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ORIGINAL ARTICLE

Experimental correlation between varying processing properties and wear behaviour of ternary Ni-Co-SiO_2 composites coating of mild steel

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Abstract Experimental correlation between varying processing and wear behaviour of ternary Ni-Co-SiO_2 composites coating was investigated. The parameter used in this research are: SiO_2 (5–25 wt%), thermal treatment (100–300 °C), applied load (5–15 N). The results show that novel ternary Ni-Co-SiO_2 nanoparticle composite coating was successful applied to mild steel. The addition SiO_2 nanoparticles in the coating Ni-Co bath lead to uniform microstructure. Thermal treatment of the coating at 300 °C decreased wear rate by (−0.031), increasing the wt% of SiO_2 from 0 to 25 decreased the wear rate by −0.018, applied load increases from 5 to 15 N raises the wear rate raises (0.0097), The lower wear rate was obtained at 25 wt% SiO_2, applied load 5 N and thermal treatment at 300 °C. Validation of the results from pin on disc test with electro-hydraulic servo PV friction testing machine shows the same wear pattern. One can concluded in this work that the wear rate of the coated materials depend on the made up of the coating and not on the type of wear mechanism. It have be established in this work that thermal treatment and SiO_2 nanoparticle can be used to enhance the wear behaviour of Ni-Co coating of mild steel.

Keywords Wear · Load · Composites coating · Microstructure and Ni-Co-SiO_2

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1 Introduction

Nickel plating is similar to other electroplating processes that employ soluble metal anodes. It requires the passage of direct current between two electrodes that are immersed in a conductive, aqueous solution of nickel salts [1, 2]. The flow of direct current causes one of the electrodes (the anode) to dissolve and the other electrode (the cathode) to become covered with nickel [3]. The nickel in solution is present in the form of divalent positively charged ions (Ni^{++}). When current flows, the positive ions react with two electrons ($2e-$) and are converted to metallic nickel (Ni) at the cathode surface [4, 5]. The reverse occurs at the anode, where metallic nickel is dissolved to form divalent positively charged ions, which enter the solution. The nickel ions discharged at the cathode are replenished by those formed at the anode [6].

Many binary alloy of nickel are currently used in industrial scale, among them is Ni-Co alloy coating. Ni-Co coating have great attention due to it good corrosion resistance, strength and wear [7, 8]. Many researchers have pay great attention to the development of Ni-Co ternary coating that will withstand harsh and high temperature condition. Among the research are: Gajendra et al.[9] reported on the deposition of Ni-Co-SiC composite coating on mild steel substrate, using nickel alloyed with cobalt as the binder phase with SiC as dispersed particles, Morphological studies of Ni-Co-SiC coating were carried out with scanning electron microscopy and X-ray diffraction analysis to correlate the mechanical and corrosion behaviour of the coating, Eliaz et al.[10] reported on the electrodeposition of Zn–Ni, Zn–Co and Zn–Ni–Co coatings on mild steel from an acidic chloride bath containing p-aminobenzenesulphonic acid (SA) and gelatin, Wang et al.[11] reported on the microstructure and properties of sol-enhanced Ni-Co-TiO_2 nano-composite coatings on mild steel, Idris et al.[12] reported on nanocrystalline Ni-Co alloy synthesis

by high speed electrodeposition. Nanocrystalline Ni-Co alloys coatings were prepared by direct current (DC) and deposited directly on steel and aluminium substrates without any pre-treatment, using high speed electrodeposition method. Based on the above literature the study of the wear of ternary composites coating of mild steel with Ni-Co-SiO_2 have not be recorded. Hence, this present work is to study the effect of processing parameter on the wear behaviour of ternary Ni-Co-SiO_2 composites coating.

2 Materials and method

2.1 Materials

A flat plate mild steel (200 mm × 200 mm) substrate is used in this research. The chemical composition of the substrate (mild steel) is described in Table 1.

Other materials employed for the purpose of this work include pure nickel anode, nickel chloride, potassium chloride, boric acid, thiourea, 2 dimethylaminoethanol, cobalt and silicon dioxide, cobalt and silicon oxide powders were used.

2.2 Method

Mild steel plate was sectioned using automatic struers high precision cut-off machine which is connected to lubricant supply to cool the blade and sample during the cutting process. The mild steel plate was cut into equal plates of dimensions of 20 mm by 20 mm. The steel was grinded and polished using grit papers and degreased in ethanol, then treated in 0.5 mol of HCl. An innovative electro-deposition condition to produce Ni-Co and Ni-Co-SiO_2 composites coating was developed on mild the steel. The bath was formulated using Table 2.

