software is used for the analysis and the experiments are planned using BBD procedure. Based on the BBD design, 29 experiments are conducted for base-oil with CeO_2 nanoparticles. In addition to the tribological studies, viscosity and flash and fire-point analyses, stability analyses and surface morphology analyses have also been conducted to recommend its use as a crank case oil.

3. RESULTS AND DISCUSSION

CeO_2 nanoparticles are synthesized by precipitation method which is a chemical process. The chemical reactions which occur during the formation of cerium oxide nanoparticles follow in two steps:

Step-1: Precipitation of cerium hydroxide from cerium nitrate

$$8\ Ce(NO_3)_3 + 22\ NH_4OH + 7\ H_2O \longrightarrow 8\ Ce(OH)_4 + 23\ NH_4NO_3$$

Step-2: Conversion of amorphous cerium hydroxide to cerium oxide on heating

$$Ce(OH)_4 \xrightarrow{\Delta} CeO_2 + 2\ H_2O$$

For the preparation of surfactant-modified CeO_2 nanoparticles, a suitable surfactant, viz., dodecenyl succinic anhydride (DDSA, $C_{16}H_{28}O_3$, light yellow liquid) is mixed with heptane solution and CeO_2 nanoparticles. The SEM and EDX images of the synthesized CeO_2 nanoparticles are shown in Figure 1. The size varies from 20 to 80 nm and the average particle size is found to be 34.86 nm.

Tribological experiments have been conducted as per the design matrix (by varying input parameters) of DOE and the corresponding results (average COF and average SWR) are analysed using ANOVA. To ascertain the model accuracy, the following tests are performed: ANOVA analysis, normality test (Figure 2) and regression analysis for COF and SWR.

Since the Box-Cox power transformation plot does not suggest a power transform for this model, and the developed statistical quadratic equations for COF and SWR are given by equations (1) and (2), respectively for paraffin oil nano-lubricants.

$$COF=0.13+(7.33E\text{-}5\times L)-(1.42E\text{-}4\times N)-(3.24E\text{-}4\times T)-(0.11\times C)-(1.7E\text{-}4\times L\times C)+(3.85E\text{-}7\times N^2)+(2.71E\text{-}6\times T^2)+(0.117\times C^2) \quad (1)$$

$SWR = 6.443E\text{-}6+(8.0E\text{-}10\times L)+(2.278E\text{-}9\times T)-(1.153E\text{-}$

$6\times C)+(1.083E\text{-}6\times C^2)$ (2)

Where L = Load (N), N = Speed (rpm), T = Temperature (°C) and C = Concentration (%)

Parametric variations of COF and SWR in terms of the significant input variables are graphically represented in Figures 3 and 4, for paraffin oil nano-lubricants.

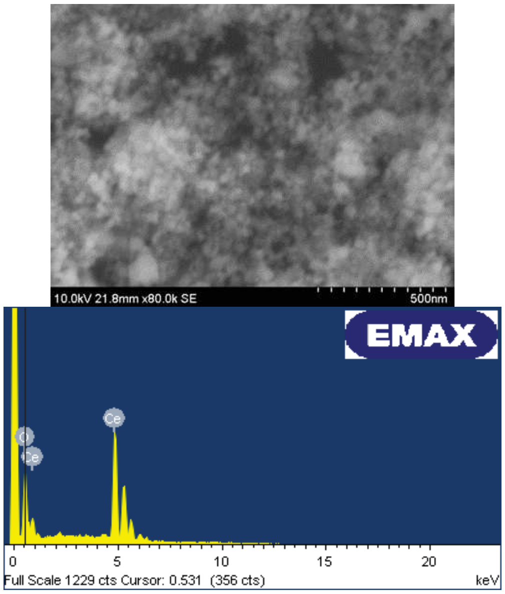

Figure 1 SEM and EDX images of CeO_2 nanoparticles.

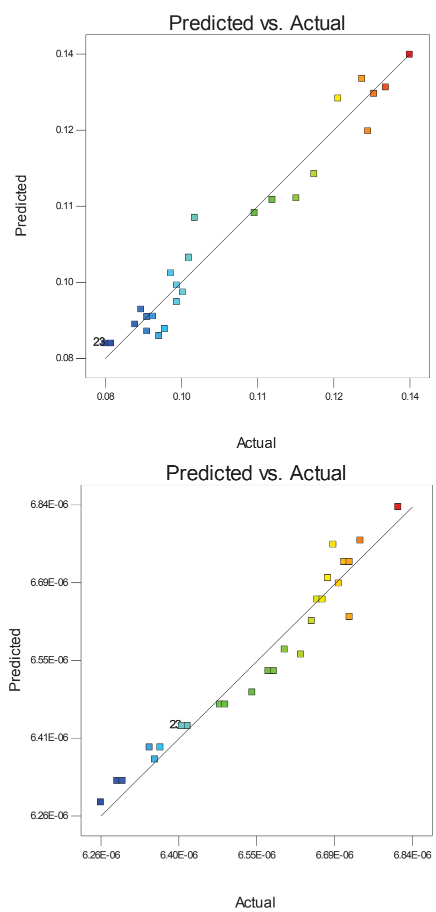

Figure 2 Predicted versus actual plot of COF and SWR for paraffin oil nano-lubricants.

