Application of Taguchi Method for Experimental Design of Dephosphorization process of steel through Induction furnace route

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Abstract: The industries who are using induction melting furnace to produce steel does not carry out any refining process, the steel produced by induction route was considered to be of inferior quality and not suitable for critical applications. Thus, in this work a methodology has been designed to produce steel from induction routes of similar quality as the steel produced through electric arc furnace and Basic Oxygen Furnace. In this new design a ladle refining furnace was implemented after induction melting. In this process the removal of phosphorous was achieved by addition of calcium oxide as a flux to maintain the basicity of the ladle refining furnace. The phosphorous has been removed in great extent and degree of dephosphorization has been achieved up to 78-85%. The extent of phosphorous removal was achieved by maintaining dephosphorization index and basicity in best possible range and is found to be in line with the design of experiment framed by Taguchi method. Taguchi method was used to find the best possible combination of degree of dephosphorization, dephosphorization index and V-ratio to remove the phosphorous from steel.

Keywords: Steel; Induction furnace; Ladle refining furnace; Flux; dephosphorization; Taguchi.

1. Introduction

The dephosphorization is a technique used to remove phosphorous from molten steel by means of oxidizing the phosphorous into their oxides in the presence of oxygen. Steel making processes are broadly categorized into primary and secondary routes. The dephosphorization in the primary route of steelmaking is achieved by addition of oxygen to the molten bath through lancing [1]. The phosphorous removal in secondary route of steelmaking is achieved in electric arc furnace and but it was difficult to remove phosphorous from steel in induction furnace steel making process. Earlier it was assumed that in induction furnace we can't make steel but only melt which is like "Garbage in" and "Garbage out". This thought has been proven wrong and now all types of steels are being manufactured by technologists through induction furnace route [2].The Ministry of steel, Government of India published a report in which it has been mentioned that the total crude steel production in India during the year 2021-2022 was 118.134 MT and out of which 28% was the contribution of induction furnace route [3].The plain carbon steel and construction quality steels are mostly produced through induction furnace route [4]. To achieve better removal of phosphorous from steel, Japan has established a duplex process where the converter for removal of phosphorous and decarburization refining converter was utilized simultaneously. Due to this technique, basic oxygen furnace productivity and molten steel quality has been greatly improved [5-9]. The induction furnaces are broadly classified into three classes like Coreless Induction Furnaces, Channel furnace and Pressure Pour Furnace. The coreless induction furnace could be a vessel type furnace having refractory lining within the alongside electrical current carrying coils surrounding the vessel. A solid charge containing scrap, Fe and ferroalloys are melted. A channel furnace could be a kind of induction furnace during which heating takes place within the channel, a comparatively small and narrow space at the bottom of the bath. The channel passes through a laminated steel core and round the coil assembly. A pressure pour is similar to channel furnace; the furnace is carefully sealed so that the metal can be moved out of the furnace by pressurizing the chamber above the molten metal bath [10].

In spite of the making efforts in this direction [11-13], there's dearth in chemistry of slag-metal reactions and measurements of thermochemical properties of steel in the induction furnace. Many efforts has been made in the purification of steel in induction furnace but shown the dearth in chemistry of slag-metal reactions and measurements of thermochemical properties of steel[11-

13].Thus, the most important objective of this research work was to come up with the experimental information helpful to industrial scale Induction melting furnace. During this work the effort has been made to get rid of phosphorous from the steel melt in induction furnace and more in ladle refinement furnace.

The method used in this study is a process of design of experiment called the Taguchi design. This method is developed by Dr. Genichi Taguchi and is a set of methodologies by which the essential variability of materials and processes are taken into account at the design stage [14]. By using the Taguchi techniques, industries are able to greatly reduce product development cycle time for both design and production, therefore reducing costs and increasing profit [15]. In this study, orthogonal arrays of Taguchi method and signal to noise (S/N) ratio are used to measure the performance of the process responses. A Contour plot analysis of regression is also performed to identify the process parameters that are statistically significant. Finally, the obtained modelling results were verified using the experimental tests.

2. Experimental Procedure

An online Minitab version (Freely available software) was utilized for the Taguchi analysis for optimizing the parameters and design of experiment. Initial values of V-ratio and dephosphorization index were obtained through random experiments which further was utilized for designing the experiment for achieving high degree of dephosphorization. The L9 run is used because neither too less or too huge data is required. Through L9 we just need moderate number of experimental data to analyze the process and thus was adopted in 3-Level design which was utilized for the studies.

A twelve Ton/5MW coreless Induction furnace procured from Inductotherm Ltd India was used for the experiments. A line diagram of the induction furnace is depicted in Figure 1. Neutral refractory was used as lining for the furnace so that lining mustn't be attacked. To measure the furnace temperature a Pt-Rh thermocouple with high accuracy was used. The raw materials used for production of steel was sponge iron 80% and less than 20% pig iron and steel scrap. Optical emission spectroscopy (OES) and X-Ray Fluorescence (XRF) was used for the determination of chemical compositions of raw material as well as the final product.

