Experimental Investigation and Optimization of Wear Characteristics of Cerium Oxide (CeO_2) Nanoparticle Based Nanolubricant under Boundary Lubrication Condition

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Abstract - *The present work aims to assess the lubricity of Cerium Oxide CeO_2 based nanolubricant by using Pin on Disc (POD) machine under boundary lubrication condition. Commercial diesel engine oil SAE15W40 is used as a base lubricant for the present study. The Design of Experiment (DOE) was carried out by using Taguchi L9 orthogonal array and Response Surface Methodology (RSM) DOE. The input parameters selected for RSM are %concentration of $Ce0_2$, Load and Sliding velocity ranges from 0.2% to 0.4%, 5 to 7N and 2 to 6m/s respectively to evaluate wear rate by weight loss method. RSM showed better results compared Taguchi. The optimum conditions obtained from RSM are 0.3111%conc., 5kg load and at Sliding velocity of 6m/s which gives the optimum wear rate. Nanolubricants with 0.3111% concentration of CeO2 nanoparticle shows significant reduction in wear rate compared to other variants at low load and high sliding velocity. SEM images of worn surfaces of pin were taken after experiment at different concentrations of CeO2 and compared with base lubricant SAE15W40 at optimum conditions.*

Key Words: **Nanolubricant, Wear rate, CeO_2 nanoparticle, Pin on Disc Tribometer, Taguchi,RSM.**

1. INTRODUCTION

About 15% of the total energy loss that takes place in the automobile engine is due to friction between its moving parts or components. And this is one of the major losses and has direct effects on durability and efficiency of engine [1].The relative motion between the parts in contact causes wear and tear of materials. In order to reduce friction and wear, different functional lubricants are used based upon the mechanical systems.

The concept of Nanotechnology is considered as the most revolutionary technique of 21st century. Due to good chemical and physical properties of nanoparticles, it has attracted a lot in interest for the researchers. Nanolubricants are the lubricant obtained by adding nanoparticle at a certain concentration in the lubricant. Researchers reported that adding nanoparticles at certain concentration is effective in reducing the friction and wear [2-6].

Qie et al.[7] investigated effect on tribological properties of WS_2 nanoparticles in paraffin oil under mixed lubrication condition on pin on disc tribometer. He suggested that three mechanisms contribute to friction reduction: a) Rolling action due to spherical shape of nanoparticle additives, 2) Spacers which eliminates the metal to metal contact and 3) Third body material transfer. A thin film of layer was formed on the surface of the pin.

Li et al[8] discussed about the enhancement effect of nanoparticle on wear mechanism.Fig-1.showed how nanoparticle acts as spacer between two metal bodies thereby acting as a third body. Results showed that rolling ability of nanoparticles greatly reduced the friction force and wear.

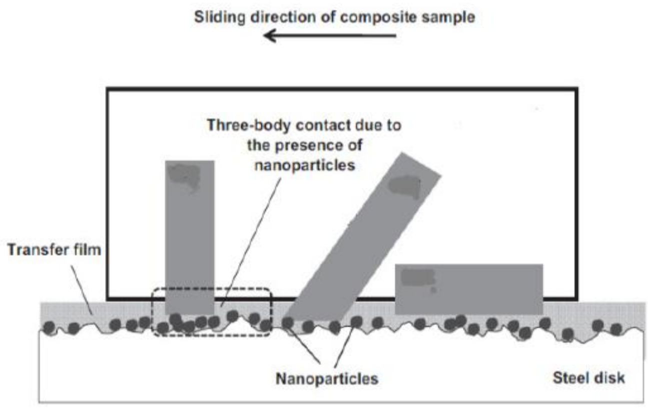

Fig-1: Nanoparticles acts as a spacer[8]

The main objective of the present study is to emphasise on use of Cerium oxide nanomaterial as an engine oil additive. Further aims to investigate wear behaviour of CeO_2 based nanolubricants and to ensure its capability at different operating conditions.

2. EXPERIMENTAL DETAILS

In the present study Cerium oxide nanoparticle is used as an Engine Oil Additive. Cerium Oxide CeO_2 nanoparticle was purchased from Nano Research Lab, Jharkhand, India. The CeO_2 nanoparticles have good properties of wear resistance, chemical erosion resistance and good polishing effect as abrasive [5].The properties of Cerium Oxide nanoparticles is given in Table-1.The lubricating oil used is commercial Diesel Engine oil SAE15W40.

