Experimental Investigation of Corrosion Properties of Mild Carbon Steel C45 Zn-Phosphating and Passivation

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Abstract-
This paper reports an investigated to evaluate the corrosive resisted characteristics of Zinc Phosphating of low carbon steel C45. Process parameters considered for the study are Zn concentration, temperature and time. The influence of the phosphate processing parameters on the corrosion resistance of zinc phosphate conversion coatings and the optimization of the process are investigated using the Taguchi method. The pieces are tested by salt spray method, and the results are analysis the quality of the oil seals was improved. In Taguchi method L9 orthogonal array has been selected. The analysis of variance (ANOVA) has been used to determine effect of each parameter on Corrosion Resistance (C.R.)

Keywords- Taguchi Method, Signal to Noise (S/N) Ratio, Optimization, Process Parameters, C.R.

I. INTRODUCTION

Phosphating is a conversion coating treatment largely used in many industries as a surface preparation for coating by paints and to increase corrosion resistance. Phosphating is the metal pre-treatment process for the surface treatment and finishing of ferrous and non-ferrous metals. Due to its economy, speed of operation and ability to afford excellent corrosion resistance, wear resistance, adhesion and lubricate properties, it plays a significant role in the automobile, process and appliance industries. Phosphate coatings serves as a conversion coating in which a dilute solution of phosphoric acid and phosphate salts is applied via spraying or immersion and chemically reacts with the surface of the part being coated to form a layer of insoluble, crystalline phosphates. During the process of Phosphating, problem is overheating of the bath solution, which causes an early conversion of the primary phosphate to tertiary phosphate before the metal has been treated which results in an increase in the free acidity of the bath and consequently delays the precipitation of the phosphate coating. The low temperature Phosphating processes have become more significant today due to the escalating energy costs. However, low temperature Phosphating processes are very slow and need to be accelerated by some means. Acceleration of the Phosphating process could be achieved by chemical, mechanical and electrochemical methods. Phosphate conversion coatings can also be used on aluminum, zinc, cadmium, silver and tin. Phosphate coatings are often used to provide corrosion resistance, however, phosphate coatings on their own do not provide this because the coating is porous. Therefore, oil or other sealers are used to achieve corrosion resistance.

II. EXPERIMENTATION

A. Methodology of Experiment

There are several optimization techniques to develop product, process or operation. Various techniques can be applied to optimize phosphating process. Sometimes different techniques are required integrate to get statistically significant results, which can lead to better conclusions and recommendations. Some extensively used methods in developing a process or a product are Build Test Fix (BTF), Design of Experiment (DOE) and One Variable at a Time (OVAT), BTF is very primitive and unorganized approach. It is iterative method of developing a process focused on improvement from last experiment. DOE is highly efficient method of investigating the effect of parameters as it varies multiple parameters at once. As more parameters are investigated, more number of new combinations is required. DOE cannot control individual parameters and more relies on statistical data. In one variable at a time (OVAT) approach, variation is done with one variable at a time and other parameters are kept constant until the effect of one parameter is studied.

It is highly precise method to study effect of each parameter at different levels. Zn concentration, temperature and time were identified as most predominant parameters affecting the phosphate process. Based on the observation, Taguchi method has been used to optimize the process parameters. OVAT analysis has been conducted to find out effective range of parameters for optimization study. L9 orthogonal array (OA) has been selected from available designs. Standard notation for OA is given below

OA = Ln (Xm)

Where n= number of experiments, X= number of levels and m= number of parameters under study. From available designs for 3 levels 3 parameters, OA with least number of experiments required to conduct (L9) has been selected. ANOVA has been conducted to find out contribution of each parameter in the output. Minitab 19 software has been used for analysis.