First of all, the furnace was dried by simply keeping pig iron within the furnace and application of 1MW power for initial hour. Then power was increased up to 2MW for melting the solid charge. The furnace was dried after dropping the Pig iron and run the furnace at 1MW power per hour. Later the pig iron was charged and melted at an influence of 2MW and the power was incrementally raised together with the addition of solid charge to 5MW. The frequency (420 (Hz) to 620(Hz)) of the furnace was controlled after complete addition of sponge iron and temperature was measured. Then total slag generated was removed by spatula and temperature of the bath was measured to be 1630°C. Steel and slag samples were collected for more investigations.

A forty Ton/8MW Ladle furnace was utilized in this work and temperature was maintained in between 1570˚C - 1590˚C. To maintain the desired temperature of the liquid steel for the phosphorous removal in the ladle furnace was achieved by the electrical power supply through electrode system. The homogenization of steel temperature and chemistry was achieved by the stirring of bath with the help of inert gas. Formation of a slag layer over the steel bath that protects refractory from arc damage and transfers heat to the liquid steel, absorb inclusions and metal oxides and provide the means for dephosphorization.

Chemical characterization:

The Chemical composition of the raw materials (sponge iron and pig iron) has been examined by OES analysis and the composition of direct reduced iron (DRI), Pig iron and steel scrap are provided in the Table 1.

Table 1: Chemical composition of Raw materials obtained using OES and XRF

Raw material	Composition (%)						
	C	Mn	Si	S	P	SiO_2	Fe
DRI	0.1	-----	------	0.03	0.096	2.31	Bal
Pig Iron	4.32	-----	------	0.05	0.12	----	Bal
Steel Scrap	0.28	0.64	0.35	0.05	0.05	-----	Bal

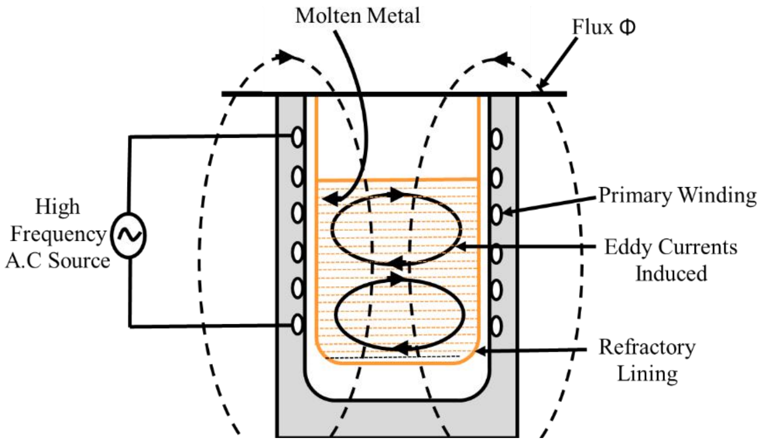

Fig.1: Line diagram of induction furnace

3. Results and Discussion

3.1 Dephosphorization of steel

For dephosphorization of steel in primary route of steel making, oxygen is used as oxidizing agent to oxidize impurities present in the steel. But in the case of induction furnace steel making was not having direct oxygen introducing in the bath. The impurities present in the steel is oxidized by introducing FeO in the ladle furnace. For oxidation of impurities and slag formation, FeO level is maintained by addition of sponge iron. The factors which catalyzed phosphorous transfer from metal to slag are high FeO content in the slag, high slag basicity, low temperature, high slag volume of good fluidity and removal of oxide slag prior to deoxidation, thereby preventing reversion.

The removal of phosphorus was due to the oxidation of phosphorus-to-phosphorus pentoxide. This phosphorous pentoxide is an acidic oxide and thus there was a requirement of basic oxide (CaO) to form a complex oxide as $CaO\text{-}P_2O_5$ (neutral oxide) to make complex oxide stable in slag phase. To neutralize above acidic oxide basicity of furnace should be maintained. The term basicity is defined as the ratio of basic oxide to acidic oxide (CaO/SiO_2) and the basicity of the steel bath is maintained by addition of calcined limestone.

The phosphorous removal reaction may be given by [16],

$[P] + 5/2[O_2] + 3/2(O^{-2}) = (PO^{-3}4)$ ……………………...(1)

The oxidation reaction of other elements may be written as

[Si]+ 2(FeO) → (SiO_2) + 2[Fe] ………………………..…...(2)
[Mn] + (FeO) → (MnO) + [Fe]………………………….….(3)
[C] + (FeO) → {CO} + [Fe] …………………...…...…(4)

To understand the dephosphorization in steel, two approaches were utilized: Taguchi Analysis and experimentation. Taguchi method was utilized to develop the DOE and to identify the proper parameters to optimize the process.

3.2 Experimental analysis by Taguchi method:

An experimental design based on the Taguchi method is proposed to achieve dephosphorization of steel as it is the most efficient way to determine the quality of the final product with respect to the process variables. In this experimental work, the Taguchi method was used to find the final phosphorus in steel in relation to the degree of dephosphorization, basicity and dephosphorization index. Experimental work was carried out based on different combinations of dephosphorization, basicity and dephosphorization index suggested by Taguchi analysis. A total of five experiments were performed in Minitab by varying the degree of dephosphorization, dephosphorization index and V-ratio. The L9 run was adopted in a 3-level design and the results are presented in Table 2. The initial 3x3 array values of V-ratio and dephosphorization index were taken from random experiments to obtain the L9 array.

