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Optimization of electrophoretic deposition of alumina onto steel substrates from its suspension in *iso*-propanol using statistical design of experiments

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Abstract

Statistical design of experiments was used to investigate the effect the process parameters on electrophoretic deposition (EPD) of alumina onto steel substrates from its suspension in *iso*-propanol. The process parameters considered were (i) concentration of particles in the suspension (solid loading), (ii) electrode separation, (iii) applied potential, and (iv) deposition time on the quantity of ceramic particles electrophoretically deposited. A 2^4 full factorial matrix, with four repetitions of the center point, was used to develop the predictive regression equation for deposition of alumina per unit area of the electrode in the design space. The results show that particle concentration has the most dominant effect with more than 50% contribution to the deposited amount. A good correlation was obtained between predicted and experimental values suggesting that the model can predict data accurately in the experimental matrix.

Keywords: A. Ceramics; A. Composites; A. Multilayers; A. Thin films

1. Introduction

Electrophoretic deposition (EPD), a colloidal deposition technique in ceramic processing has recently gained increased application in a variety of field including preparation of thick film of silica [1], nano-size zeolite membrane [2], hydroxyapatite coating on metal substrate for biomedical applications [3,4], luminescent materials [5–7], high-T_c superconducting films [8,9], gas diffusion electrodes and sensors [10,11], multi-layer composites [12], glass and ceramic matrix composites by infiltration of ceramic particles onto fibre fabrics [13], oxide nano-rods [14], carbon nanotube film [15], functionally graded ceramics [16,17], layered ceramics [18], superconductors [19,20], piezoelectric materials [21], etc.

In EPD process, charged powder particles, dispersed stably in a liquid medium are attracted and deposited onto a substrate of opposite charge on application of a dc electric field. The advantages of EPD include simplicity, low cost equipment, good control of deposition thickness, short formation time, little restriction on the shape of the substrate, suitability for mass production, and no requirement for binder burnout as the green coating contains fewer or no

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organics. The driving force for the electrophoretic mobility of the particles is mainly the magnitude of surface charge/zeta potential of the particle in suspension. But there are also other parameters which can be grouped into two broad categories [22–26]: (i) those related to the suspension and substrate and (ii) those related to the process. The parameters related to the suspension and substrate include particle size, dielectric constant of the solvent, conductivity of the suspension, viscosity of the suspension and zeta potential and conductivity of the substrate. The parameters related to the process include particle mass concentration in the suspension, separation between the electrodes, applied voltage, and deposition time. But once the solvent, particle and substrates are fixed, the variable parameters which can be effectively controlled to optimize EPD are the process parameters. In a classical approach, optimization is done by varying one-factor-at-a-time while keeping the others constant. This method consists of successively varying each factor over its range with the other factors are held constant. This experimentation strategy does not provide any idea about the individual contribution of each factor towards the response and fails to consider any interactional effect of two or more variables which is more likely in an actual operating environment.

The statistical optimization technique using full factorial design of experiments is an useful tool which allows one to obtain appropriate data that can be analyzed to arrive at an objective conclusions and determine the optimum conditions through a relatively smaller number of systematic experiments [27]. Using a proper design matrix and systematically varying different variables one can obtain regression equations, which highlights the effect of individual parameters and their relative importance in given operation/process. In the conventional experimentation method of one-factor-at-a-time, only one factor is varied over its range with the other factors held constant. The interaction effect of two or more variables cannot be determined using this approach. The primary advantage of statistical methods is that the interactional effects of two or more variables can also be known. It also adds objectivity to the decision-making process. They allow us to measure the likely error in a conclusion or to attach a level of confidence to a statement. When the problem involves data that are subject to experimental errors, statistical methodology is the only objective approach to analysis. In this paper we present a systematic investigation on the use of statistical design of experiments to optimize and develop quantitative understanding on the effect of process variables on the yield of electrophoretic deposition of alumina on steel substrates and highlight the methodologies and significance of each analysis in arriving at the optimized condition. The effects of individual parameters as well as their interactional effects have also been highlighted.

2. Experimentals

2.1. Materials

The calcined alumina powder (Grade CT 3000SG) used for electrophoretic deposition in the present investigation was supplied by Alcoa, India. The powder had a mean particle size of 0.7 μm, BET surface area of 7.0 m^2/g and sintered density of 3.9 g/cm^3. The organic solvent, propan-2-ol (C_3H_8O), used as the dispersing medium, was supplied by SD Fine Chemicals, Mumbai. The solvent was 99.5% pure with 0.1% residual water content in it. Stainless steel plates (20.5 mm × 20.5 mm × 5 mm) were used as the substrates for electrophoretic deposition. A stainless steel strip of the same dimension was used as the counter electrode. The substrates and the counter electrodes were thoroughly cleaned before use.

2.2. Methods

The suspension for electrophoretic deposition was prepared by dispersing alumina powder in the *iso*-propanol solvent media. The suspension was first magnetically stirred (REMI EQUIPMENTS) at moderate speed for 10 min followed by ultrasonication for 20 min by Vibronic Ultrasonic Processor (Model P2) at 200 V. The surface charge of alumina suspension measured by particle charge detector (PCD-03-pH, Mutek, Germany) was −0.18 C/g. Conductivity of the suspension varied between 1.64 μS at 10% (w/v) particle loading to 3.34 μS for 30% (w/v) particle loading.

