

Optimisation of process parameters to minimize defects using response surface methodology in a ceramic industry

Neelakandan B.¹ and Dr. C. Muralidharan²

¹Assistant professor, Manufacturing Engineering, Annamalai University, Annamalinagar 608002, Tamil Nadu, India.

²Professor, Manufacturing Engineering, Annamalai University, Annamalinagar 608002, Tamil Nadu, India.

Abstract

This study utilize pareto analysis and cause and effect analysis to screen various defects which are possible to occur during the production of, industrial oriented small ceramic tile. The process parameters speed/ stroke of the ram of a high speed mechanical press, amount of binder added to the raw material, cleaning of the punch and room temperature (humidity) were used as design variables. These process variables were mathematically related to quantum of defects occurred using response surface methodology (RSM) technique. Non linear mathematical functions were derived based on the process parameters, optimal conditions were found and optimized.

Key words: Response surface methodology(RSM), pareto analysis, cause and effect analysis, industrial ceramic tiles.

Introduction

Ceramic products are widely used in almost all types of industries. These ceramic products are usually classified into four main types; Structural- including bricks, pipes, floor and roof tiles, refractories-such as kiln linings, gas fire radiants, steel and glass making crucibles, white wares - including tableware, cookware, wall tiles, pottery products and sanitary ware and technical, also known as engineering, advanced, special, and fine ceramics (in general industrial tiles) - for example, gas burner nozzles, ballistic protection, nuclear fuel uranium oxide pellets, biomedical implants, coatings of jet engine turbine blades, ceramic disk brake, missile nose cones, bearing (mechanical). These industrial tiles and particularly small tiles (less than 100mm in size) are used in various areas such as grinding media, ball mill liners, power distribution equipments, wear resistance liners and lined equipments to steel industry, power generation industries, electronics industry, textile machinery, defense, automobile

industry, avionics, high power/high temperature applications. In this study, data is gathered from a leading manufacturer dedicated to the manufacturing of various ceramic products for industrial applications. The industry is well known in its sector, located in south India and continuously improving to be the best in the market with more than 3000 employees. The company has achieved a reputation for its quality and innovation. The company is trying hard to reduce defects particularly in the stage of green tiles rather than baked tiles to reduce cost of recycling.

Objective of this study

1. To identify the causes of various defects
2. Selection of predominant process parameters in order to minimize defects
3. To optimize the selected process parameters using RSM.

Selection of tile

Even though the company is manufacturing nearly 18,000 varieties, a small tile of 20×20×5 size with small projection is taken for study (Figure 1). Its area of application are grinding media, ball mill liners, power distribution equipments, wear resistance liners and lined equipments to steel industry, power generation industries. The major factors influence in selection of this tile includes

- a. Volume of production – moderately high.
- b. Rejection rate – high.
- c. Acute care needed during production- only trained operator.
- d. Profit – high.
- e. Production throughout the year (not Seasonal).
- f. Dust & dirt should be eliminated.
- g. Alumina spray is done during re-stacking.

h. Only manual inspection is carried out as the size of the tile is small and more over the volume of production is moderately high.

i.

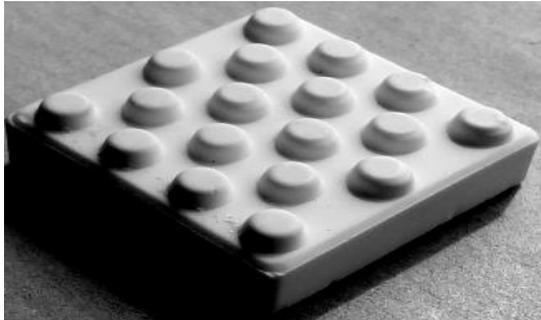


Figure 1 20x20x5 small projection tile.

Stages in production of small tiles

1. Raw material preparation

In industrial ceramics tiles three distinct material categories are used:

- Oxides: alumina, beryllia, ceria, zirconia
- Nonoxides: carbide, boride, nitride, silicide
- Composite materials: particulate reinforced, fiber reinforced, combinations of oxides and nonoxides.