Table-1: Properties of Cerium Oxide Nanoparticles

Average Particle size (APS)	20-30 nm
Surface area	40-45 m2/g
Color	Light yellow
Bulk Density	1.3 g/cm3
True Density	6.5 g/cm3
Crystallographic Structure	Spherical

The pin material to be tested on Pin on disc Machine is Phosphor Bronze against EN31- steel plate hardened to 60HRc. Phosphor Bronze is a copper alloy and has its applications in Journal Bearings of automotive parts. The chemical composition of the material is given in Table-2.

Table-2: Chemical Composition of Material

Composition	Cu	Sn	Pb	P	Ni	Zn
Weight %	94.24	2.43	2.08	0.12	0.20	0.9

2.1: Preparation of Pin

The Phosphor Bronze rod of 16mm diameter and 750mm length was purchased from the market. The size of pin selected was 12mm diameter and 30 mm length. So diameter of rod was reduced to 12mm by manually operating at lathe machine. After the turning operation, the surfaces was polished using polish papers. Pin samples were made as per ASTM standard G99 to conduct test on Pin on Disc Machine.

2.2: Preparation of Nanolubricants

Nanoparticle to be added in lubricating oil was 0.2%wt, 0.3%wt, 0.4%wt.The right quantity of nanoparticle was measured using precision electronic balance having least count of 0.0001 gm. Samples were made of 2gm,3gm and 4gm. Dodecenyl succinic anhydride and Tween 20 were used as a surfactant. Surfactants are used to reduce the settling trend of nanoparticle and for prolonged use of nanolubricant. Surfactant are mixed with nanoparticle in the ratio of 1:1 to form a paste and then mixed with lubricating oil of 1000ml for each sample. The mixer is then kept in Ultrasonicator for 1 hour at 40kHz to vibrate and disperse the particle adequately. Immediately after that the sample goes to Overhead Mechanical stirrer for 2 hours at high speed of 1300-1400rpm and heating mantle was used to heat at 85^0 C temperature to get well mixed and get suspended for longer period of time [5]. After the mixer is agitated, nanolubricant is formed.

2.3: Pin on Disc Test

Computerised Pin on Disc Machine(TR-20LE-PTM) was used to carry out wear analysis of nanolubricants.

Fig-2: Overhead Mechanical Stirrer.

Input Parameters selected are mentioned below.

Table-3: Input parameters with their values.

Input Parameters	Levels of Parameters		
	Level 1	Level 2	Level 3
%Conc. of CeO2	0.2	0.3	0.4
Load(Kg)	5	6	7
Sliding Velocity(m/s)	2	4	6

All the tests were carried out for the duration of 20mins each. The experimentation was carried out as per ASTM Standard. Loads were applied on the pin by pulley string arrangement having counterweights.

Fig-3: Pin on Disc machine Setup

Lubricant was applied between the pin and disc surface in such a way that boundary lubrication condition is achieved. Wear rate was calculated by weight loss method. Three readings for each runs were recorded and the averages of these readings were considered to ensure accuracy. After every run the pin and disc were cleaned by acetone to remove any dirt or wear debris.

Wear Rate Calculated by the formula

Wear rate = (Δm*(10^3))/ρLF (mm^3/N-m)

Where, Δm is the weight loss in grams.

P is the Density of material in gm/cc

L is the sliding distance in meter

F is the load in kg

2.4: Design of Experiments

Design of Experiments for number of test runs and analysis is done on computer software MINITAB-17.Two different techniques are used for Design of Experiments.

2.4.1: Taguchi DOE

Taguchi DOE minimizes the number of data by recording data. The experiments were carried out for 3 parameters each at 3 levels so L9 orthogonal array is used with total degree of freedom of 8.

Table-4: Experimental Data from Taguchi L9 orthogonal array

Exp No	%conc.	Load (Kg)	Sliding Velocity (m/s)	Wear Rate (mm^3/Nm) x10^{-6}
1	0.2	5	2	5.7592
2	0.2	6	4	6.3990
3	0.2	7	6	5.7134
4	0.3	5	4	4.3194
5	0.3	6	6	4.7993
6	0.3	7	2	5.5672
7	0.4	5	6	4.7993
8	0.4	6	2	6.1431
9	0.4	7	4	6.4310

2.4.2: Response Surface Methodology (RSM) DOE

Box-Behnken design of RSM is used to study the effect of individual input parameters and their interactions on the wear rate. The Low and high values of input parameters are given as factors,

%conc.- 0.2 and 0.4

Load(kg) – 5 and 7

Sliding Velocity (m/s)- 2 and 6 ,respectively.