Deposition experiments were conducted in a setup (Fig. 1) similar to that used by Besra et al. [28]. It consists of two electrode holders made of Teflon as the principal components. One of the electrode holders is fixed and the other is movable and can slide along two parallel rods at the bottom so that the distance between the electrodes can be adjusted to desirable position. The electrodes are fixed onto the holders such that they face each other. Adequate clearance was provided beneath the setup to accommodate a magnetic bead. Each of the electrode holders has a square window

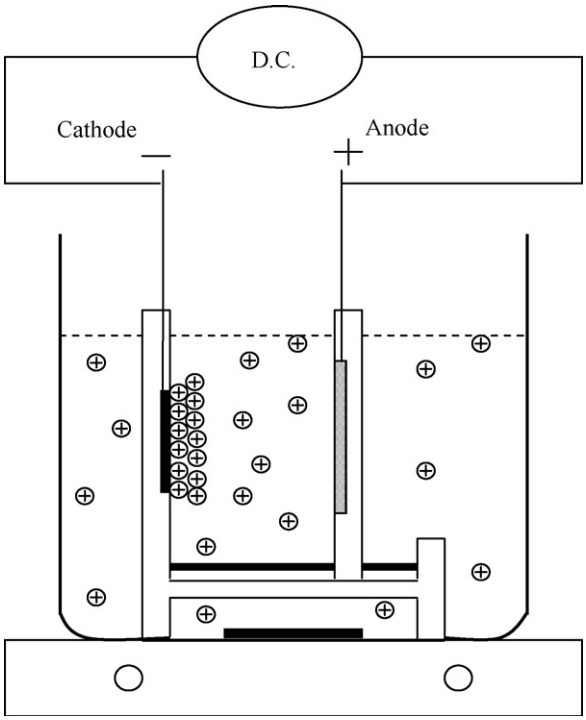

Fig. 1. Electrophoretic deposition setup.

facing each other to fix the electrodes on it. The area of the deposition as well as counter electrode exposed to the suspension was 4 cm^2. The stainless steel substrates were mounted on the holders with a spring contact at the back to act as electrical contact. The holders along with electrodes were dipped into the silica glass reservoir containing the alumina suspension followed by application of desired dc voltage for a prerequisite time. Sedimentation of the alumina particles was prevented by mild stirring using a magnetic bead stirrer as shown in Fig. 1. Constant voltage electrophoretic deposition experiments were carried out at conditions within the statistical design matrix. The negatively charged particles got deposited on the anode. After deposition, the electrodes were carefully taken out and the deposits were allowed to dry at room temperature for 24 h. The deposits along with the substrate were then weighed to determine the yield. The suspension was replenished after every three deposits [29].

2.3. Statistical design and modeling

Factorial designs allow to analyse the effects of several different factors and combine them into a response model. They are the most commonly used statistical designs due to their simplicity with regard to both preparation and analysis of the results. Although primarily used for screening significant factors, they are also used sequentially to model and refine a process. The 2^k design provides the smallest number of runs with which k factors can be studied in a complete factorial design. In a 2^k design, all combinations of k-factors are set at two levels with respect to center point and are evaluated. The two levels are the allowable limits, i.e., the maximum and minimum values set on the basis of preliminary trials. The final relationship that is eventually determined must hold within these limits. The assumptions for making the 2^k design valid include linearity of response over the range of the factors chosen, randomization of the designs and satisfaction of the usual normality assumptions. Perfect linearity, however, is unnecessary and the 2^k system will work quite well even when the linearity assumption holds very approximately [27]. In fact, the addition of interaction terms to the main effects provides a model that is capable of representing some curvature in the response function. The 2^k design augmented with center point replicates is an excellent way to obtain an indication of potential non-linearity or curvature of the response. It allows one to keep the size and complexity of the design low and simultaneously obtain some protection against curvature. Also center points do not impact the usual effects estimate in a 2^k design [27].

2.4. Design matrix

A full 2^4 factorial design with addition of four center point experiments was chosen to model particle deposition Design Expert v.7 statistical software (Stat Ease Inc.) was used for the analysis of the experimental data and for statistical modeling. The four factors investigated were: (i) concentration, (ii) electrode separation, (iii) applied voltage, and (iv) deposition time by A, B, C and D, respectively. Each factor was run at two levels and the intermediate response was assumed to be linear, which is necessary for 2^k designs. The possibility of non-linearity within the design space has been accounted for through the introduction of center points and model augmentation. The center points are essentially used to test for evidence of pure second-order or quadratic effects in the response region of exploration. The high and low levels for each factor as given in Table 1 were chosen on the basis of preliminary trials. These high and low levels are expressed in coded form as −1 and +1, respectively to convert the absolute quantity into a dimensionless quantity making the handling of the experimental data convenient. Also since all variables used in the model are normalized to vary in this way, the relative change of a variable is directly related to the size of its regression coefficient. The weight of the alumina deposit was studied as the response for the different combinations of factor levels. Table 2 shows the experimental matrix in actual and coded factors along with the weight of alumina deposited as response. The regression equation for the matrix is then represented by the following expression:

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_4 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{14}X_1X_4 + b_{23}X_2X_3 + b_{24}X_2X_4 + b_{34}X_3X_4 + b_{123}X_1X_2X_3 + b_{124}X_1X_2X_4 + b_{134}X_1X_3X_4 + b_{234}X_2X_3X_4 + b_{1234}X_1X_2X_3X_4$$

where Y is the response (alumina deposited weight), b_0 a constant, i.e., response at the zero level (center point) experiment, b_1, b_2, b_3 and b_4 the linear coefficients (independent parameters), $b_{12}, b_{13}, b_{14}, b_{23}, b_{24}, b_{34}, b_{123}, b_{134}, b_{234}$, b_{1234} are interaction coefficients representing the parameters in their coded form. The relationship between the actual and coded values are given below:

$$X_1 = \frac{x_1 - 20}{10}, \qquad X_2 = \frac{x_2 - 2}{1}, \qquad X_3 = \frac{x_3 - 225}{75}, \quad \text{and} \quad X_4 = \frac{x_4 - 2}{1}$$