Deionised water was used to prepare the plating solution. The pH of the bath was adjusted to 4.5 by adding HCl and NaOH. Before co-deposition, the SiO_2 particulates of a mean diameter 55 nm were distributed in the bath electrolyte in the presence of other additives. The deposition setup test was achieved on a set up vessel connected to a laboratory rectifier. The bath was stirred by a magnetic stirrer consistently with about 200 rpm at 40 °C. The experiments were conducted at current density of 1 mA cm^{-2}, applied potential of 0.3–0.6 V and time of 20 min (see Table 3). The thermal stability of the coated composite alloy was verified in a muffle furnace at temperatures of 100, 200 and 300 °C for 4 h. JOEL JSM 5900LV scanning electron microscope equipped with an Oxford INCA^{TM} energy dispersive spectroscopy system was used to determine the morphologies of the composite coatings.

For the wear test the samples were cut to 10 mm × 10 mm and all weighed before the test. Dry sand wear abrasive rig test uses a rubber-rimmed wheel as the bed for silica abrasive that is fed from a hopper by a nozzle between the sample and the wheel. The sample was inserted in sample holder and tightened to avoid falling of sample. The test was run for a set 200 revolutions with automatic stop. The sample was pressed into the wheel by a 5 N weight loaded lever and 15 N after the first test is done. The wear is measured by measuring the volume of material lost through mass loss and density measurements on mass scale and recorded the value, from the mass before and after reduction shows the mass loss of the surface specimen.

Table 1 Chemical composition of mild steel used (wt%)

Element	C	Mn	Si	P	S	Al	Ni	Fe
Composition	0.15	0.45	0.18	0.01	0.031	0.005	0.008	Balance

Table 2 Bath composition of Ni-Co-SiO_2 alloy co-deposition matrix

Composition	Mass concentration g/L
Nickel chloride	100
Potassium chloride	30
Boric acid	10
Thiourea	5
2 Dimethylaminoethanol	5
Silicon dioxide	10_25

3 Results and discussion

Figure 1a, shown the transmission electron microscopy analysis of the SiO_2 nanoparticles. From Fig. 1a it was observed that the average particles size is 55 nm. The particles were very fine without agglomeration.

Figure 1b showed the SEM/EDS image of the mild used in this work. It was observed that the SEM microstructure show the ferrite phase (white) and cementite phase (dark). It was

Table 3 Formulated designed bath composition of Ni-Co-SiO_2

Sample Order	Matrix sample	Time of deposition (min)	Current (A/cm^2)
1	Ni-10Co	20	1.0
2	Ni-10Co-10SiO_2	20	1.0
3	Ni-10Co-15SiO_2	20	1.0
4	Ni-10Co-25SiO_2	20	1.0

observed that the ferrite phase is more than the cementite phase in Fig. 1b and from the EDS also supports the SEM observation that the steel contain high α-Fe phase. The EDS analysis clearly show that Fe, C, Mn, Si are present in the microstructure this supported the composition analysis of the steel in Table 1.

Figure 2 showed the SEM/EDS of the mild steel after coating, it can be seen clearly that there is a morphological change of the microstructure after coating. By comparing Fig. 1b with Fig. 2 it can be seen that the surface of the mild steel is cover with hard layer of Ni-Co-SiO_2. Figure 2a show the SEM/EDS of the Ni-10Co deposition on mild steel, while Fig. 2b show the SEM/EDS of Ni-10Co-25SiO_2. It can be observed that the surface of coating have no porosity and defects. The surface of Fig. 2b is more uniform distribute than Fig. 2a. The uniformly distribution of the Fig. 2b may be attributed of the fine SiO_2 nanoparticle used in the deposition this help to increases the average surface area of particle during deposition. From the EDS of Fig. 2 it can be seen that there is evidence of Ni, Co, Fe, C (Fig. 2a) and Ni, Co, Fe, O, Si (Fig. 2b) which is difference from the EDS of Fig. 1b.

Figure 3 showed the thermal treatment of the composites coating in order seen the thermal stability of composite coating as the temperature increase from room temperature. It can be observed that after the thermal treatment there is a great difference between in the composites coating by comparing Fig. 3 with Fig. 2. In Fig. 3a the thermal treatment of Ni-10Co composite coating the structure turn into coarse hard Ni-10Co coating on the mild steel surface but in Fig. 3b, the thermal treatment of Ni-Co-25SiO_2 composite coating the presence of Si aid in the precipitation of the various phase in the microstructure into fine uniformly precipitate.