Table 2. Taguchi analysis: Effect of process parameters on final % of phosphorous.

Experiment Number - 1

Sample	Degree of Dephosphorization	Dephosphorization Index	V-Ratio	Final % P	Initial % P
1	0.82	570.45	2.736	0.022	0.12
2	0.8	454.55	2.611	0.022	0.112
3	0.78	403.75	3.453	0.024	0.11
4	0.72	378.82	2.757	0.034	0.122
5	0.78	476.15	3.073	0.026	0.119
6	0.78	415.56	2.919	0.027	0.121
7	0.81	478.18	2.674	0.022	0.116
8	0.78	464.81	2.737	0.027	0.122
9	0.78	455.28	2.871	0.025	0.117

Experiment Number - 2

Sample	Degree of Dephosphorization	Dephosphorization Index	V-Ratio	Final % P	Initial % P
1	0.78	540	2.518	0.026	0.119
2	0.78	472.96	2.381	0.027	0.121
3	0.81	526.36	2.359	0.022	0.116
4	0.78	392.22	2.672	0.027	0.122
5	0.81	476.8	2.785	0.025	0.133
6	0.81	432.08	3.22	0.024	0.124
7	0.8	484.55	3.341	0.022	0.11
8	0.78	471.25	3.167	0.024	0.109
9	0.79	474.52	2.805	0.024	0.119

Experiment Number - 3

Sample	Degree of Dephosphorization	Dephosphorization Index	V-Ratio	Final % P	Initial % P
1	0.83	600.95	2.327	0.021	0.12
2	0.8	550.45	2.789	0.022	0.108
3	0.77	368.28	2.95	0.029	0.125
4	0.81	470.91	2.806	0.022	0.117
5	0.79	464.4	3.094	0.025	0.12
6	0.73	352.41	2.852	0.029	0.108
7	0.79	480	3.055	0.022	0.104
8	0.78	422.31	2.61	0.026	0.116
9	0.78	463.71	2.81	0.024	0.114

Experiment Number - 4

Sample	Degree of Dephosphorization	Dephosphorization Index	V-Ratio	Final % P	Initial % P
1	0.8	522.92	2.736	0.024	0.123
2	0.82	476.19	2.611	0.021	0.116
3	0.8	362.08	3.453	0.024	0.119
4	0.76	427.31	2.757	0.026	0.109
5	0.78	596.52	3.073	0.023	0.106
6	0.79	467.5	2.919	0.024	0.116
7	0.79	457.39	2.674	0.023	0.109
8	0.81	591.36	2.674	0.022	0.115
9	0.79	487.65	2.862	0.023	0.114

Experiment Number - 5

Sample	Degree of Dephosphorization	Dephosphorization Index	V-Ratio	Final % P	Initial % P
1	0.78	419.2	2.518	0.026	0.116
2	0.8	453.64	2.381	0.022	0.109
3	0.81	526.36	2.359	0.022	0.115
4	0.75	365.17	2.672	0.029	0.116
5	0.78	458.46	2.785	0.026	0.117
6	0.82	518.5	3.22	0.02	0.11
7	0.85	627.06	3.341	0.017	0.114
8	0.68	290	3.167	0.039	0.12
9	0.78	457.29	2.805	0.025	0.114

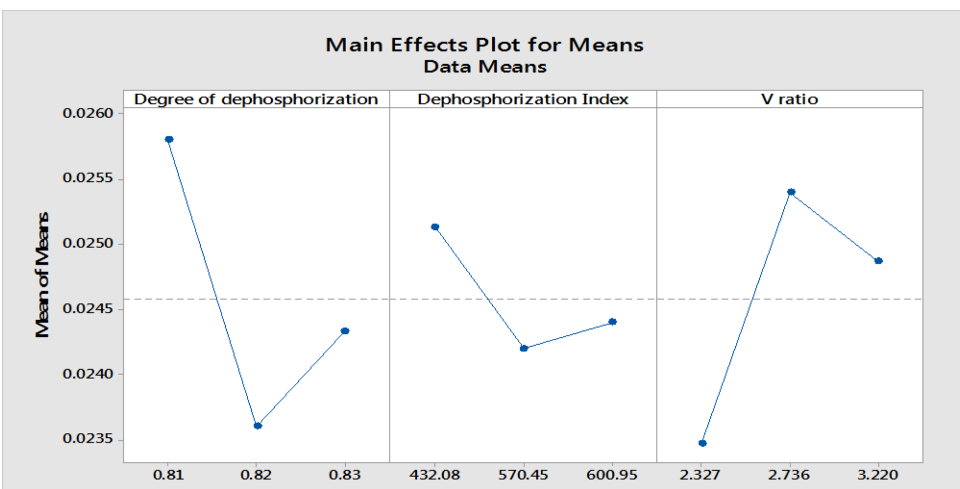

Fig. 2 Main effects plots showing the mean of mean for experiments 1-5

Figure 2 depicts the effect of process parameters on final % of phosphorous. It was observed from Mean of Mean plot of Taguchi method, the minimum value of mean of mean was achieved for lower i.e.is better for dephosphorization of steel by keeping degree of dephosphorization, dephosphorization index and V-ratio as 0.82, 570.45 and 2.327 respectively. The higher the value of degree of dephosphorization and dephosphorization index implies more entrapment of phosphorous in slag thus leading to higher the dephosphorization in steel. [16, 17] Higher the basicity in metal bath generates the atmosphere to activate the chemical reaction between calcium oxide and phosphorous which leads to more removal of phosphorous.