The regression coefficients were estimated by the following expression:

$$b_o = \sum_{i=1,2,3,\cdots,n} \frac{Y_i}{N}, \qquad b_j = \sum_{j,i=1,2,3,\cdots,n} \frac{X_jY_i}{N}, \qquad b_{jk\cdots n} = \sum_{j,k,\ldots,i=1,2,3,\cdots,n} \frac{(X_jX_k\ldots X_n)Y_i}{N}$$

The experimental order for obtaining the responses were done by a completely randomized design in which the allocations of the experimental parameters as well as the order in which the individual runs or trials of the experiment are to be performed are randomly determined. Such randomization of the order of experiments tend to average out the effect of any uncontrolled variables and validate the usual normality assumptions. All the factors except concentration have been randomly selected. Randomization with respect to concentration was not possible because each alumina suspension was used for three deposition experiments before replenishing it with a fresh one. Table 3 shows weight deposited for same suspension as well for second suspension under similar deposition conditions. It was found that the difference in weight of alumina deposited varies by about 7% (max) for the replicated center points and by 4% (max) for runs replicated from the same suspension. The center point placement was non-random as this is a well-known process. Two center points were front loaded in the run order and the remaining two were run at the end.

Transformation of the response data was necessary in our analysis since the ratio of maximum to minimum value of response obtained in the design matrix was very large. Transformations apply a mathematical function to all the

Table 1
Actual vis-à-vis coded values of parameters

Level	Concentratioin (A) (wt/100 ml)		Electrode separation (B) (cm)		Applied potential (C) (V)		Deposition time (D) (min)	
	Actual (x_1)	Code (X_1)	Actual (x_2)	Code (X_2)	Actual (x_3)	Code (X_3)	Actual (x_4)	Code (X_4)
Max level	30	+	3	+	300	+	3	+
Min level	10	–	1	–	150	–	1	–
Zero level	20	0	2	0	225	0	2	0

Table 2
Experimental matrix (according to standard order)

Standard order	Experiment order	Concentration (% w/v)		Electrode separation (cm)		Applied potential (V)		Deposition time (min)		Weight deposited (mg/cm^2)
		Actual (x_1)	Coded (X_1)	Actual (x_2)	Coded (X_2)	Actual (x_3)	Coded (X_3)	Actual (x_4)	Coded (X_4)	
1	6	10	−1	1	−1	150	−1	1	−1	10.45
2	16	30	1	1	−1	150	−1	1	−1	37.425
3	3	10	−1	3	1	150	−1	1	−1	4.15
4	9	30	1	3	1	150	−1	1	−1	24.175
5	18	10	−1	1	−1	300	1	1	−1	14.7
6	11	30	1	1	−1	300	1	1	−1	58.35
7	5	10	−1	3	1	300	1	1	−1	9.975
8	13	30	1	3	1	300	1	1	−1	43.975
9	7	10	−1	1	−1	150	−1	3	1	18.975
10	12	30	1	1	−1	150	−1	3	1	99.9
11	4	10	−1	3	1	150	−1	3	1	14.15
12	10	30	1	3	1	150	−1	3	1	63.225
13	8	10	−1	1	−1	300	1	3	1	38.025
14	15	30	1	1	−1	300	1	3	1	161.225
15	17	10	−1	3	1	300	1	3	1	28.525
16	14	30	1	3	1	300	1	3	1	106.85
17	2	20	0	2	0	225	0	2	0	38.125
18	19	20	0	2	0	225	0	2	0	40.95
19	1	20	0	2	0	225	0	2	0	38.025
20	20	20	0	2	0	225	0	2	0	39.325

Table 3
Error in repeating experimental trials from the same suspension

Suspension	Weight deposited		Difference	% Difference in deposit
	Deposit 1	Deposit 2		
Suspension 1	38.025	38.125	0.100	0.26
Suspension 2	40.95	39.325	1.625	3.96

Maximum difference between replicates = 7.14%.

response data and are generally used for three purposes: (i) stabilizing response variance, (ii) making the distribution of the response variable closer to the normal distribution, and (iii) improving the fit of the model to the data [27]. Transforming the response will make a difference only if the ratio of the maximum response to the minimum response is large. A ratio greater than 10 usually indicates that a transformation is required. Since the ratio of the maximum response to the minimum within the design space was 38.85, we applied a square transformation as suggested by the Box–Cox plot.

2.5. *Effects*

The EFFECT of a factor is the change in the response produced by a change in the level of the factor. The total number of effects in a 2^4 factorial design is 15. There are four main effects namely A–D which are due to variation of a single factor at a time. There are 11 interaction effects namely AB, AC, AD, BC, BD, CD, ABC, ABD, CD, BCD and ABCD that are due to variation in two or more factors, simultaneously. An interaction occurs when the effect of one factor depends on the level of another factor. The interaction effects are generally different from sum of the effects expected from either factor alone. The effects along with their contribution towards the response is summarized in Tables 4a and 4b. Concentration contributes more towards the weight of the alumina deposited than all other effects combined together (more than 50%). Other major contributing factors are deposition time, applied potential and

Table 4a
Effects list and their contribution towards response

Effects list	Term	Contribution (%)
Model	A (Concentration)	56.1126
Model	B (Electrode separation)	4.65128
Model	C (Applied potential)	8.56088
Model	D (Deposition time)	25.1722
Model	AB	0.52309
Model	AC	0.48906
Model	AD	3.27755
Error	BC	0.00129
Model	BD	0.14437
Model	CD	0.58734
Error	ABC	0.00806
Model	ABD	0.33236
Error	ACD	0.00163
Error	BCD	0.07546
Error	ABCD	0.00274
Model	Curvature	0.03325
Error	Lack of fit	0
Error	Pure error	0.02682

''Model'' indicates the inclusion of the factor in the final regression model while ''error'' indicates exclusion of the effects as they were found to be insignificant.