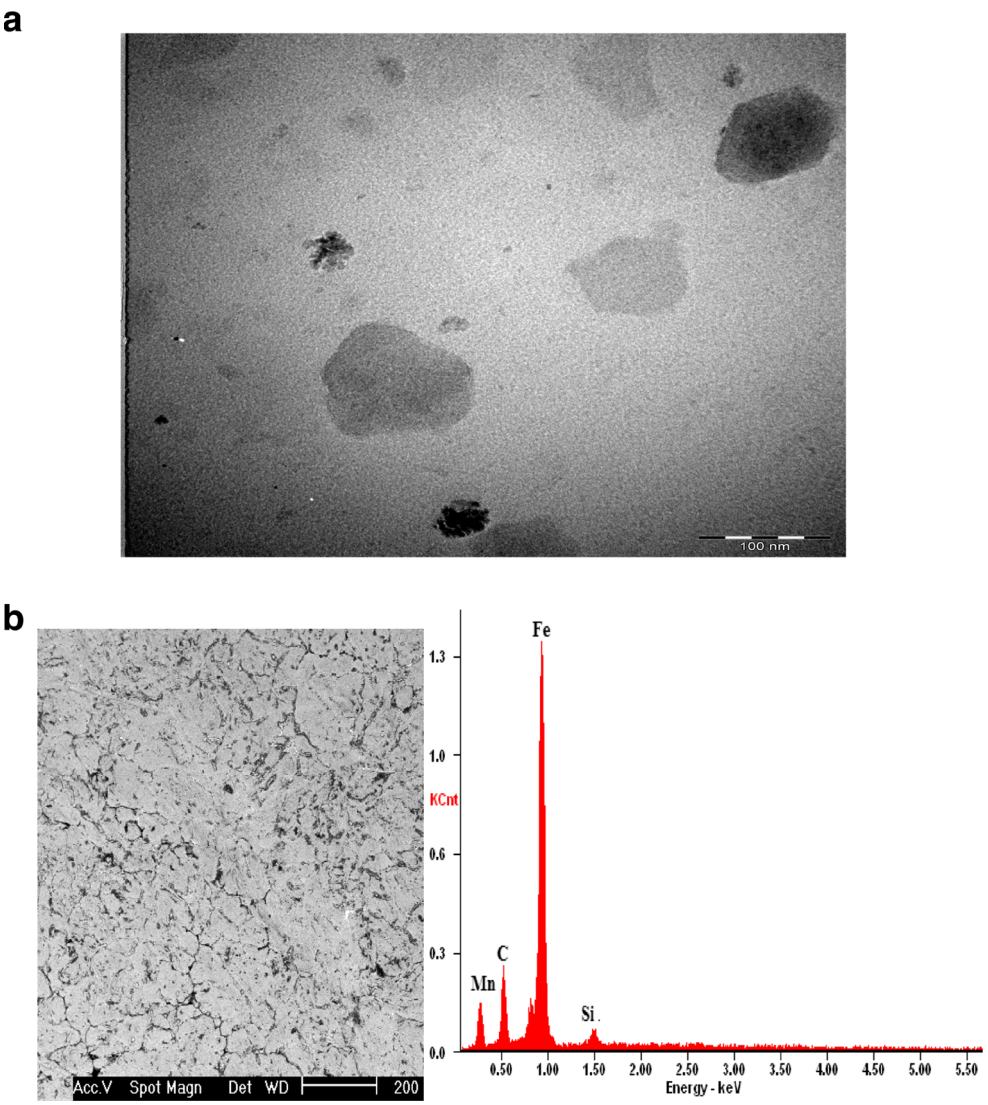

Fig. 1 **a** TEM image of the SiO_2 nanoparticles. **b** SEM/EDS image of the mild steel before coating