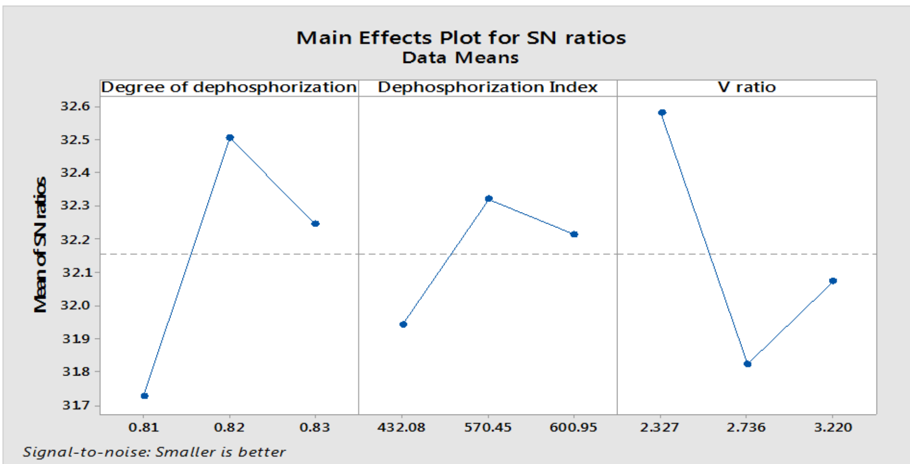

Fig. 3: Main effects plots for SN ratios for experiment 1-5

Figure 3 is a plot of the mean to Signal to Noise (SN) ratio which shows that the higher the value of the mean, the better will be the process. From the plot it is found that a higher value of noise ratio can be achieved by keeping the degree of dephosphorization, dephosphorization index and V-ratio at values of 0.82, 570.45 and 2.327 to obtain the desired dephosphorization of steel. This combination of parameters will give the best results for phosphorus removal in the induction furnace route via ladle refining. The same was observed in the experimentation which is discussed in the later section of this paper.

Table 3: Signal to noise ratio

Level	Degree of Dephosphorization	Dephosphorization Index	V ratio
Level-1	31.72	31.94	32.58
Level-2	32.51	32.32	31.82
Level-3	32.24	32.21	32.07
Delta	0.79	0.38	0.76
Rank	1	3	2

The mean of SN ratio was calculated for the variable DD, DI and V-ratio by using a mathematical expression given in equation 5

$$\text{Signal to noise ratio (smaller the better)} = -10log\frac{1}{n}\sum R^2 \quad \ldots\ldots\ldots\ldots\ldots(5)$$

Where, n = No. of observations

R = Observed data for each response

The table 3 reveals the result of signal to noise ratio at level-1, level-2 and Level-3 for degree of dephosphorization, dephosphorization index and V ratio. The delta values are the difference between the highest and lowest average response values of signal to noise ratio for each factor. Ranks are given based on the higher to lower value of the delta.

3.3 Contour Plot graph for dephosphorization

Contour plots are used to see how a response variable relates to two predictor variables. A contour plot provides a two-dimensional view in which all points that have the same response are connected to produce contour lines of constant responses. Contour plots are useful for investigating desirable response values and operating conditions. A contour plot contains the following elements: Predictors on the x- and y-axes. Contour lines connect points that have the same response value.