Table-5: Experimental Data from RSM DOE

Exp No	%conc.	Load (Kg)	Sliding Velocity(m/s)	Wear Rate (mm^3/Nm) x 10^{-6}
1	0.4	7	4	6.4310
2	0.3	7	6	5.1014
3	0.3	7	2	5.5672
4	0.2	7	4	5.7134
5	0.2	5	4	5.5641
6	0.2	6	6	5.9139
7	0.4	6	2	6.1431
8	0.3	5	6	4.6201
9	0.2	6	2	6.1625
10	0.4	5	4	5.6931
11	0.3	6	4	4.7993
12	0.3	6	4	4.7993
13	0.4	6	6	5.2993
14	0.3	5	4	4.5194
15	0.3	6	4	4.7993

3. RESULTS AND DISCUSSION

3.1: Taguchi Optimization

Taguchi method uses response variations by means of S/N ratio, because it results in minimization of quality characteristic variation, due to uncontrollable parameters. Wear Rate using concept of Smaller the better is considered. [15]

$$S/N = -10\log_{10}\sum\frac{y2}{n}$$

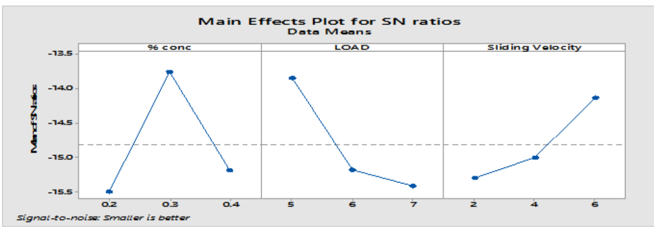

Fig-4: Effect of various parameters on S/N Ratio for Wear Rate

From the main effects plot of S/N ratios for wear rate (Fig-4)we get the optimum levels for % conc. is level2 (0.3%), for Load is level 1 (5kg) and for Sliding velocity is level 3 (6m/s).

3.1.1: ANOVA analysis for Taguchi DOE

The testing (Statistical) of the model which comprises of interaction is done by ANOVA (Analysis of Variance) with F-value to get interaction between input parameters between input and output factors.

Table-6: ANOVA analysis of Taguchi DOE

Factors	DoF	Sum of Square	Mean of sum of Square	F-value	% Contribution
% conc.	2	5.1888	2.5944	14.99	43.29
Load	2	4.2403	2.1201	12.25	35.38
SV	2	2.2092	1.1046	6.38	18.43
Residual Error	2	0.3462	0.1731		2.89
Total	8	11.9844			100

The result from analysis shows that %concentration of CeO2 is the most influencing parameter with 43.29% contribution followed by Load (35.38%) and Sliding velocity (18.43%).

The Generalized Wear Rate equation obtained from Regression Analysis is

Wear Rate = 3.68 - 0.83 % conc + 0.472 Load - 0.180 SV

The optimum parameters obtained from Taguchi Optimization are: %conc. 0.3%, Load 5kg, Sliding Velocity 6m/s. By putting these values of optimum parameters in above regression equation we get the optimum wear value as 4.711 X 10^{-6} mm^3/Nm.

3.2: RSM Optimization

RSM is a mathematical and statistical technique which is used to analyse the output response which is affected by input factors. Based upon the experimental values obtained, the following 2nd order I-O interaction is appropriate.

$$R = \beta_0 + \sum_{i=1}^{4}\beta_i I_i + \sum_{i=1}^{4}\beta_{ii} I_i^2 + \sum_{i=1}^{k-1}\sum_{j\geq 1}^{k}\beta_{ij} I_i I_j$$

Where, R is Response, I_i, I_j are variables, β_o is the intercept coefficient, β_i is the ith linearity coefficient, β_{ii} is the coefficient of quadratic, β_{ij} is the coefficient of interaction (linear by linear) [13].