Each one of these classes can develop unique material properties, because ceramics tend to be crystalline. The small tile taken for study utilize aluminum oxide, more commonly known as alumina as its raw material. Operations such as milling, batching, blending- mixing of alumina, refractories and binders are carried out to get uniform fine particle powder. As this small tile is designed with small projection, folic acid is used as an additive (binder) in order to ease the moulding process.

Even though a variety of moulding operations performed in the industry, moulding of the small tiles limits with high speed moulding mechanical presses, followed by other operations in the manner of stacking, re-stacking, baking in furnace and finally inspection and packing as shown in Figure 2.

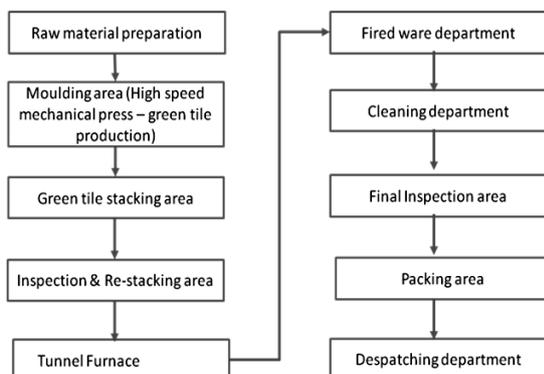


Figure 2. process flow chart

The blended mix is introduced into a mould cavity or a die and pressed to produce a green tile (compact).

This moulding operation is done in a high speed mechanical press. The green tiles produced are initially stacked in the wooden bats and stored. These green tiles are then inspected and re-stacked in silicon bats. Dry alumina powder is sprayed over each layer to avoid sticking of tiles with other and then sintered in the tunnel furnace.

3. Sintering

The small green tile moulded will be rather very fragile, and handling should be done with acute care to avoid breakage. The principal goal of sintering is the reduction of porosity in the green tile and to strengthen it. The initial spaces between compacted grains in small tiles are called “voids”. Sintering process is concerned with closing of voids present in the green small tiles. Finally the sintered small tiles are inspected, packed and despatched.

Screening of defects

In order to screen the defects occurs during the production of small tile - 20x20x5 small projection tile a pilot experiments was conducted. The weight of the green small tile ranges from 0.48 grams to 0.50 grams. Two thousand kilograms of virgin powder with folic acid as additive is utilized to produce 1,00,000 small projection tiles. High speed mechanical moulding press is used to make the green compact. These green tiles are stacked initially at the moulding area in the wooden bats and stored separately. Then green tiles are inspected and re-stacked in silicon bats. Dry alumina powder is sprayed over each layer of the green tile to avoid sticking of tiles with each other during sintering. These green tiles are then loaded in furnace and sintered for 36 hours. Finally the fired tiles are then cleaned and inspected. The defects found after inspection of the fired tiles were tabulated in Table 1 and a pareto analysis for defects is attained (Figure 3)

Table 1 Details of defects accounted during pilot study

Defects	Count	Cumulative Count	Cumulative %
Mo.Green Chip	14200	14200	26.56%
Green chip	12050	26250	49.11%
Fired ware Chip	11100	37350	69.87%
Join	9900	47250	88.39%
Body stick	4375	51625	96.58%
Black Spot	925	52550	98.31%
Before Fired Chip	500	53050	99.24%
Sic stic	375	53425	99.94%
Alumina	30	53455	100.00%

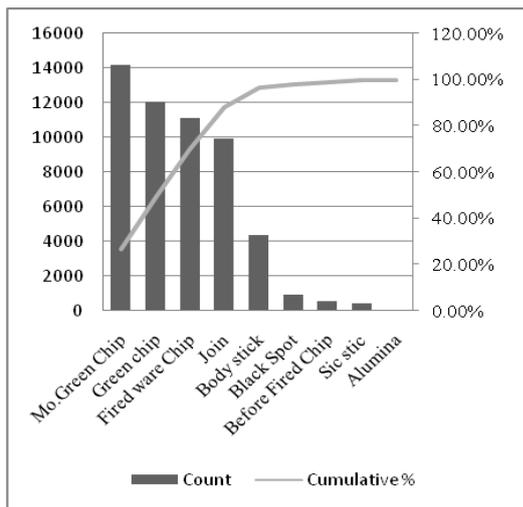


Figure 3 Pareto analysis on defects.