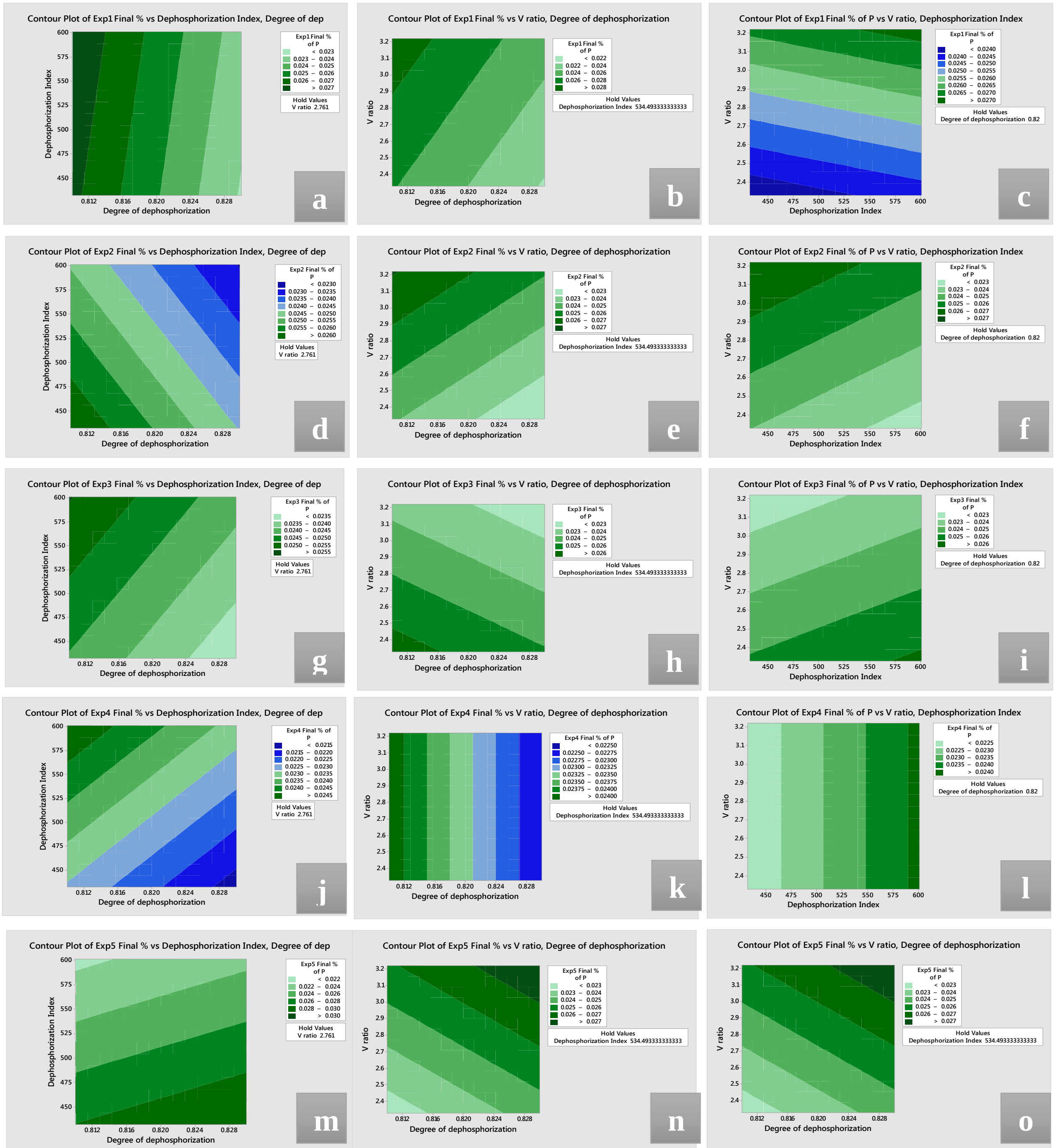

Fig. 4: Contour plot for Experiment 1: (a) DI Vs DD (b) V-ratio Vs DD (c) V-ratio Vs DI Experiment 2: (d) DI Vs DD (e) V-ratio Vs DD (f) V-ratio Vs DI, Experiment 3: (g) DI Vs DD (h) V-ratio Vs DD (i) V-ratio Vs DI, Experiment 4: (j) DI Vs DD (k) V-ratio Vs DD (l) V-ratio Vs DI, Experiment 5: (m) DI Vs DD (n) V-ratio Vs DD (o) V-ratio Vs DI

This contour plot shows the relationship between DI and DD, V ratio and DD, V ratio and DI, parameters used to achieve high removal of phosphorus from steel. lighter regions in the contour plots indicate higher quality. These lighter response values seem to form a right combination of DI, DD and V ratio to achieve the high dephosphorization of steel. The dark-colored portion in the various contour plots gives the information about the ineffective condition for phosphorous removal. The data from the contour plot can provide us clear insights about the right recipe for achieving higher dephosphorization in steel.

For the experiment 1, in Fig (4a) lighter regions indicate higher possibility of dephosphorization in DI vs DD. The condition for higher response for phosphorous removal in the steel is shown by a ridge of upper right to lower right of the plot. In Fig (4b), Lighter regions indicate higher possibility of dephosphorization in V Ratio vs DD. These higher response values seem to form a patch from the upper right to the lower right of the graph. In Fig (4c), Dark blue regions indicate higher possibility of dephosphorization in V Ratio vs DI. These higher response values seem to form a patch from the Lower left to the lower middle of the graph. The similar trends were also observed for experiment 2,3 ,4 and 5.

In order to validate the Taguchi method a detailed experimentation was also performed in small induction furnace. The experimental trials have been performed to achieve the maximum level of dephosphorization in the ladle refining furnace. A total of forty heats (experiments) were performed using hot metal made from pig iron and sponge iron. Throughout every individual heat, the sample of liquid metal was collected. The initial composition of steel, final phosphorous content of steel and slag composition were examined. According to literature available, the dephosphorization will occur only when the slag basicity and FeO content in the steel bath is high [17]. Gaurav et.al. studied the removal of phosphorous from the steel in induction furnace. They have achieved the degree of dephosphorization upto 55.55% by maintaining the FeO content within the slag in the range of 15–35% for effective dephosphorization.[18]

Bedarkar et.al. conjointly studied the dephosphorization of steel and found the degree of dephosphorization 77.53% and FeO content within the slag was 31.08 % for effective dephosphorization and basicity was 2.05 [16]. In this current work the basicity and FeO content has been maintain in the range of 2.757 - 3.453 and 32.52 - 41.36 % respectively to achieve dephosphorization of steel in the range of 72 - 85%. To achieve a similar degree of dephosphorization, the dephosphorization index, phosphorous distribution ratio was kept in between 362.08 - 600.95. For dephosphorization to occur a lowest possible temperature is required to maintain [19]. This has been maintained in this work and kept in the range of 1570 - 1590˚C to achieve high dephosphorization.