B. Experimental Machine Selection

The finer the salt spray particles, the larger the surface area formed, and the more oxygen is adsorbed, the stronger the corrosively. Conventional spray methods include a gas jet method and a spray tower method. The artificial simulated salt spray environment test uses a test equipment with a certain volume space, the Salt Spray Testing Chamber, to artificially use the salt spray environment in the volume space to assess the salt spray corrosion resistance quality of the product. Compared with the natural environment, the salt concentration of the chloride in the salt spray environment can be several times or several times that of the salt spray content of the general natural environment, so that the corrosion rate is greatly improved, and the product is subjected to a Salt Spray Testing Chamber to obtain a result. The time is also greatly shortened. If a product sample is tested in a natural exposure environment, it may take 1 year to corrode, and in a simulated simulated. salt spray environment, as long as 24 hours, similar results can be obtained. The artificial simulated salt spray test includes a neutral Salt Spray Testing Chamber, an acetate salt spray test, a copper salt accelerated acetate spray test, and an alternating salt spray test. Neutral salt spray test (NSS test) is one of the earliest applications in the field of accelerated corrosion test. It uses a 5%aqueous solution of sodium chloride solution, and the pH of the solution is adjusted to a neutral range (6-7) as a solution for spraying. The test temperature is taken at 35 ° C, and the sedimentation rate of the salt spray is required to be between 1 and 2 ml / 80 $cm^2 \cdot h$.

***Figure 1**. Complete arrangement of experimental setup*

C. Selection of material

C45 is a medium carbon steel grade offering reasonable tensile strength. This grade can be flame or induction hardened to produce a good surface hardness with moderate wears resistance, C45 grade steel is a medium carbon steel offering moderate tensile strengths. The material is capable of through hardening by quenching and tempering on limited sections but can also be flame or induction hardened to HRC 55. Chemical composition of Sisal material is shown in Table 1

***Table 1** Chemical Composition of Sisal material.*

Compound	Value	Compound	Value
Si	0.40	**B**	0.0009
Mn	0.65	**Be**	0.0001
Cr	0.4	**Ca**	0.0005
Ni	0.4	**Cd**	0.0008
Zn	0.5	**P**	0.045
Ti	0.005	**Fe**	88.07

***Figure 2**. Mild Carbon steel C45 Material*

III. RESULTS AND DISCUSSION

To get complete understanding of effects of input parameters Zn concentration, temperature and time on output C.R., you usually assess signal to noise ratio or main effects plot for means. For this purpose, Minitab 19 statistical software has been used. Modeling of C.R. has been done. ANOVA has been conducted to find out effect of each parameter on the C.R. and linear regression model has been established to predict the values of C.R..

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A. Experimental Result

Table 2 shows the L9 orthogonal array with measurement of C.R. for runs one to nine. It also shows S/N ratio for all nine experiments.

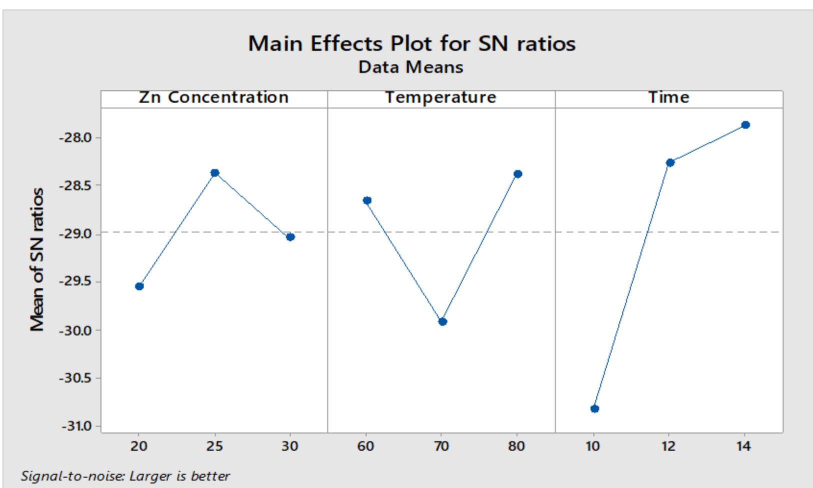
Table 2 *L9 orthogonal array with response characteristic.*

Experiments	Inputs Factors			Output Responses	
Trial No.	**Time**	**Temperature**	**Zn Concentration**	**C.R.**	**SNRA**
1	20	60	10	0.0282	-30.9950
2	20	70	12	0.0333	-29.5511
3	20	80	14	0.0394	-28.0901
4	25	60	12	0.0418	-27.5765
5	25	70	14	0.0393	-28.1121
6	25	80	10	0.0339	-29.3960
7	30	60	14	0.0426	-27.4118
8	30	70	10	0.0249	-32.0760
9	30	80	12	0.0415	-27.6390

The S/N ratio values are calculated with help of Minitab 19 software. It can be seen that variation in S/N ratio is minimum for all experiment.