From the pareto analysis it is found that nearly 50% of the defects were due to Mo.green chip and green chip. Based on industrial expert's opinion and with a brain storming session the causes for the defects in the small projection tile is arrived and the corresponding fishbone diagram (Figure 4) is drawn.

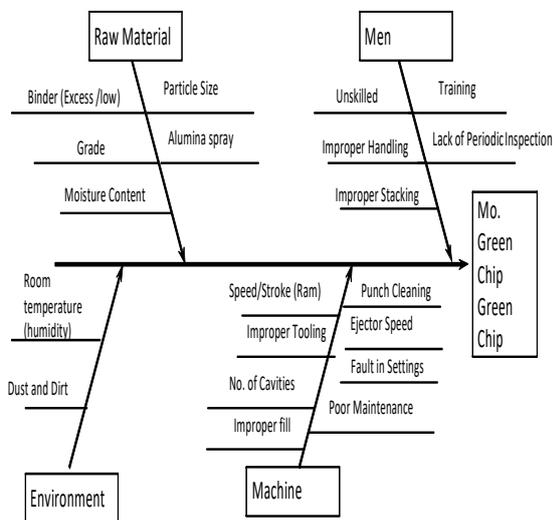


Figure 4 Fishbone diagram

Selection of process parameters and fixation of limits

From the pilot experiments conducted and with expert's opinion, the predominant process parameters that have greatest influence on defects during the production of small projection tile were identified. They are:

1. Room temperature (humidity)
2. Punch cleaning
3. Binder
4. Speed / stroke of Ram

These are the primary process parameters(factors) contributing to the Mo. Green chip and green chip during the production of 20x20x5 small projection tile. The selection of process parameter's levels were based on the process knowledge of both practical and theoretical, trial runs, pilot experiments and expert's suggestion. The chosen level of important process parameters with their units and notations are presented in Table 2. The feasible limits of the parameters were chosen in such a way that the defects in the green tiles should be minimum.

Table 2 Important process parameters and their levels

Factors	Units	Notation	Factor levels				
			-2	-1	0	1	2
Room temperature (humidity)	°C	A	24	25	26	27	28
Punch cleaning	strokes / cleaning	B	700	750	800	850	900
Binder	ml / kg	C	40	45	50	55	60
Speed / stroke of Ram	strokes / min	D	30	40	50	60	70

Developing the experimental design matrix

In response surface methodology (RSM), central composite rotatable design of second order was found to be the most efficient tool to establish the mathematical relation of the response surface using the smallest possible number of experiments without losing its accuracy (Gunaraj and Murugan 1999). (Box et al., 2005; Cochran and Cox, 1957). The relation between actual/ natural value of the factors and coded value of the factor is

$$F_{Nat} \longrightarrow_{coded} = [(F_{Nat} - F_{Nat\ Center})/\Delta F] ;$$

Equation - (1)

F_{Nat} is the natural/actual value of a factor, $F_{Nat\ Center}$ is the middle value of a factor and ΔF is the incremental value of a factor. The experiments are performed in random order. The response (defect) was investigated as per design layout, accounted and tabulated in Table 3. The obtained results are analyzed using Design Expert 8.0 software.