The experimental calculations used are discussed below. Similar kind of calculations were made for all the heats.

Experimental calculation:

Weight of steel used for dephosphorization = 32T

(i) Opening Phosphorous in liquid metal before entering in to LRF = 0.120%

Lime added for dephosphorization = 132 Kg

Temperature maintained during addition: 1580˚C

Closing Phosphorous in liquid metal = 0.022%

P_2O_5 in Steel Slag = 12.55% , CaO in Steel Slag = 26.87%, SiO_2 in Steel Slag = 9.82%

- $\text{Degree of Dephosphorization} = \frac{\text{Initial phosphorous content} - \text{Final phosphorous content}}{\text{Initial phosphorous content}}$

  Degree of Dephosphorization = (0.120-0.022)/0.120 = 0.816 = 81.6%

- $\text{Dephosphorization Index} = \frac{\text{Phosphorous content in slag}}{\text{Phosphorous content in final steel}}$ = 12.55/0.022 = 570.45

- $\text{Basicity of Steel Slag} = \frac{\text{Basic oxide}}{\text{Acidic Oxide}} = \frac{\text{CaO}}{\text{SiO2}}$ = 26.87/9.82 = 3.736

From the 40 experiments, the results of best five are shown here to exhibit the obtained trend.

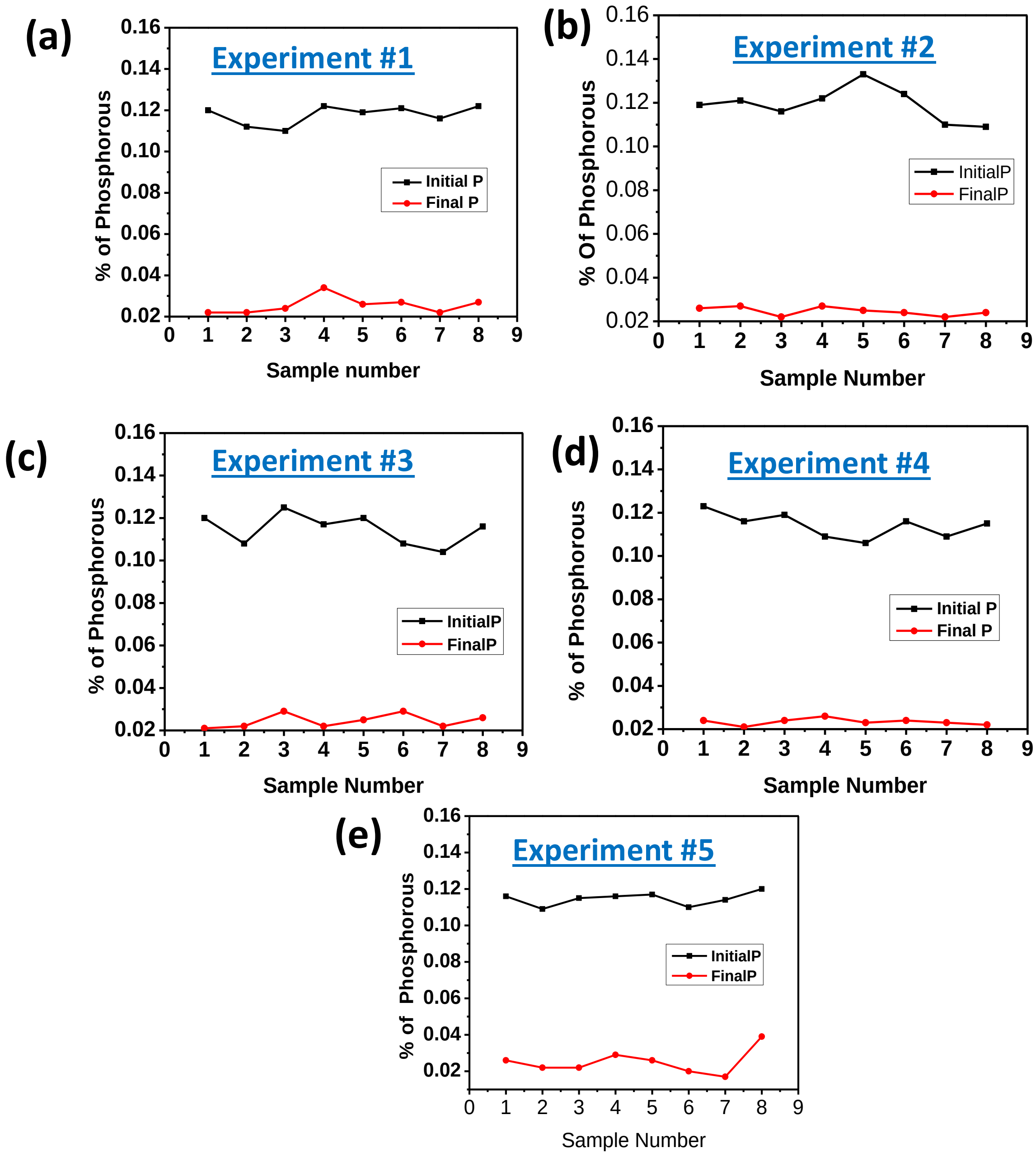

Fig. 5: Above graph shows the variation of P in raw steel and final steel with respect to sample number. Variation of P in raw steel ranges between 0.106- 0.123 % and in final steel 0.021- 0.039 %.