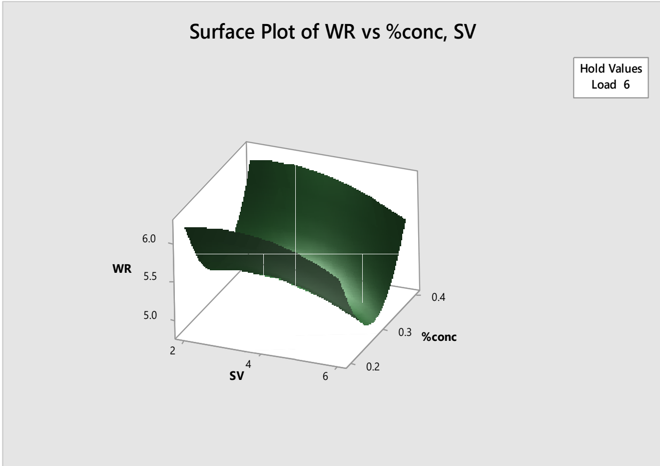

Fig-5a: Surface plot of Wear Rate Vs %conc., sliding velocity

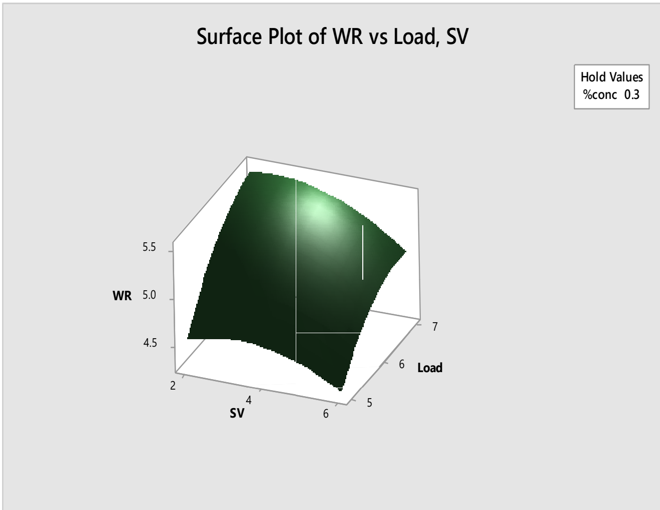

Fig-5b: Surface plot of Wear Rate Vs Load, sliding velocity

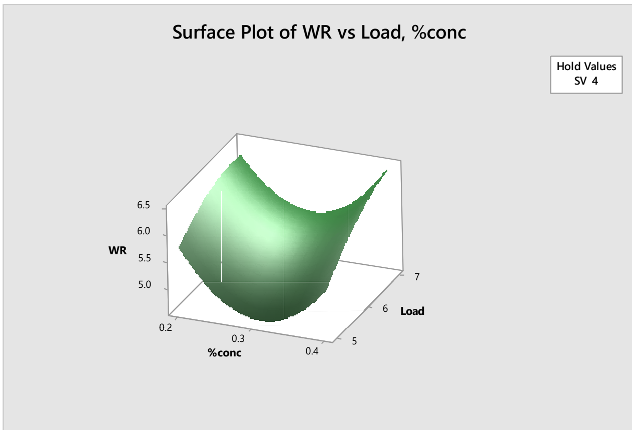

Fig-5c: Surface plot of Wear Rate Vs Load, %conc.

Fig-5(a-c) shows surface plot of wear rate for various combination of factors. The curvilinear surface of graph will always give the optimum solution in RSM technique. To get the Minimum values of Response, response optimizer function is used and the optimum values for Minimum wear rate is deduced from Fig-6.

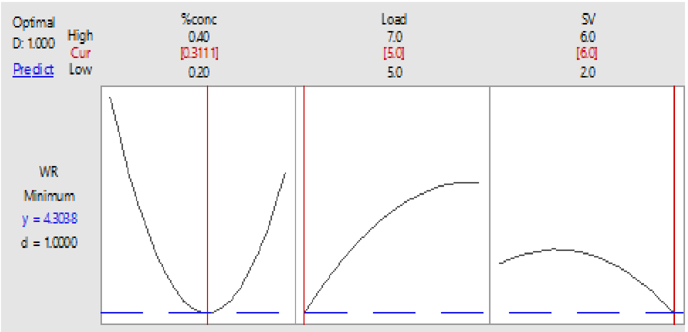

Fig-6: Response optimizer graph with response value of wear rate in Red Color

From Fig-6, the optimum value of input parameter for optimum output response of wear rate by RSM technique is 0.3111% conc., 5 kg load and 6 m/s sliding velocity.

3.2.1: ANOVA Analysis for RSM

Table-7: ANOVA for RSM

Factors	DoF	Sum of Square	Mean of SS	F-value	% Contribution
% conc	2	2.5307	1.26535	13.21	41.18
Load	2	1.9970	0.9985	10.81	32.50
SV	2	1.2055	0.60275	6.28	19.61
Error	8	0.4123	0.20615		6.71
Total	14	6.1447			100

The result shows that the most effecting parameter is %conc. with 41.18% contribution followed by Load (32.50) and Sliding velocity(19.61).