Table 3 Experimental design matrix and results- CCD -2⁴ factorial design with centre and axial points of star design

Standard order	Run order	Coded values				Actual values				Defects (W)
		Temp (A)	Cleaning (B)	Binder (C)	Speed (D)	Temp (A)	Cleaning (B)	Binder (C)	Speed (D)	
1	18	-1	-1	-1	-1	24	750	45	30	135
2	3	1	-1	-1	-1	28	750	45	30	170
3	21	-1	1	-1	-1	24	850	45	30	122
4	22	1	1	-1	-1	28	850	45	30	168
5	26	-1	-1	1	-1	24	750	55	30	144
6	13	1	-1	1	-1	28	750	55	30	175
7	19	-1	1	1	-1	24	850	55	30	130
8	12	1	1	1	-1	28	850	55	30	182
9	14	-1	-1	-1	1	24	750	45	70	175
10	27	1	-1	-1	1	28	750	45	70	155
11	28	-1	1	-1	1	24	850	45	70	189
12	16	1	1	-1	1	28	850	45	70	180
13	8	-1	-1	1	1	24	750	55	70	186
14	23	1	-1	1	1	28	750	55	70	162
15	29	-1	1	1	1	24	850	55	70	198
16	25	1	1	1	1	28	850	55	70	186
17	9	-2	0	0	0	22	800	50	50	180
18	4	2	0	0	0	30	800	50	50	202
19	15	0	-2	0	0	26	700	50	50	198
20	11	0	2	0	0	26	900	50	50	209
21	24	0	0	-2	0	26	800	40	50	159
22	1	0	0	2	0	26	800	60	50	173
23	7	0	0	0	-2	26	800	50	10	90
24	17	0	0	0	2	26	800	50	90	128
25	30	0	0	0	0	26	800	50	50	99
26	10	0	0	0	0	26	800	50	50	96
27	5	0	0	0	0	26	800	50	50	94
28	20	0	0	0	0	26	800	50	50	96
29	2	0	0	0	0	26	800	50	50	92
30	6	0	0	0	0	26	800	50	50	93

Model adequacy check

According to Montgomery (2007); Box et al., (1978) higher order interactions i.e., more than two factors interactions are practically insignificant and hence not considered. This method is also used to summarize the test for significance of regression model, test for significance for individual model coefficient and test for lack of fit. Summary output shows that the quadratic model is statistically significant for response at the two different conditions. Significant model terms were identified at 95% significance level. Goodness of fit was evaluated from R² (coefficient of correlation) and CV (coefficient of variation) in order to check the reliability and precision of the model.

The adequacy of the developed model was tested using the ANOVA method and results of second-order response surface model are given in Table 6. The ANOVA results show that all the predominant factors (A, B, C, D) and interactions AB, AD, BD and A², B², C², D² are considered as significant factors. The value of the coefficient of correlation (R²=0.9969) indicates that 99.69% of the

total variability is explained by the model after considering the significant factors. The comparison of R² and adjusted R² values shows that the model is not over fitted and only less than 1% of the total variations are not explained by the model. The value of adjusted coefficient of correlation (adjusted R²= 0.9940) is also high which reveals the high significance of the model. Predicted R² = 0.9851 is in a good agreement with the adjusted R² and indicates that the model is capable to explain 98.51% of the variability of result in the case of new data.

Adequate precision ratio greater than 4 is preferred. In this case it was found to be 57.946 which were well above the preferred one. Similarly, the lower value of coefficient of variation CV= 1.91 shows that the experimental values attained were with high degree of precision and with a good deal of reliability. Small values of PRESS are desirable. The model F- value of 341.54 broadcast that the model is significant. The probability > F for the model in Table 4 is less than 0.05 which indicates that the model is significant. Lack of fit is insignificant thereby it indicates that the model fits well with the experimental data.