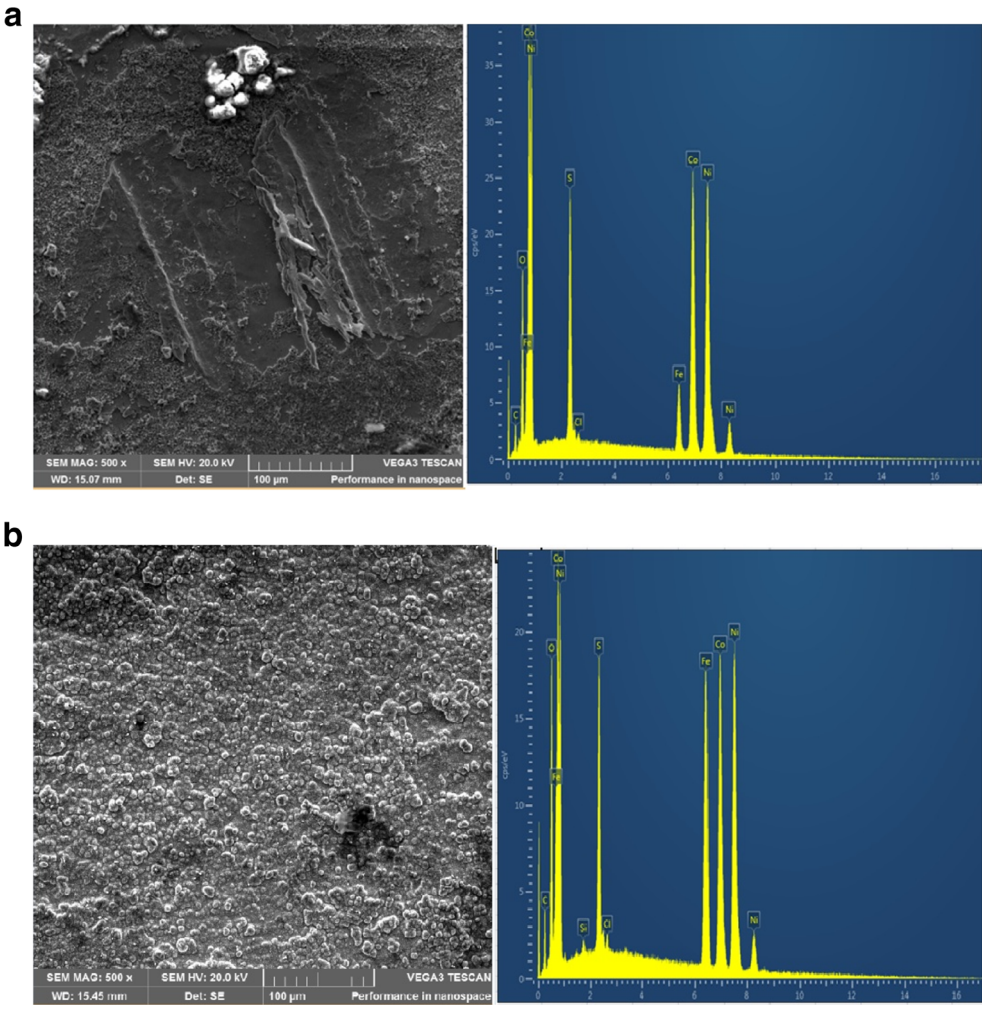

Fig. 2 **a** SEM/EDS OF Ni-10Co coating. **b** SEM/EDS OF Ni-Co-25SiO_2

The suitability of the developed composites coating at various composition was determined using wear test. Figure 4 showed the wear rate of the composites coating. It was observed that the wear rate of the composites coating is better than that of the mild steel for example at room temperature (37 °C) and applied load of 5 N, the wear rate of 0.012, 0.008, 0.005, 0.003, 0.002 g/m were obtained for mild steel (control), Ni-10Co, Ni-Co-$x$$SiO_2$ (x = 10, 15, 25 wt%), respectively (see Fig. 4a). The mean that 83.33 % wear resistance was achieved at Ni-Co-25SiO_2. This improvement in wear resistance after composites coating could be attributed to the hard and fine structure obtained after coating, this help in strengthening the composite coating and increase the load bearing capacity of the composites coating in respect to wear. The better wear resistance obtained when SiO_2 was added than Ni-Co coating was attributed to the fine structure obtained in Fig. 2b than Fig. 2a, which lead to decreases in dislocation movement and harder the surface.

The wear rate of the composites coating rises with increases in the applied load from 5 to 15 N. By comparing Fig. 4a with Fig. 4b it can be seen that at all composition the wear rate of 5 N is lower than at 15 N, increase in load raise the contact between the sample and the disc and also increases the contact pressure which resulted to higher wear. The wear rate of the materials is best at thermal treatment of 300 °C, than 100 and 200 °C. Decreases in wear resistance at a lower thermal treatment could be attributed to under ageing of the composites coating surface which lead to few precipitate. Increase in wear resistance of the thermal ageing of the composites coating at 300 °C than the untreated was attributed to the fine precipitate form which help to strain harder the composites coating than the untreated composites coating.