Table 4b
Analysis of variance (ANOVA) of the selected model

Source	Sum of squares	Degrees of freedom	Mean square	F-value	p-Value Prob $> F$	
Model	0.131594	10	0.013159	688.6568	<0.0001	Significant
A-concentration	0.073951	1	0.073951	3870.007	<0.0001	
B-elec Sep	0.00613	1	0.00613	320.7922	<0.0001	
C-app potential	0.011282	1	0.011282	590.4317	<0.0001	
D-dep time	0.033175	1	0.033175	1736.095	<0.0001	
AB	0.000689	1	0.000689	36.07678	0.0003	
AC	0.000645	1	0.000645	33.72949	0.0004	
AD	0.00432	1	0.00432	226.0479	<0.0001	
BD	0.00019	1	0.00019	9.95687	0.0135	
CD	0.000774	1	0.000774	40.50827	0.0002	
ABD	0.000438	1	0.000438	22.92264	0.0014	
Curvature	0.0000438	1	0.0000438	2.293185	0.1684	Not significant
Residual	0.000153	8	0.0000191			
Lack of fit	0.000118	5	0.0000235	1.994685	0.3023	Not significant
Pure error	0.0000354	3	0.0000118			

electrode separation. The dominant interaction effect is AD (interaction between concentration and deposition time) and is shown in Fig. 2. This is expected as both concentration and deposition time are the two major contributing factors.

2.6. Model refinement

Model refinement is primarily achieved through exclusion of the factors that are found to be insignificant. Exclusion of insignificant variables releases degrees of freedom for the calculation of confidence intervals [28]. Factor effects with a significance level of 0.05 or lower (p-value ≤ 0.05; 95% confidence level) were included in the regression model. This resulted in inclusion of 10 effects in the model. The positive effects were: A, C, D, AD, AC, BC, CD, ACD and ABCD while the negative effects were: B, AB, BD, ABC, ABD and BCD. An effect is said to be positive when increase in its level results in increase in the response and negative when increase in its level results in decrease in the response. It is

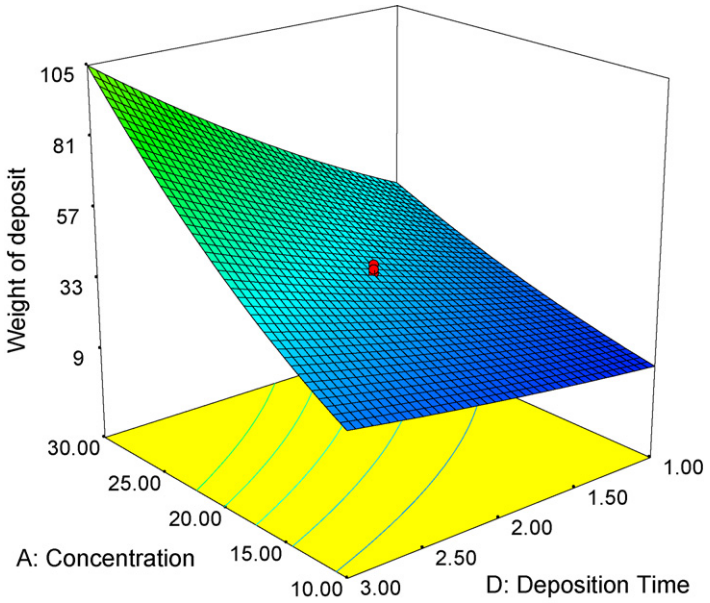

Fig. 2. A typical interaction effect between concentration and deposition time on deposition.

interesting to note that all interaction factors containing B (electrode separation) except BC contribute negatively towards the weight of alumina deposit obtained. This is expected as increase in electrode separation results in decrease in electric field which results in lesser rate of deposition. Also electrode separation term is inversely proportional to the quantity of particles deposited electrophoretically according to Hamaker equation [26,29].

2.7. ANOVA

Analysis of variance (ANOVA) of the selected model was carried out. ANOVA is a statistical technique which subdivides the total variation of a set of data into component parts associated with specific sources of variation for the purpose of testing a hypothesis on the parameters of a model. Tables 4a, 4b and 5 show the results of analysis of variance for the weight of alumina deposited.

Sufficient degrees of freedom were available for the evaluation of the model as can be seen in Table 6. Larger degrees of freedom increase the discrimination between adequate and inadequate models. The conclusions of the analysis of variance apply to the transformed data.

High model F-value of 688.66 indicated that the model is significant (Tables 4a and 4b). Insignificant curvature F-value of 2.29 validated our initial assumption of approximate linearity of the response variable in the design space. Hence design augmentation by introduction of higher order terms to the model was felt unnecessary. Lack of fit is the variation of data around the fitted model. An F-value of 1.99 corresponding to insignificant lack of fit suggested that the model fits well with the data. Hence the model was used to navigate the design space.