Table 4 Anova result for defects(df is degrees of freedom, F is fisher's ratio; p is probability)

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	43911.05	14	3136.504	341.543	< 0.0001	significant
A-Temp	852.0417	1	852.0417	92.78131	< 0.0001	
B-cleaning	234.375	1	234.375	25.52178	0.0001	
C-binder	392.0417	1	392.0417	42.69056	< 0.0001	
D-Speed	3290.042	1	3290.042	358.2623	< 0.0001	
AB	189.0625	1	189.0625	20.58757	0.0004	
AC	1.5625	1	1.5625	0.170145	0.6858	Insignificant
AD	3277.563	1	3277.563	356.9034	< 0.0001	
BC	1.5625	1	1.5625	0.170145	0.6858	Insignificant
BD	588.0625	1	588.0625	64.03584	< 0.0001	
CD	0.5625	1	0.5625	0.061252	0.8079	Insignificant
A^2	15512.17	1	15512.17	1689.165	< 0.0001	
B^2	19856.81	1	19856.81	2162.266	< 0.0001	
C^2	8430.027	1	8430.027	917.9702	< 0.0001	
D^2	295.3125	1	295.3125	32.15744	< 0.0001	
Residual	137.75	15	9.183333			
Lack of Fit	105.75	10	10.575	1.652344	0.3017	not significant
Pure Error	32	5	6.4			
Cor Total	44048.8	29				
Std. Dev.	3.030402		R-Squared	0.996873		
Mean	152.2		Adj R-Squared	0.993954		
C.V. %	1.991065		Pred R-Squared	0.985126		
PRESS	655.2		Adeq Precision	57.94552		

Developing an empirical relationship

The second order polynomial regression equation used to represent the response surface 'Y' is given by (Sivakumar et al.,2010)

$$Y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{i < j} \beta_{ij} x_i x_j + \epsilon$$

Equation - 2

And for four factors, the selected polynomial can be expressed as

$$W = b_0 + b_1(A) + b_2(B) + b_3(C) + b_4(D) + b_{12}(A \times B) + b_{13}(A \times C) + b_{14}(A \times D) + b_{23}(B \times C) + b_{24}(B \times D) + b_{34}(C \times D) + b_{11}(A^2) + b_{22}(B^2) + b_{33}(C^2) + b_{44}(D^2)$$

Equation - 3

All the coefficients of equation 3 are obtained by applying central composite design using Design Expert 8.0 statistical software package. After determining the coefficients, the final model is developed using these coefficients. The response(W) 'defects' of the 20x20x5 small projection tile in the actual form is given by

$$W = 95.00 + (5.96 \times A) + (3.12 \times B) + (4.04 \times C) + (11.71 \times D) + (3.44 \times A \times B) - (0.31 \times A \times C) - (14.31 \times A \times D) + (0.31 \times B \times C) + (6.06 \times$$

$$B \times D) - (0.19 \times C \times D) + (23.78 \times A^2) + (26.91 \times B^2) + (17.53 \times C^2) + (3.28 \times D^2)$$

Equation - 4

Where; A,B,C,D are actual levels of the factors. The value of A,B,C,D is taken from maximum and minimum actual levels. All the coefficients of equation 4 are tested for their significance at 95% confidence level applying t-test using Minitab 15 software and Microsoft Excel 2007 package.

Table 5 shows the significant coefficients for the response (W) 'defect'. The coefficient of the main parameters of 20x20x5 small projection tile A,B,C,D, and interactions AB,AD,BD and A²,B²,C²,D² are significant and interactions AC,BC and CD are insignificant in t-test. After determination of the significant coefficients, the confidence interval and standard error of the equation 4 are also tabulated and model reduction is carried out. At 95% confidence level, the reduced model for 20x20x5 small projection tile for the response(W) 'defect' is given by

$$W = 95.00 + (5.96 \times A) + (3.12 \times B) + (4.04 \times C) + (11.71 \times D) + (3.44 \times A \times B) - (14.31 \times A \times D) + (6.06 \times B \times D) + (23.78 \times A^2) + (26.91 \times B^2) + (17.53 \times C^2) + (3.28 \times D^2)$$

Equation - 4a

Table 5. Significance of coefficients for the response ‘defect’.