The Generalized wear rate equation obtained from regression analysis is

Wear Rate = 4.61 - 0.73 %conc + 0.309 Load - 0.1915 SV

The optimum values of input parameters obtained from RSM technique are 0.3111% Conc., Load 5kg, Sliding Velocity 6m/s.

By putting the values of optimum parameters in above regression equation we get the optimum wear value as 4.7789 X 10^{-6} mm^3/Nm

3.3: Confirmation Test

Optimization Method	Optimum Parameter	Response Wear rate x10^{-6} mm^3/Nm.		Error (%)
		Experimental value	Predicted value	
Taguchi	Conc.(0.3%) Load(5Kg) Sliding Velocity(6m/s)	4.9314	4.711	4.67
RSM	Conc.(0.3111 %) Load(5Kg) Sliding Velocity(6m/s)	4.8520	4.7789	1.53

Both the optimization techniques showed almost similar results but the error in confirmation test of RSM is less compared to Taguchi .This may be due to more number of experiments taken in RSM to get better fit Thereby selecting the results obtained from RSM Optimization as the optimum results.

The results obtained at low load shows that third body rolling action of spherical nanoparticle may have happened which leads to less direct metal to metal and at high load, the load itself gets prominent and increases the wear rate of material.

By increasing concentration of CeO_2 nanoparticles in the oil, the wear rate decreases and attains a value of 0.3111 and then increases. At high concentration, the presence of more nanomaterial might be the reason of increase in wear rate.

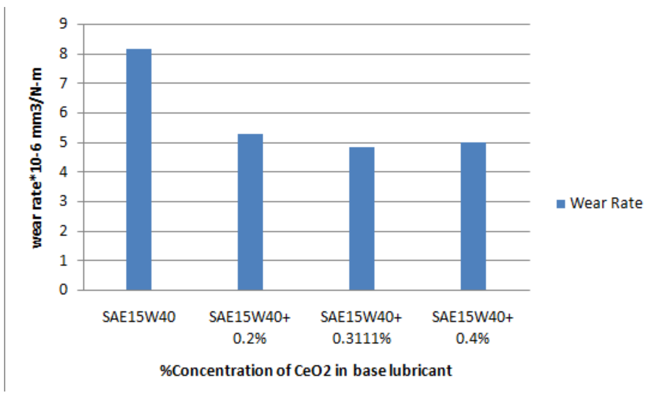

Chart-1: Wear rate at various concentrations at 5kg Load and 6m/s Sliding Velocity

3.4 Worn Surface Analysis

SEM images of worn surfaces of pin were taken at different concentrations of nanolubricants, at 5kg load and sliding velocity of 6m/s.

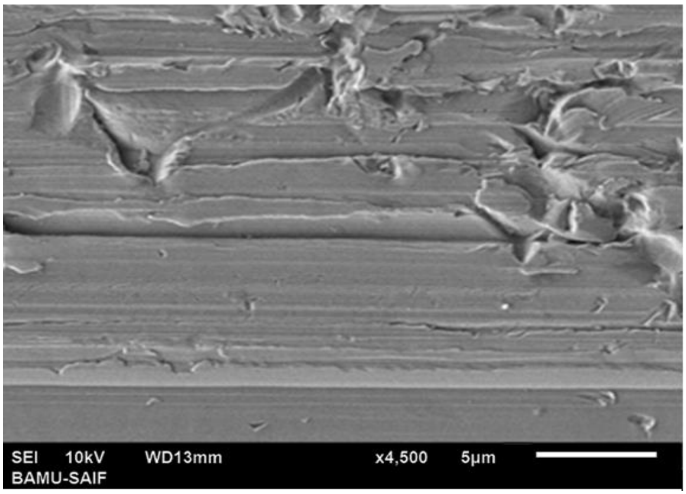

Fig-7a: SEM images of surface of pin with Base oil SAE15W40 at 5kg load and sliding velocity of 6m/s.