A full factorial design of experiments of the type P^n was used in the study the wear behaviour, where n corresponds to the number of factors and P represents the number of levels. Here, i.e., n corresponds to the number of factors (applied load, wt% SiO_2 and materials condition) and p the number of levels (P = 2) (upper and lower levels of each variable). For the modelling of the upper level and the lower level of each variable along with their coded values was used in this research (see Table 4). The design of the experiments and the values of respond variables, corresponding to each set of trial,

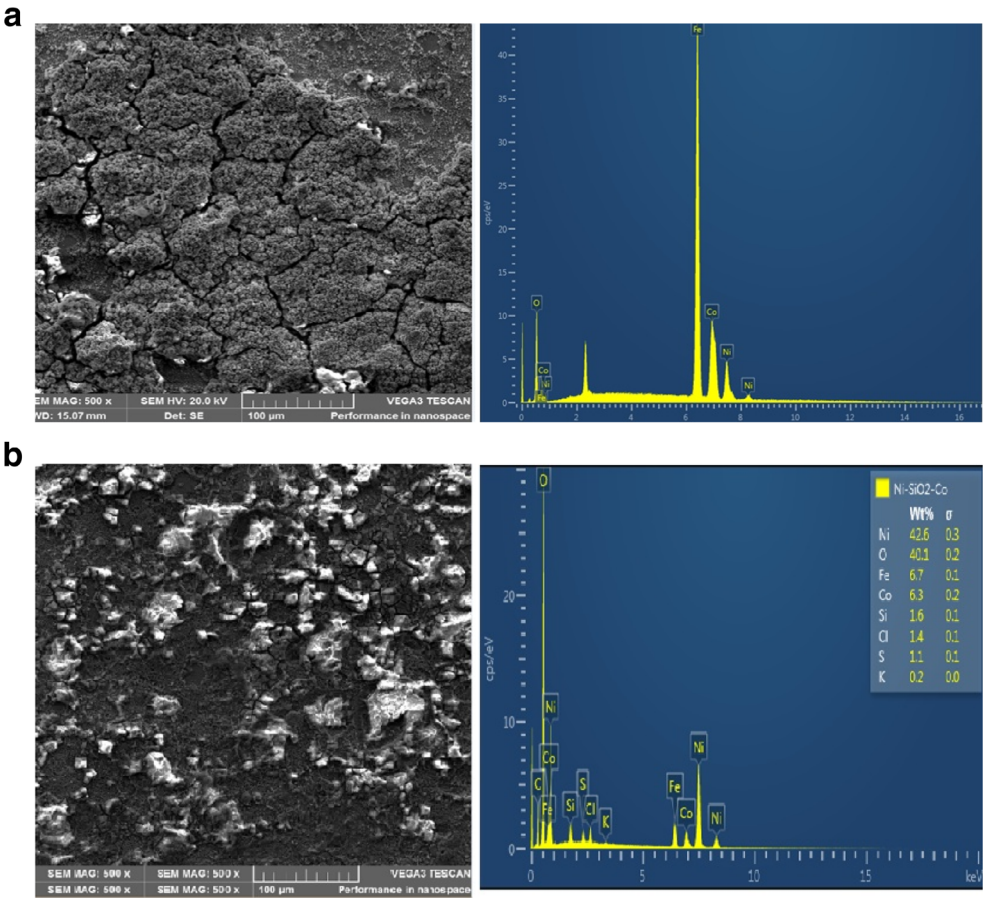

Fig. 3 **a** Thermal effect of SEM/EDS OF Ni-10Co at 300 °C. **b** Thermal effect of SEM/EDS OF Ni-Co-25SiO_2 at 300 °C

are reported in Table 5. The respond variables, in each trial, represent the average of three measured data at identical experimental conditions.

From Table 5, it can be seen that thermal treatment (A) of the coating at 300 °C decreased wear rate by (−0.031), increasing the wt% of SiO_2 (B) from 0 to 25 decreased THE wear rate by (−0.018), when the applied load increases from 5 to 15 N (C) the wear rate raises by (0.0097), also increasing the three factors (ABC) decreased the wear rate by (−00019). This can be seen clearly in Fig. 5a–c. From Fig. 5a–c it was observed that using the coding values by change the sample condition, wt% SiO_2 from −1 to +1 the wear rate decreases, also by increasing the applied load from −1 to +1 the wear rate increases. This is used to support the earlier observation in Fig. 4. However, the estimated response surface have the lower wear rate at 25 wt% SiO_2, applied load 5 N and thermal treatment at 300 °C (see Fig. 5a–c).