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	T	P
Intercept	95	1	1.237156	92.36306	97.63694	98.920	0.000
A-Temp	5.958333	1	0.618578	4.639865	7.276801	11.845	0.000
B-cleaning	3.125	1	0.618578	1.806532	4.443468	6.043	0.000
C-binder	4.041667	1	0.618578	2.723199	5.360135	7.816	0.000
D-Speed	11.70833	1	0.618578	10.38987	13.0268	22.803	0.000
AB	3.4375	1	0.7576	1.822713	5.052287	5.428	0.000
AC	-0.3125	1	0.7576	-1.92729	1.302287	-0.493	0.628 (insignificant)
AD	-14.3125	1	0.7576	-15.9273	-12.6977	-22.994	0.000
BC	0.3125	1	0.7576	-1.30229	1.927287	0.099	0.923 (insignificant)
BD	6.0625	1	0.7576	4.447713	7.677287	9.573	0.000
CD	-0.1875	1	0.7576	-1.80229	1.427287	-0.296	0.771 (insignificant)
A ²	23.78125	1	0.578627	22.54794	25.01456	50.307	0.000
B ²	26.90625	1	0.578627	25.67294	28.13956	56.904	0.000
C ²	17.53125	1	0.578627	16.29794	18.76456	37.114	0.000
D ²	3.28125	1	0.578627	2.047936	4.514564	7.825	0.000

Normal probability plot

The normal probability plot of the residuals for defects, in Figure 5 shows that the residuals are falling on the straight line, which means that the errors are distributed normally (Kumar et al.,2007). Based on the above considerations, it is justified that the adequacy of the regression model is good.

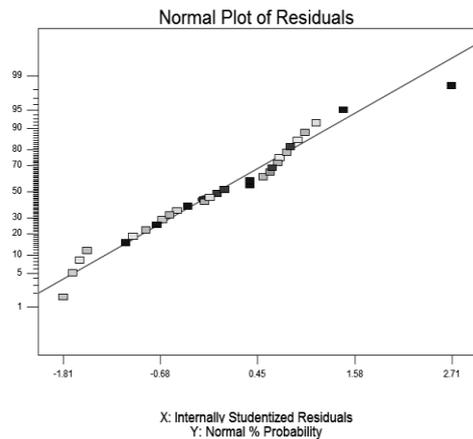


Figure 5 normal probability plot effects of residuals for defects

Optimising the parameters

In the present investigation the process parameters corresponding to the minimum defects (W) is considered as optimum (analyzing the contour graphs and by solving Equation 4). Hence, when these optimized process parameters are used, it will be possible to attain the minimum defects for the 20×20×5 small projection tile.

Figure 6 presents three dimensional surface plots for the response defects (W) obtained from the regression model. The optimum defects (W) is exhibited by the corners of the response surface plots. Contour plots plays a vital role in the study of the response surface analysis which are generated using Design Expert 8.0 software. The optimum is identified with reasonable accuracy by characterizing the shape of the surface. If a contour patterning of circular shaped contour occurs, it tends to suggest independence of factor effects, whereas elliptical contours may indicate factor interactions (Box et al., 2005).

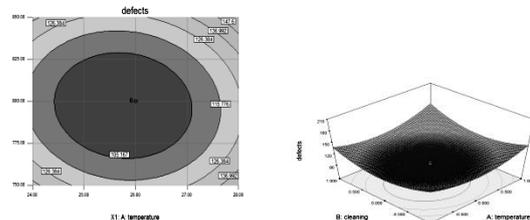


Fig: 6-a: Surface and Contour Plot (AB)

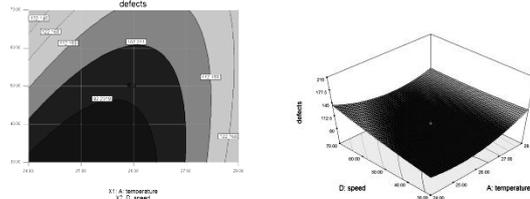


Fig: 6-b: Surface and Contour Plot (AD)