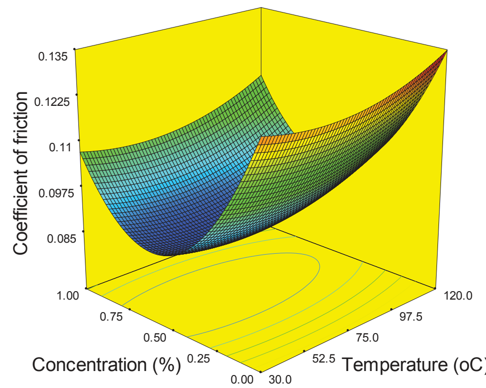

Figure 3 Influence of C and T on COF.

A multi-objective optimization methodology has been formulated and is implemented with the help of desirability function. The optimum concentration of CeO_2 nanoparticles is estimated to be 0.56% with a desirability of 0.924 (maximum value) in paraffin oil.

A viscosity improvement of 38.85% is estimated for paraffin oil lubricant when formulated with surfactant-modified CeO_2 nanoparticles at the optimum concentration level of 0.56% at 75 °C. An improvement of almost 25 °C is attained for flash and fire-points on addition of 0.56% CeO_2 nanoparticles in paraffin oil, restricting its combustibility at higher temperatures. Table 1 shows that maximum zeta potential is obtained for the lubricant containing 30% weight of DDSA in 0.56% CeO_2 nanoparticles. The value of 43.74 mV (after 100 days) indicates a higher zeta potential for 0.56% surfactant-modified CeO_2 added paraffin oil and this reaffirms the edge of using this lubricant in long term stationary applications.

Figure 5 shows the FESEM image and the EDS spectrum of the pin surface after sliding at 75 °C in presence of paraffin oil nano-lubricant with surfactant-modified 0.56% CeO_2 nanoparticles. The EDS spectrum shows clear evidence of the traces of Ce and O at the worn pin surfaces. This indicates the formation of a consistent tribo-film between the mating surfaces.

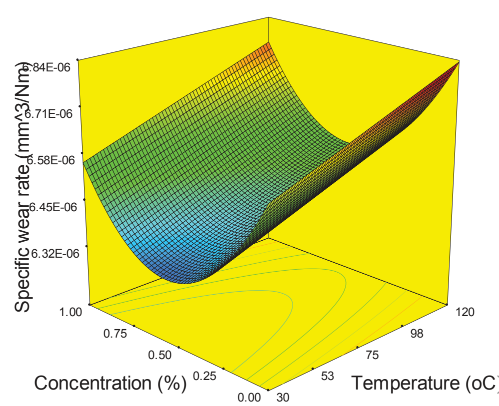

Figure 4 Influence of C and T on SWR.

Table 1 Zeta potential values of the nano-lubricants.

Sample No.	Description	Zeta potential (mV)	
		Immediately after preparation	After 100 days
1	Paraffin + 0.56% CeO_2 nanoparticles	31.25	22.37
2	Paraffin + 0.56% surfactant-modified CeO_2 nanoparticles	44.63	43.74

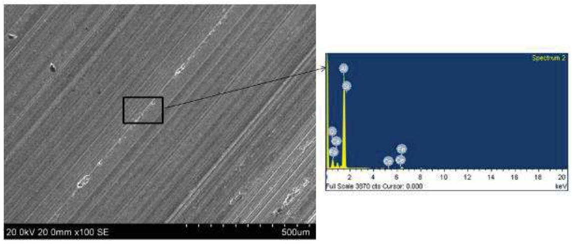

Figure 5 Surface of pin after sliding in presence of paraffin oil with surfactant-modified 0.56% CeO_2 nanoparticles at 75 °C.

4. CONCLUSIONS

The following conclusions are derived from the experimental study:

(a) Spherical CeO_2 nanoparticles are synthesized by precipitation method and the characterization of the nanoparticles is carried out using FESEM and EDS.

(b) CeO_2 nano-lubricants applied at the sliding interface induce less friction and wear rate compared to the base lubricant. Friction and wear continue to improve with increase in concentration of nano-additives in the lubricants till the optimum concentration of 0.56% is reached for paraffin oil nano-lubricants.

(c) Enhancement in thermo-physical properties such as viscosity and flash and fire-point of the nano-lubricant coupled with its tribological properties highlights the potential of nano-lubrication in industrial rotating machines.

(d) FESEM coupled with EDS analysis identifies the possible mechanisms of lubrication.

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