Figure 5 shows the initial and final phosphorous values in which the phosphorous content in raw steel initially were in the range of 0.106- 0.123 % and in final steel was 0.021- 0.039 %. The achievement of highest percentage of dephosphorization in this process was 85% by maintaining the basicity 3.341 and dephosphorization index 627.06 in the molten bath. While comparing with the Taguchi analysis explained above, it confirms that selecting the appropriate basicity of the bath to activate the phosphorous to transfer from steel to slag and make themselves stable in slag phase.

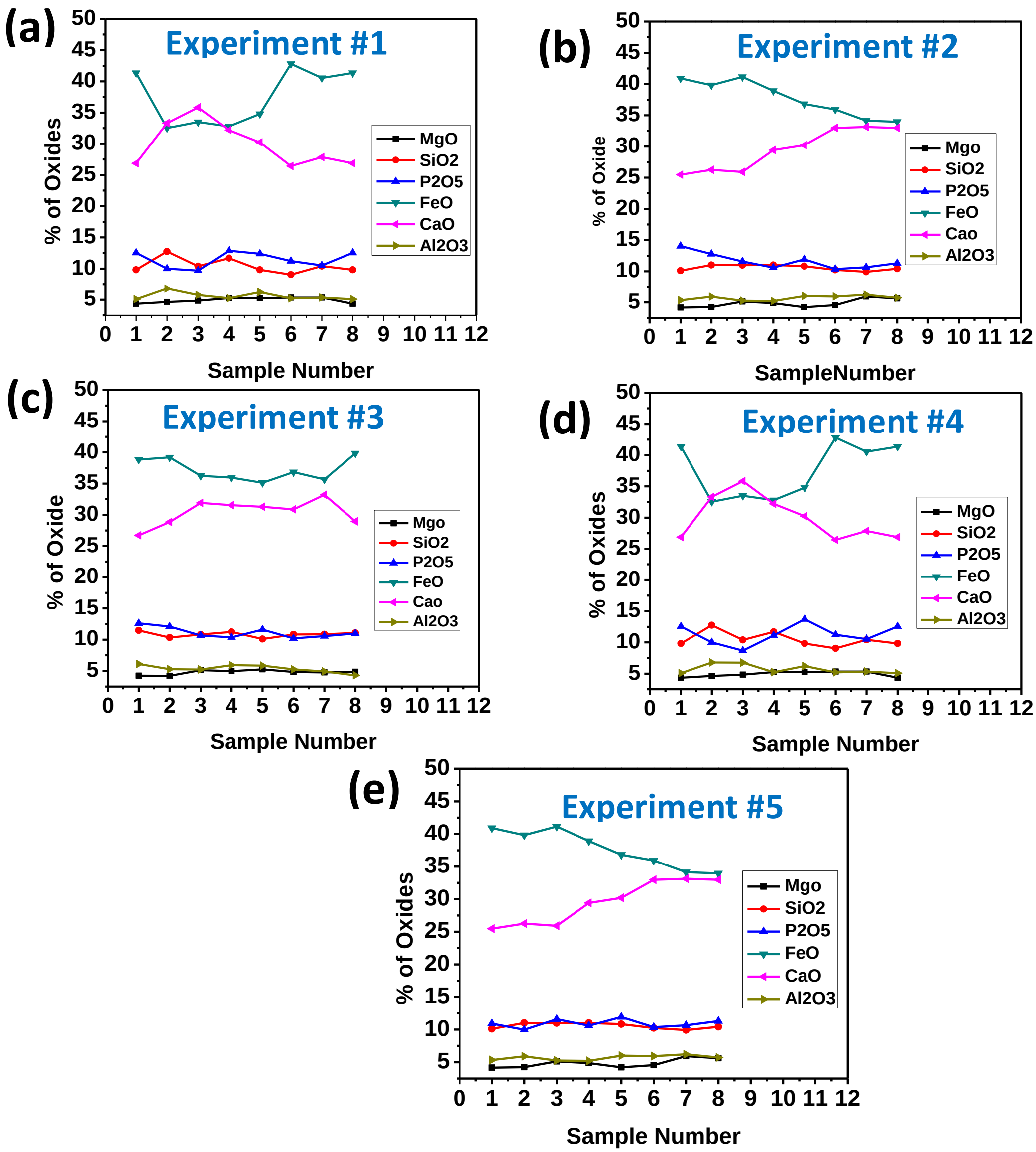

Fig. 6: Above graph shows the variation of MgO, SiO_2, P_2O_5, FeO, CaO and Al_2O_3 in slag with respect to slag sample and the variations are MgO: 4.15- 5.92%, SiO_2: 9.05- 12.76%, P_2O_5: 9.69- 12.88%, FeO : 32.52- 41.36 % ,CaO : 25.46- 33.32% and Al_2O_3 :4.29 -6.77 %.