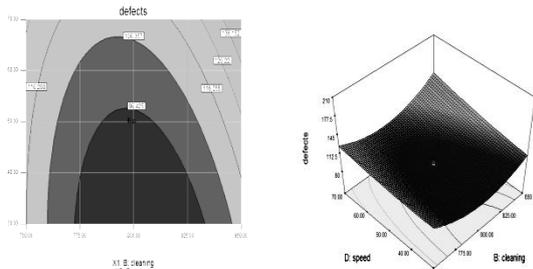


Fig: 6-c: Surface and Contour Plot (BD)

Figure 6(a), 6(b) and 6 (c) exhibits curved lines and elliptical shaped contour, which reveals the interaction of factors. It is relatively easy, while examining the contour plots in Figures 6 (a) to 6 (c) that changes in defect are more sensitive to changes in speed/stroke of the Ram than to temperature, binder and cleaning. The interaction effect between temperature and speed/stroke of ram is more significant than the interaction effect between other combinations of parameters. The minimum defects predicted from the contour graph and surface plots is 95. The values were also verified using statistical software Minitab 15. The corresponding optimization plot is shown in Figure 7.

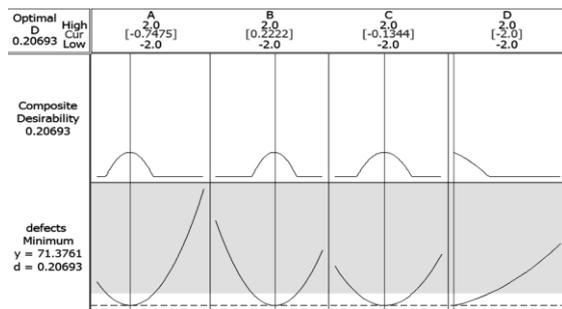


Figure 7 Optimization plot for individual parameters.

To optimum values from the optimization plot for the process parameter: temperature (A) cleaning (B), Binder(C) and for speed /stroke of Ram(D) are -0.7475, 0.2222, -0.1344 and -2 respectively, which are coded values. The corresponding natural or actual values of the process parameters A,B,C,D are 25.25, 811.11, 49.31 and 30 respectively. These values were attained by using the Equation 1. Confirmatory experiments was conducted with the optimum parameters, temperature 25°C, cleaning carried at every 800 strokes of the ram,50 ml of binder is added

and the machine runs at 30 strokes per minute. These values are set to the nearest point due to the limitation of the machine.

Discussion

In this paper, the application of response surface methodology from the point of 20×20×5 small projection ceramic tile is discussed. In this experimental design, four factors were studied. The development of response surface model has been founded on central composite design of experiments with five factor levels. The analysis of variance proved that the speed/ stroke of the ram, most significantly contributes defects. The defects is additionally contributed by temperature, binder and cleaning of the punch. Then the optimization mode was made; objective function as the relationship between the process parameters and responses which have been obtained from the response surface methodology.

Reference

- [1] Box, G.E.P., Hunter, J.S., & Hunter, W.G. (1978). Statistics for experiments. John Wiley Publisher, New York.
- [2] Box, G.E.P., Hunter, J.S., & Hunter, W.G. (2005). Statistics for experiments. Hoboken, NJ: John Wiley & Sons.
- [3] Cochran, W. G & Cox, G. M. (1957). Experimental designs. UK, London: John Wiley.
- [4] Gunaraj, V. and Murugan, N. (1999), 'Prediction and optimization of weld bead volume for the submerged arc process, Part 2', Welding Journal, Vol. 79, No.11, pp. 331-338.
- [5] .Kumar, S., Kumar, P. and Shan, H.S. (2007) ' Effect of evaporative pattern casting process parameters on the surface roughness of Al-7% Si alloy castings', Journal of Materials Processing Technology, Vol.182, pp.615-623.
- [6] Montgomery, D.C. (2007) Design and Analysis of Experiments, 5th ed., John Wiley & Sons, New York.
- [7] Sivakumar, T., Manavalan, R. and Muralidharan, C. (2007) ' An improvement HPLC method with the aid of a chemometric protocol: simultaneous analysis of amlodipine and atorvastatin in pharmaceutical formulations' , Journal of Separation Science, Vol.30, pp. 3143- 3156.