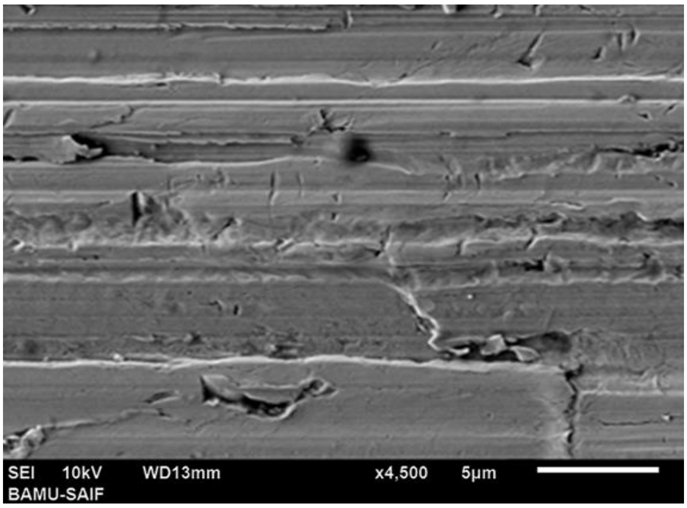

Fig-7b: SEM images of surface of pin with SAE15W40+0.2% CeO_2 at 5kg load and SV of 6m/s.

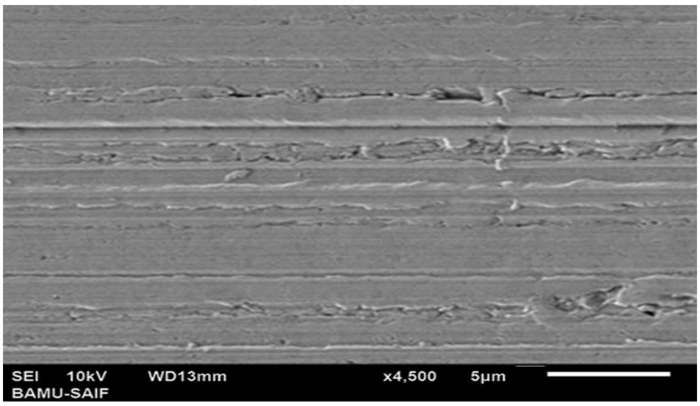

Fig-7c: SEM images of surface of pin with SAE15W40+0.3.111% CeO_2 at 5kg load and sliding velocity of 6m/s.

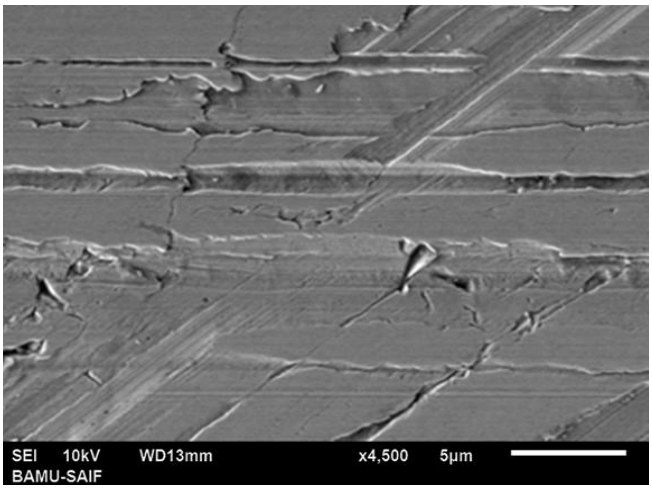

Fig-7d: SEM images of surface of pin with SAE15W40+0.4% CeO_2 at 5kg load and sliding velocity of 6m/s.

Fig-7 a) shows uniform grooves, scars and wear as there is direct contact between the mating surfaces. Whereas lubricants containing nanoparticles fig 7.b-d) shows less wear and scars indicating spherical nanoparticles reducing wear significantly.

Fig 7.c shows that at optimum concentration (0.3111%), the wear scar is smooth.

4. CONCLUSIONS

From the present experimental investigation, the following conclusions are drawn:

- Taguchi optimization and Response surface methodology (RSM) are compared and the latter showed closer values to actual experimental values.
- RSM optimization shows that the optimal combination of input parameters for %conc., load and Sliding velocity are 0.3111%, 5Kg, 6m/s respectively.
- ANOVA analysis showed %concentration of CeO_2 is most influencing parameter with 41.18% contribution and other influencing parameters of load and sliding velocity with 32.50% and 19.61% respectively.
- SEM images of worn surfaces of pin after wear test showed that wear scars on surfaces used with base oil are more compared to lubricants containing nanoparticles.
- Nanolubricants with 0.3111 concentration of CeO_2 nanoparticle shows significant reduction in wear rate compared to other variants at low load and high sliding velocity.

Addition of CeO_2 nanoparticles in the lubricating oil enhances the anti wear properties of oil which shows that CeO_2 nanoparticle can be used to as engine oil additives.

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