Analysis of variance (ANOVA) was further used to study the factors that have great influences on the wear rate of the composite coating. From Table 6, it can be seen that Model *F* value of 29.36 implies the model is significant. There is only a 3.33 % chance that a "Model *F* value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case B (wt% SiO_2), C (applied load), BC (wt% SiO_2 and applied load) are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. The "Pred R-Squared" of 0.8849 is in reasonable agreement with the "Adj R-Squared" of 0.9530 with standard deviation of 2.652E-003 and mean of 0.011. The developed model equation is showed in Eq. 1.

$$\begin{aligned}\text{Wear rate} = {} & +0.011 - 1.525\text{E}{-}003 * A - 9.150\text{E}{-}003 * B \\ & + 4.825\text{E}{-}003 * C \\ & + 1.100\text{E}003 * A * B - 4.300\text{E}{-}003 * B * C \end{aligned} \quad (1)$$

Table 4 Upper and lower levels of each factor along with their coded values

S/No	Variables	Upper level	Lower level
A	Materials condition	treated (+1)	untreated (−1)
B	SiO_2 (wt%)	25 (+)	0 (−1)
C	Applied load (N)	15 (+1)	5 (−1)

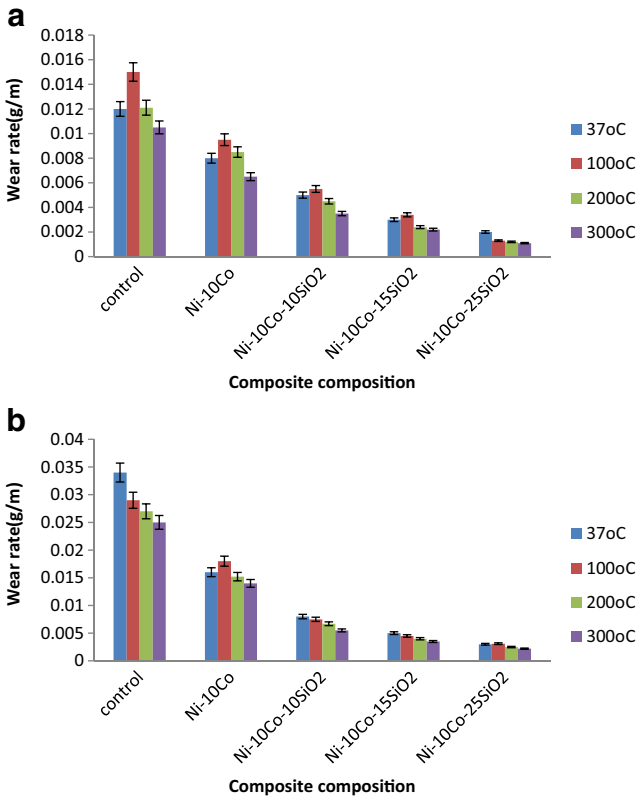

Fig. 4 **a** Wear rate of the composites coating at 5 N. **b** Wear rate of the composites coating at 15 N

From Eq. 1, it can be observed that the coefficients of A is (−0.001525), B (−0.00915) and C (0.004825). Since the coefficients of A, B is negative, means increasing wt% SiO2, thermal treatment decreased the wear rate, while the coefficient of C is positive, means increasing the applied load increases the wear rate. Confirmation experiments were conducted for eight different set of conditions. The actual values and the predicted values obtained from the regression model were compared (see Fig. 5d). The percentage of error was calculated using Eq. (1) for the validation of the regression model.

$$\%\,\text{of error} = (\text{Actual value} - \text{Predicted value})/\text{Actual value} \times 100\% \quad (2)$$

The averages absolute error obtained for the wear rate is 0.47, which means that a better accuracy was obtained using the developed regression models.

The wear results from pin on disc was later validated with an electro-hydraulic servo PV friction testing machine with a cast iron axis rotating on a sliding brushing to since the behaviour of the materials under direction wear condition. The PV unit has a linear velocity and normal pressure under which materials will fails. It is most critical in most operating and manufacturing conditions. The test was conducted accordance to GB7948-1987 of P.R. A load of 2 MPa per 1 h and velocity of 2 m/s was used until failure occurred. The speed, normal load, temperature and friction torque were taken from the PC attached to the machine. The counter pressure was recorded by $P = \frac{F_N}{d_l}$ where F_N is the normal load, d and l = thickness and length of sample. Since the samples that were thermal treatment at 300 °C have the lowest wear rate from the pin on disc test was used for the validation of the work. The specific wear rate was calculated with equation below:

$V_S = {}^{\Delta m}/_{\rho} F_N L(mm^3/Nm)$, where: V is the relative sliding velocity, F_N = normal load, L = total sliding distance and ρ is density of the material. The results of the specific wear rate are shows in Fig. 6a. From Fig. 6a, it was observed that the results still showed that same pattern from the results obtained from Pin on disc test, which shows that sample with 25 wt% SiO_2 has the lowest wear rate of all of them. This can