Table 5
Additional data from ANOVA

S.D.	0.00437
Mean	0.19
C.V. (%)	2.24
PRESS	0.00127
R^2	0.9988
Adj R^2	0.9974
Pred R^2	0.9904
Adeq precision	97.388

Table 6
Degrees of freedom for evaluation

Model	10
Residuals	9
Lack of fit	6
Pure error	3
Corr total	19

Standard deviation of 0.004 suggested low deviation associated with the experiment (Table 5). Mean is overall average of all the response data. C.V. (coefficient of variation) measures the unexplained or residual variability of the data as the percentage of the mean of the response variable. Low C.V. value of 2.24 indicated that the proportion of variability unexplained by the model was extremely low. In other words, the model was able to explain most of the variability of the data. Predicted residual error sum of squares (PRESS) is a measure of how the model fits each point in the design. Low PRESS value of 1.27 indicated that the observations did not highly influence the model which is desirable. R^2 gives a measure of the amount of variation around the mean explained by the model. This model was able to explain more than 99% of the variability in the weight of alumina deposited. Adj R^2 is a measure of the amount of variation around the mean explained by the model, adjusted for the number of terms in the model. The adjusted R^2 decreases as the number of terms in the model increases if those additional terms do not add value to the model. The adjusted R^2-value of 0.9974 indicated that all the selected terms contributed significantly to the model. Predicted R^2 gives some indication of the predictive capability of the regression model. Predicted R^2 of 0.9904 indicated that the model can be expected to explain more than 99% of the variability in predicting new observations in the design space. The overall predictive capability of the model based on this criterion can be considered to be extremely satisfactory. Adeq precision measures the signal to noise ratio. Adeq precision ratio of 97.388 indicated an adequate signal for proper model discimination. Since the noise in the system was insignificant compared to the signal as measured from the ''adeq precision'', it was decided not to replicate the design. Besides replication would have resulted in increased costs.

The regression equation for the alumina deposit obtained in terms of actual factors is

$$\begin{aligned}\text{Sqrt (weight of deposit)} &= +0.0738191 + 0.000828180 \times \text{concentration} - 0.0204777 \times \text{electrode separation} - 6.67949\text{E} - 007 \\ &\times \text{applied potential} - 0.0222254 \times \text{deposition time} + 0.000390049 \times \text{concentration} \\ &\times \text{electrode separation} + 8.46254\text{E} - 006 \times \text{concentration} \times \text{applied potential} + 0.00268953 \\ &\times \text{concentration} \times \text{deposition time} + 0.00701612 \times \text{electrode separation} \times \text{deposition time} + 9.27401\text{E} \\ &- 005 \times \text{applied potential} \times \text{deposition time} - 0.000523226 \times \text{concentration} \times \text{electrode separation} \\ &\times \text{deposition time}\end{aligned}$$

The regression equation in terms of coded factors is

$$\begin{aligned}\text{Sqrt (weight of deposit)} &= +0.194030 + 0.0679850 \times A - 0.0195735 \times B + 0.0265547 \times C + 0.0455348 \times D - 0.00656404 \times A \\ &\times B + 0.00634691 \times A \times C + 0.0164307 \times A \times D - 0.00344841 \times B \times D + 0.00695551 \times C \times D \\ &- 0.00523226 \times A \times B \times D\end{aligned}$$

2.8. Model adequacy testing

Residuals analysis is the primary diagnostic tool for checking violations of the basic assumptions, like normality, and model adequacy. The examination of the residuals from an unreplicated 2^k design can also provide information

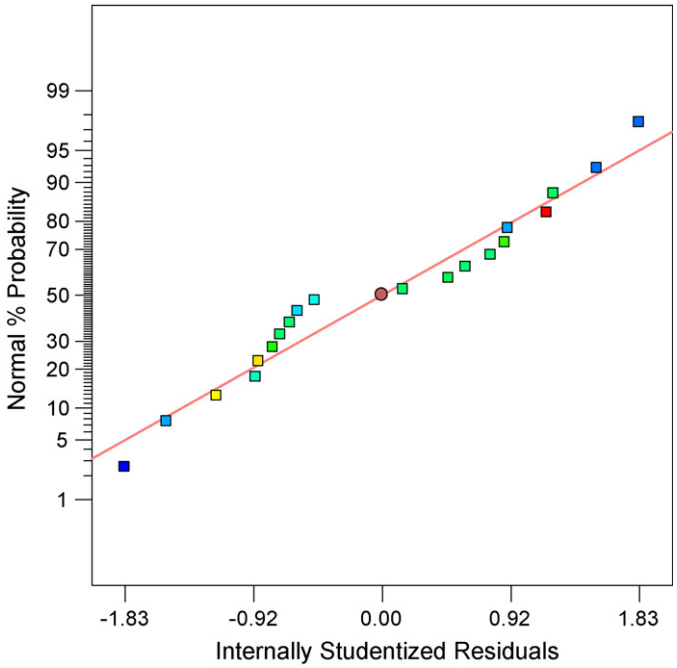

Fig. 3. Normal plot of residuals.

about process variability. Residual is the difference between the observed response and the value predicted by the model for a particular design point. The residuals should be structureless if the model is adequate.