B. Main Effects of C.R.

Graph 1 shows the main effects plot from S/N ratios.



Graph 1. *Main Effects Plot for S/N Ratio*

From the graph, it is observed that the optimum C.R. was in the higher values of the in the response graph. The optimal input parameters were Zn Concentration 25% (level 2), Temperature 80^{o}C (level 3) and Time 14 minutes (level 3). The graph graphically shows the effect of the control factors on Low carbon steel C45 material.

C. ANOVA Result

ANOVA, the ratio between the variance of the cutting parameter and the error variance is called Fisher's ratio (F). It is used to determine whether the parameter has a significant effect on the quality characteristic by comparing the F test value of the parameter with the standard F table value at the P significance level. If the F test value is greater than P test the cutting parameter is considered significant. Relevance of the models is tested by analysis of variance (ANOVA). It is a statistical tool for testing the null hypothesis for planned experiments, in which several different variables are studied simultaneously. ANOVA is used to quickly analyze the variances in the experiment using the Fisher test (F test). ANOVA table shown the result of the ANOVA analysis. ANOVA analysis makes it possible to observe that the value of P is less than 0.05 in the three parametric sources. It is therefore clear that Zn concentration, temperature and time of the material have an influence on the low carbon steel C45. The last column of cumulative ANOVA showed the percentage of each factor in the total variance that indicates the degree of impact on the outcome. Table 3 shows results obtained from analysis of variance

Table 3 *ANOVA Result.*

Source	DF	Adj SS	Adj MS	F-Value	P-Value	% Contribution
Zn Concentration	2	0.000033	0.000033	13.21	0.070	10.12
Temperature	2	0.000059	0.000059	23.40	0.041	18.09
Time	2	0.000231	0.000231	91.24	0.011	70.85
Residual Error	2	0.000003	0.000003			
Total	8	0.000326				

ANOVA is a statistical tool used in several ways to develop and confirm explanations for the observed data , the relevance of the models is tested by analysis of variance (ANOVA). It shows that the ANOVA of MS C45 material. The table shows that the Zn Concentration (10.12%), the temperature (18.09%) and the time (70.85%) have a major influence on C.R.

D. Development of Regression Model for C.R.

Regression model has been developed using Minitab software. Substituting the experimental values of the parameters in regression equation, values for C.R. have been predicted for all levels of study parameters. Graphical representation also shows that a predicted and experimental value of M.C. correlates with each other.

Regression Equation –

C.R. = -0.0075 + 0.000270 [Zn Concentration] + 0.000037 [Temp] + 0.002858 [Time]

Table number 4 gives comparison between experimentally measured and predicted moisture content by developed mathematical equation

Table 4 *Experimental and Predicted Values of C.R.*

Sr. No.	Experimental value	Predicted value	Error %
1	0.0282	0.0287	1.74
2	0.0333	0.0347	4.03
3	0.0394	0.0408	3.43
4	0.0388	0.0357	8.68
5	0.0393	0.0418	5.98
6	0.0336	0.0307	9.44
7	0.0426	0.0428	0.46
8	0.0279	0.0317	9.87
9	0.0415	0.0378	9.78

Difference between C.R. values calculated using regression equation and experimental values for each experience found less than 10%. Hence, we can say that the regression equation is valid.

E. Confirmation Experiment Result

Table 5 shows the difference between value of C.R. of confirmation experiment and value predicted from regression model developed.

Table 5 *Confirmation Experiment Result*

Parameter	Model value	Experimental value	Error %
C.R.	0.0422	0.0441	4.50

Confirmation experiment is conducted by keeping parameters at optimum levels suggested by Taguchi method and the C.R. value obtained has been compared with value predicted by the regression model keeping the parameters at same levels. It can be seen that the difference between experimental result and the predicted result is 4.50%. This indicates that the experimental value correlates to the estimated value.