Table 5 Expanded plan matrix for factorial design

S/n	x_0	A	B	C	AB	AC	BC	ABC	Strength (N/mm^2) CS (N/mm^2) Wear rate (g/m)
1	1	−1	−1	−1	+1	+1	+1	−1	0.012
2	1	1	−1	−1	−1	−1	+1	+1	0.0105
3	1	−1	1	−1	−1	1	−1	+1	0.002
4	1	+1	+1	−1	+1	−1	−1	−1	0.0011
5	1	−1	−1	+1	+1	−1	−1	+1	0.034
6	1	+1	−1	+1	−1	+1	−1	−1	0.025
7	1	−1	+1	+1	−1	−1	+1	−1	0.003
8	1	1	1	1	1	1	1	1	0.0022
Effects	0.011	−0.0031	−0.018	0.0097	0.0022	−0.0019	−0.086	−0.0019	

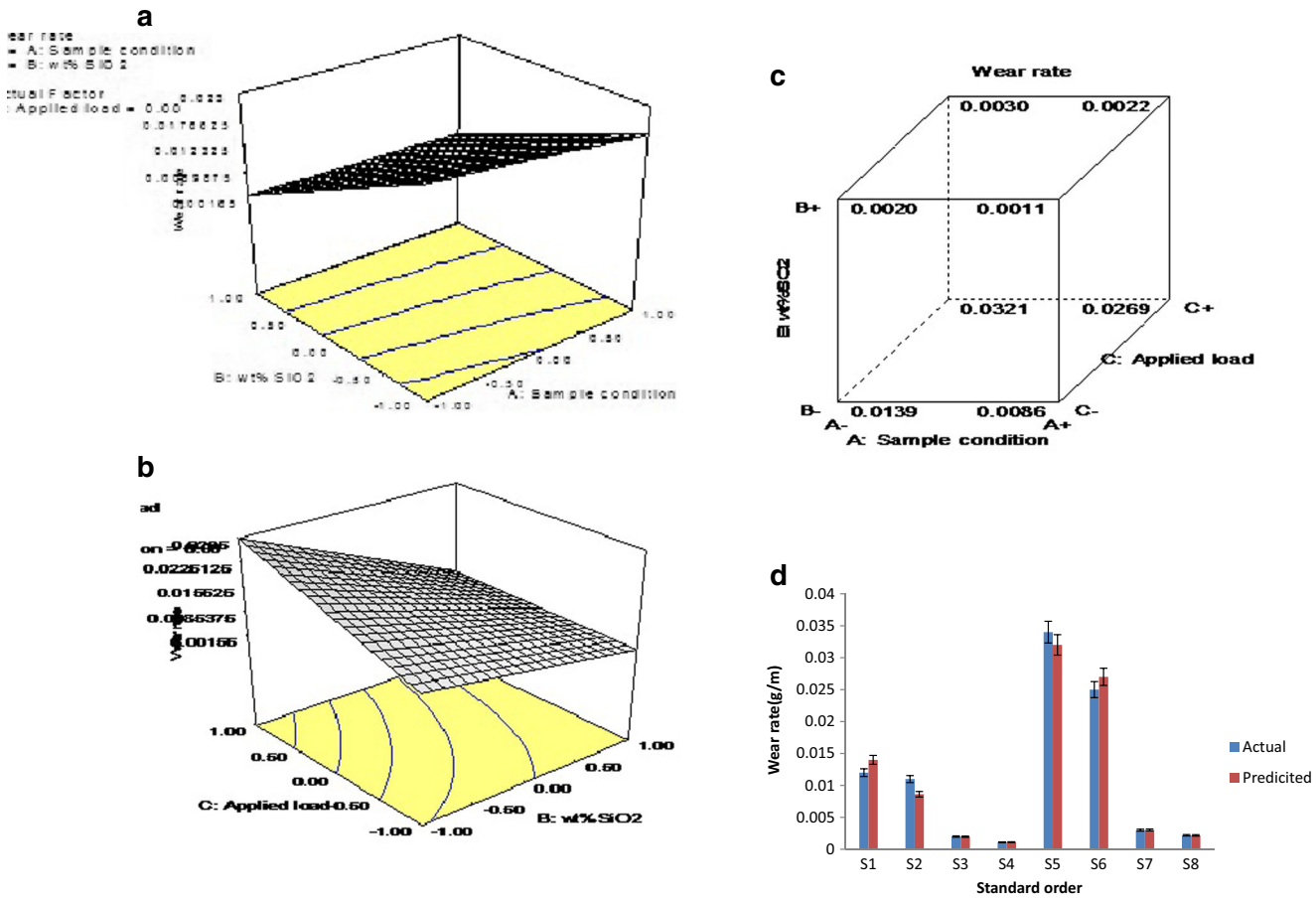

Fig. 5 **a** 3D plot of wear rate against sample condition and wt% SiO_2. **b** 3D plot of wear rate against applied load and wt% SiO_2. **c** Cube plot of wear rate against applied load, wt% SiO_2 and sample condition. **d** Variation of wear rate with standard order

be confirmed from the PV limit (LPV) (see Fig. 6b). The 25 wt% SiO_2 sample has the highest LPV, mean it have the lowest wear rate. The LPV is the product of the normal pressure and linear velocity when the sample fails. One can concluded in this work that the wear rate of the coated materials depend on the made up of the coating and not on the type of wear mechanism used.

4 Conclusions

From the results and discussion above the following conclusions can be made:

1. Ternary Ni-Co-SiO_2 nanoparticle composite coating was successful applied to mild steel

Table 6 ANOVA for selected factorial model

		Wear rate				
Source	Sum of Squares	DF	Mean square	*F* value	*P* value	Remarks
Model	1.032E-003	5	2.064E-004	29.36	0.0333	Significant