2.9. Diagnostics

The normal probability plot is used to determine whether the residuals follow a normal distribution. The residuals seem to follow a normal probability distribution as they lie approximately on a straight line on the normal plot of residuals (Fig. 3). In general, moderate departures from normality are of little concern in the fixed effects analysis of variance which Design Expert uses [27]. Fig. 4 shows the plot of residuals versus predicted data points. A random scatter of data points in the residuals versus predicted plot validates our initial assumption of constant variance. Random scatter in the plot of residuals versus experimental run order (Fig. 5) eliminates the possibility of a time-related variable lurking in the background. The predicted and the actual values also show excellent agreement as can be seen from Fig. 6. Hence, no obvious patterns were found in the analysis of residuals.

2.10. Influence plots

Influence plots are primarily used for detection of outliers. Externally studentized residual (also called Outlier *t*) shown in Fig. 7 gives a measure of how many standard deviations the actual value deviates from the value predicted after deleting the point in question. All the data points (Fig. 7) lie within the limits.

Leverage is a measure of the influence of a point on the model fit. Leverages are numerical value between 0 and 1 that indicate the potential for a design point to influence the model fit. Leverage of 1 indicates that the model will be forced to go through the point and the point will control the model. Since the leverages of all runs (Fig. 8) are less than 1, there is no point which unduly influences the model.

DFFITS plot (Fig. 9), which measures the influence of each point on the predicted value, suggested four points (corresponding to runs 3, 5, 6 and 18) which influence the regression equation and the response very disproportionately. However, DFBETAS plot (Fig. 10) showed no undue/large influence of each observation on each of the regression coefficients. Cook's distance provides a measure of how much the regression would change if the case is omitted from the analysis. Cook's distance plot (Fig. 11) also suggested that the four points lying outside the

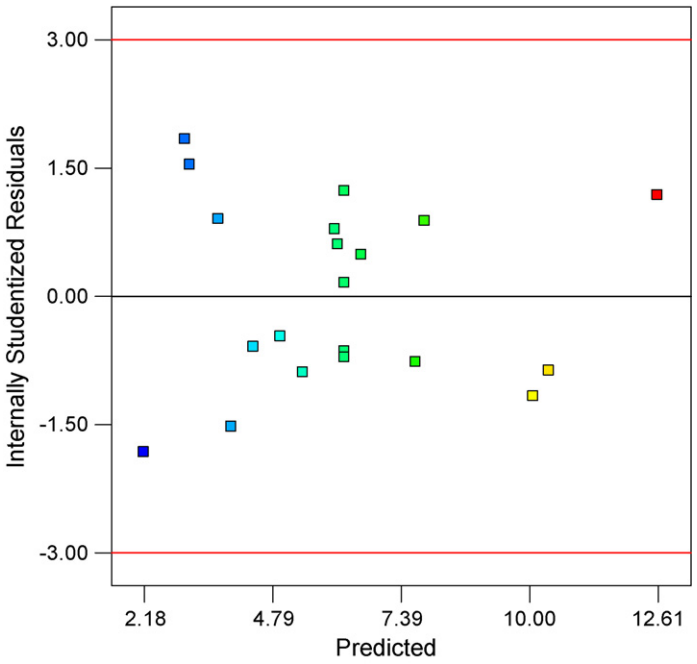

Fig. 4. Plot of residuals vs. predicted data points.

influence the regression equation more compared to other points. But since the Cook's distance values of none of these observations exceed 1, there is no strong evidence of influential observations in these data. These points are well within the limits in the externally studentized residual plot as well. And these four observations have the same leverage as the rest of the factorial run points. Hence these four points cannot be considered as outliers. Therefore, there are no outliers in the experimental data.

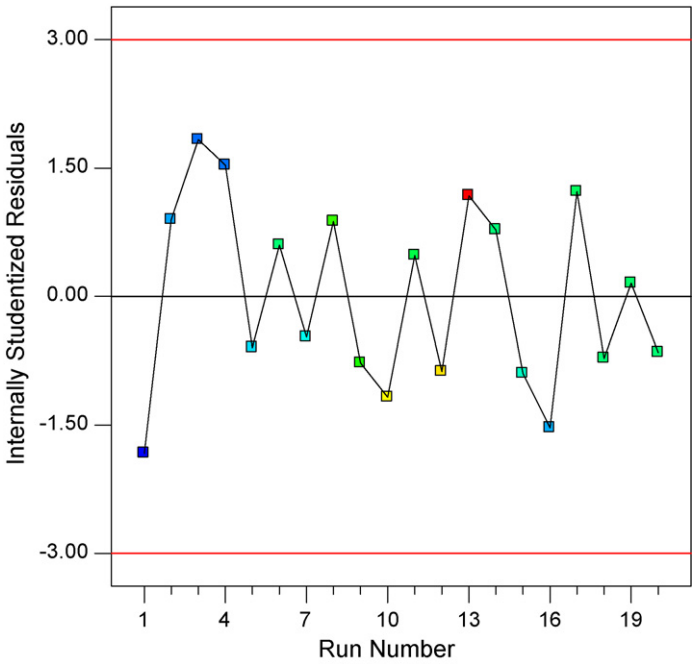

Fig. 5. Plot of residuals vs. experimental run order.