IV. CONCLUSIONS

This paper investigates the implementation of Taguchi design in the estimation of optimum minimum corrosion resistance of mild-steel in cooling tower that uses saline solution of different concentration and different working parameters. It had been concluded that Taguchi design prepare a useful methodology for the setup and optimization of corrosion resistance with minimum numbers of trials in comparison to other experimental design

- The optimal solution obtained for C.R. based on the combination of Phosphating and passivation parameters and their levels is (i.e. Zn Concentration 12% at level 2, temperature 80^{o}C mm/min at level 3 and time 14min at level 3. The time more significant Parameters than Zn Concentration and temperature
- ANOVA results indicate that time plays prominent role in determining the C.R. The contribution of Zn Concentration, temperature, time to the quality characteristics C.R. is 10.12 %, 18.09% and 70.18 % respectively.
- The optimal process parameters are determined using Taguchi methods match with the experimental values by minimum errors i.e 4.50% .
- Through the developed mathematical models, any experimental results of C.R. with any combination of Phosphating and Passivation parameters can be estimated.

V. REFERENCES

1) I. B. Obot, "Adsorption behavior of glucosamine-based, pyrimidine-fused heterocycles as green corrosion inhibitors for mild steel: experimental and theoretical studies," e Journal of Physical Chemistry C, vol. 120, pp. 11598–11611, 2016.

2) F. M. Alkhrafi, A. M. El-Shamy, and B. G. Ateya, "Comparative effect of tolytrialzole and benzotriazole against sulfideattack on copper," International Journal of Electrochemical Science, vol. 4, pp. 1351–1364, 2019.
3) M. Alkhrafi, A. M. El-Shamy, and B. G. Ateya, "Comparative effect of tolytrialzole and benzotriazole against sulfideattack on copper," International Journal of Electrochemical Science, vol. 4, pp. 1351–1364, 2019.
4) C. Verma, H. Lgaz, D. K. Verma, E. E. Ebenso, I. Bahadur, and M. A. Quraishi, "Molecular dynamics and monte carlo simulations as powerful tools for study of interfacial adsorption behavior of corrosion inhibitors in aqueous phase: a review," Journal of Molecular Liquids, vol. 260, pp. 99–120, 2018.
5) C. Verma, E. E. Ebenso, and M. A. Quraishi, "Ionic liquids as green corrosion inhibitors for industrial metals and alloys," Green Chemistry, IntechOpen Limited, London, UK, 2018.
6) M. E. Hussein, A. S. El Senousy, W. H. Abd-Elsalam et al., "Roselle seed oil and its nano-formulation alleviated oxidative stress, activated Nrf2 and downregulated m-RNA expression genes of pro-inflammatory cytokines in paracetamol-intoxicated rat model," Records of Natural Products, vol. 14, pp. 1–17, 2019.
7) M. F. Shehata, A. M. El-Shamy, K. M. Zohdy, E. S. M. Sherif, and S. Zein El Abedin, "Studies on the antibacterial influence of two ionic liquids and their corrosion inhibition performance," Applied Sciences, vol. 10, pp. 1444–1453, 2020.
8) K. M. Zohdy, A. M. El-Shamy, A. Kalmouch, and E. A. M. Gad, ")e corrosion inhibition of (2Z,2′ Z)-4,4′-(1,2- phenylene bis (azanediyl)) bis (4-oxobut-2-enoic acid) for carbon steel in acidic media using DFT," Egyptian Journal of Petroleum, vol. 28, pp. 355–359, 2019.
9) Y. Morozov, L. M. Calado, R. A. Shakoor et al., "Epoxy coatings modified with a new cerium phosphate inhibitor for smart corrosion protection of steel," Corrosion Science vol. 159, Article ID 108, 2019.
10) M. Yeganeh, N. Asadi, M. Omidi, and M. Mahdavian, "An investigation on the corrosion behavior of the epoxy coating embedded with mesoporous silica nanocontainer loaded by sulfamethazine inhibitor," Progress in Organic Coatings, vol. 128, pp. 75–81, 2019.
11) P. Mahato, S. K. Mishra, M. Murmu, N. C. Murmu, H. Hirani, and P. Banerjee, "A prolonged exposure of Ti-Si-B-C nanocomposite coating in 3.5 wt.% NaCl solution: electrochemical and morphological analysis," Surface and Coatings Technology, vol.375, pp. 477–488, 2019.