A	1.861E-005	1	1.861E-005	2.65	0.2454	Not significant
B	6.698E-004	1	6.698E-004	95.24	0.0103	Significant
C	1.862E-004	1	1.862E-004	26.48	0.0357	Significant
AB	9.680E-006	1	9.680E-006	1.38	0.3615	Not significant
AC	1.479E-004	1	1.479E-004	21.03	0.0444	Significant
Residual	1.406E-005	2	7.032E-006			
CorTotal	1.406E-005	7				

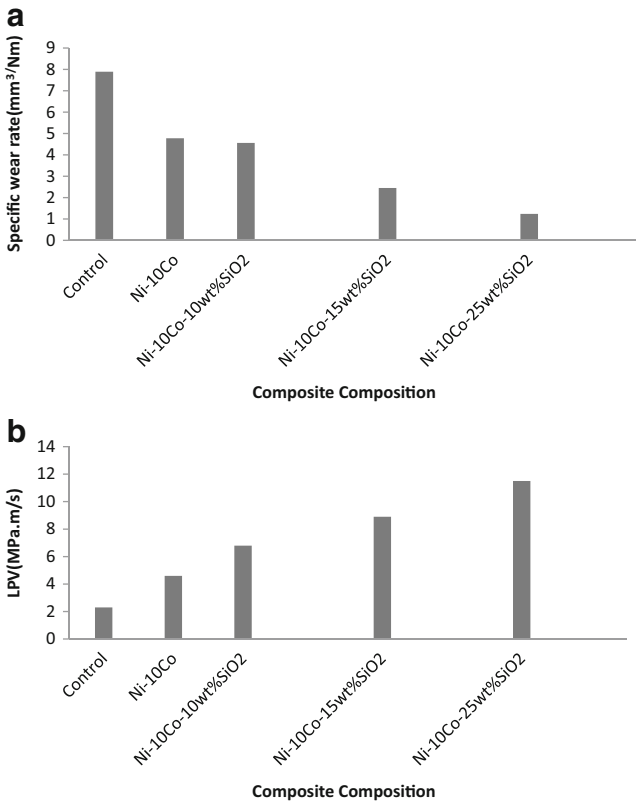

Fig. 6 **a** Variation of specific wear rate with composite composition. **b** Variation of LPV with composite composition

2. The addition SiO_2 nanoparticles in the coating Ni-Co bath lead to uniform microstructure.
3. Thermal treatment of the coating at 300 °C decreased wear rate by (−0.031), increasing the wt% of SiO_2 from 0 to 25 decreased the wear rate by (−0.018), applied load increases from 5 to 15 N raises the wear rate raises (0.0097)
4. The lower wear rate was obtained at 25 wt% SiO_2, applied load 5 N and thermal treatment at 300 °C
5. Validation of the results from pin on disc test with electro-hydraulic servo PV friction testing machine shows the same wear pattern.
6. One can concluded in this work that the wear rate of the coated materials depend on the made up of the coating and not on the type of wear mechanism.
7. It have be established in this work that thermal treatment and SiO_2 nanoparticle can be used to enhance the wear behaviour of Ni-Co composites coating of mild steel.

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