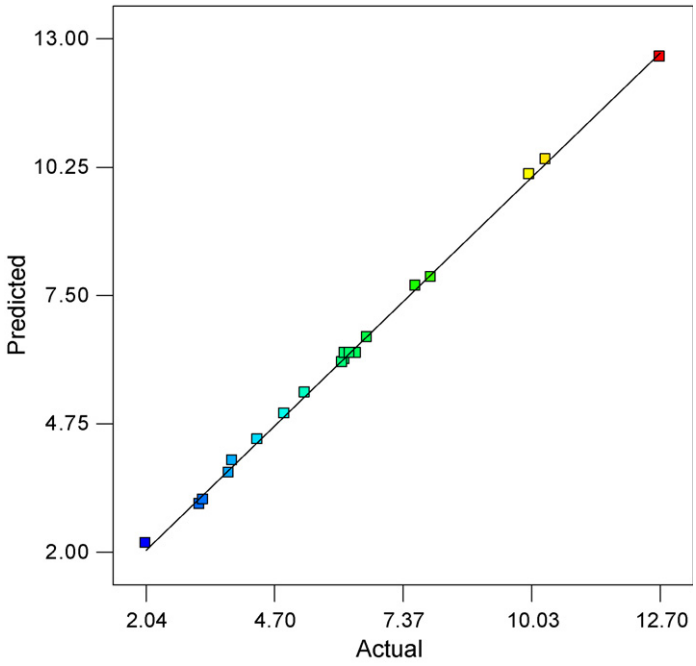

Fig. 6. Plot of predicted and the actual values.

2.11. Model evaluation

The power of a design is the ability of the design to detect that specific terms are statistically significant or the ability to find significant effects. In other words, it is the probability of detecting an effect of a specific size. The present model has a 94.4% chance of detecting the significant effects at 95% confidence level if the effect is the size of twice the standard deviation of the process (Table 7). This provides the model adequate discriminating power.

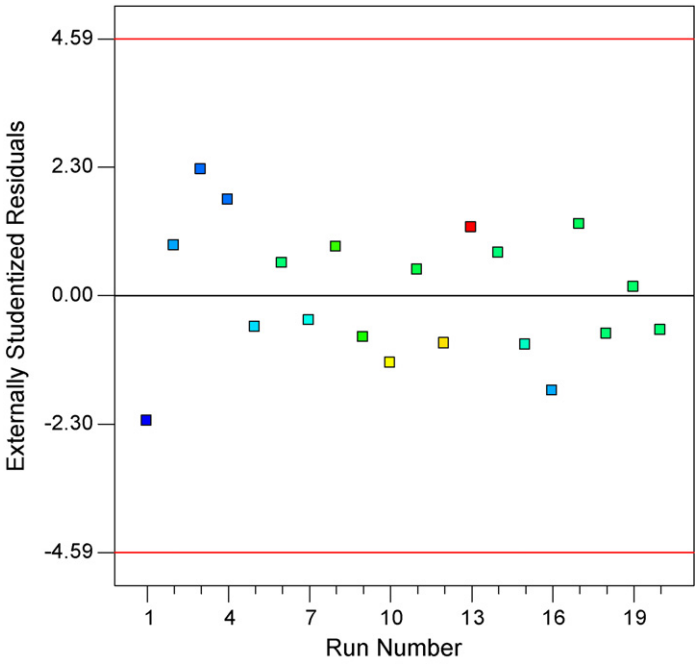

Fig. 7. Plot of externally studentized residuals.

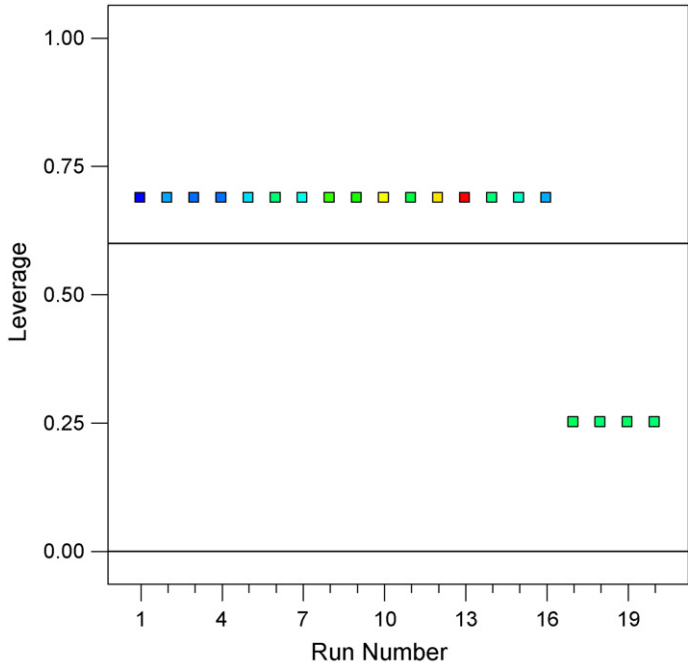

Fig. 8. Leverage of all runs.

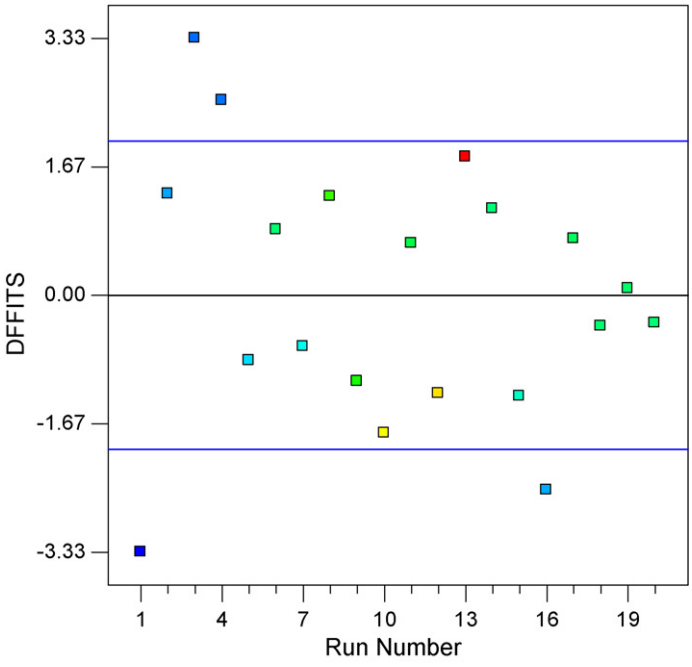

Fig. 9. DFFITS plot reflecting the influences of each point on the predicted value.

2.12. Model validation

Two experiments were carried out in order to check the predictive capability of the model that has been developed. The point prediction feature of the software allows the response to be predicted at any point in the design space. The

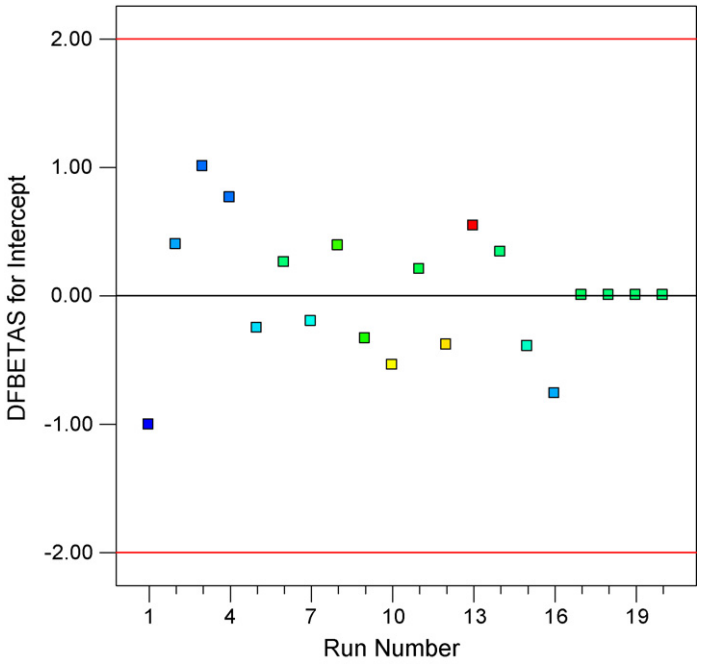

Fig. 10. DFBETAS plot showing influence of each observation on the regression coefficients.

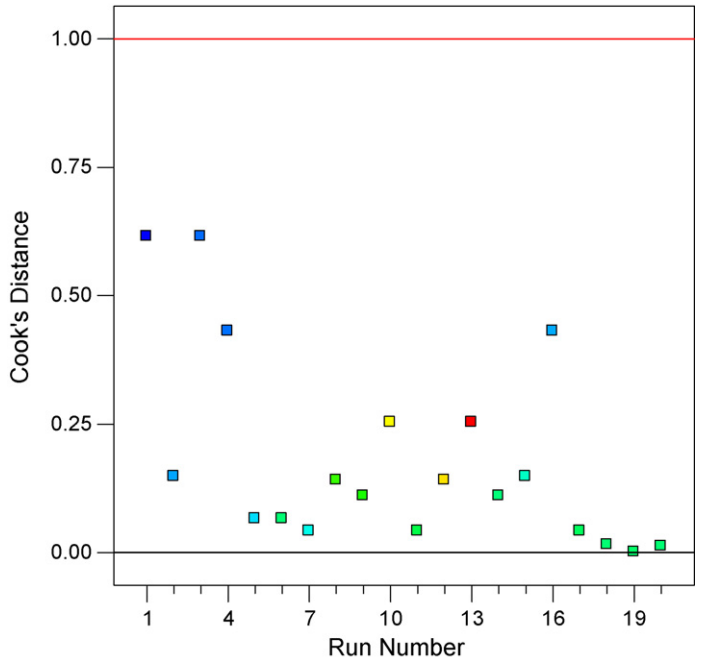

Fig. 11. Cook's distance plot showing the influence of each run on regression equation.

Table 7
Model evaluation data

Term	Power at 5% alpha level for effect of		
	0.5 S.D. (%)	1 S.D. (%)	2 S.D. (%)
A	14.60	43.10	94.40
B	14.60	43.10	94.40
C	14.60	43.10	94.40
D	14.60	43.10	94.40
AB	14.60	43.10	94.40
AC	14.60	43.10	94.40
AD	14.60	43.10	94.40
BD	14.60	43.10	94.40
CD	14.60	43.10	94.40
ABD	14.60	43.10	94.40

Basis S.D. = 1.0.

Table 8
Model validation data

Run	Concentration (g/100 ml)	Electrode separation (cm)	Applied potential (V)	Deposition time (min)	Weight of alumina deposit (mg/cm^2)	Point Prediction	Error (%)
1	10	1.5	200	2	15.35	15.8292	3.1218
2	10	2.5	250	1	8.825	8.82882	0.1591

two runs were arbitrarily chosen. The predicted and the experimental values of these two runs are tabulated in Table 8. There is a good correlation between the predicted and experimental values which suggest that the model can predict data accurately in the experimental matrix.

3. Conclusions

Statistical design of experiments was used to model the EPD of alumina from *iso*-propanol onto steel substrates. The dominant effects in decreasing order are: concentration, deposition time, applied potential, and electrode separation. Among the independent variables, the concentration contributes more than 50% towards the weight of deposit and hence is the most dominant factor. The most dominant interaction effect is that between concentration and deposition time. Electrode separation does not have much effect on the deposit as compared to the other main effects. The model was analyzed and validated. A good correlation was observed between the predicted and experimental values suggesting that the model can predict data accurately in the experimental matrix.

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