Figure 6 shows the of oxide constituent of slag phase such as MgO, SiO_2, P_2O_5, FeO, CaO and Al_2O_3 .These all oxides were present in this range as MgO: 4.15- 5.92%, SiO_2: 9.05- 12.76%, P_2O_5: 9.69- 12.88%, FeO : 32.52- 41.36 % ,CaO : 25.46- 33.32% and Al_2O_3 :4.29 -6.77 %. To transfer phosphorous from liquid steel to slag there is demand of high FeO presence and the FeO content were maintained between 32.52-41.36% to achieve the highest degree of dephosphorization. Only the FeO and CaO contents vary significantly and are the main cause for achieving the maximum dephosphorization. The FeO and CaO contents need to be maintained higher for the better removal of the Phosphorus. As per the Bedarkar et al. work, the degree of dephosphorization has been achieved up to 77.53 for 31.80 % FeO in slag [14].

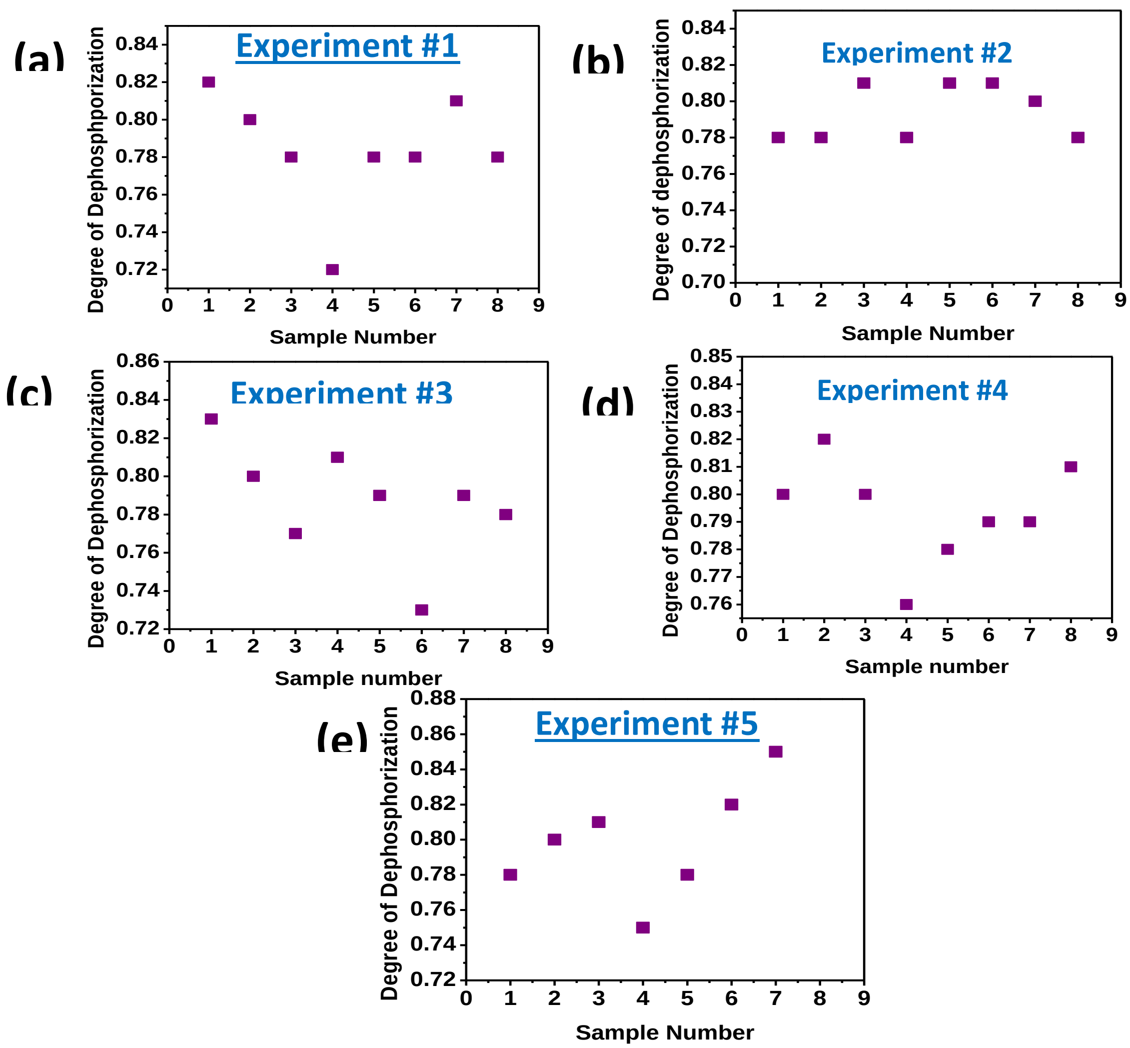

Fig. 7: Graph shows the variation of degree of dephosphorization respective to sample and variations which are in the range between 0.72- 0.85.

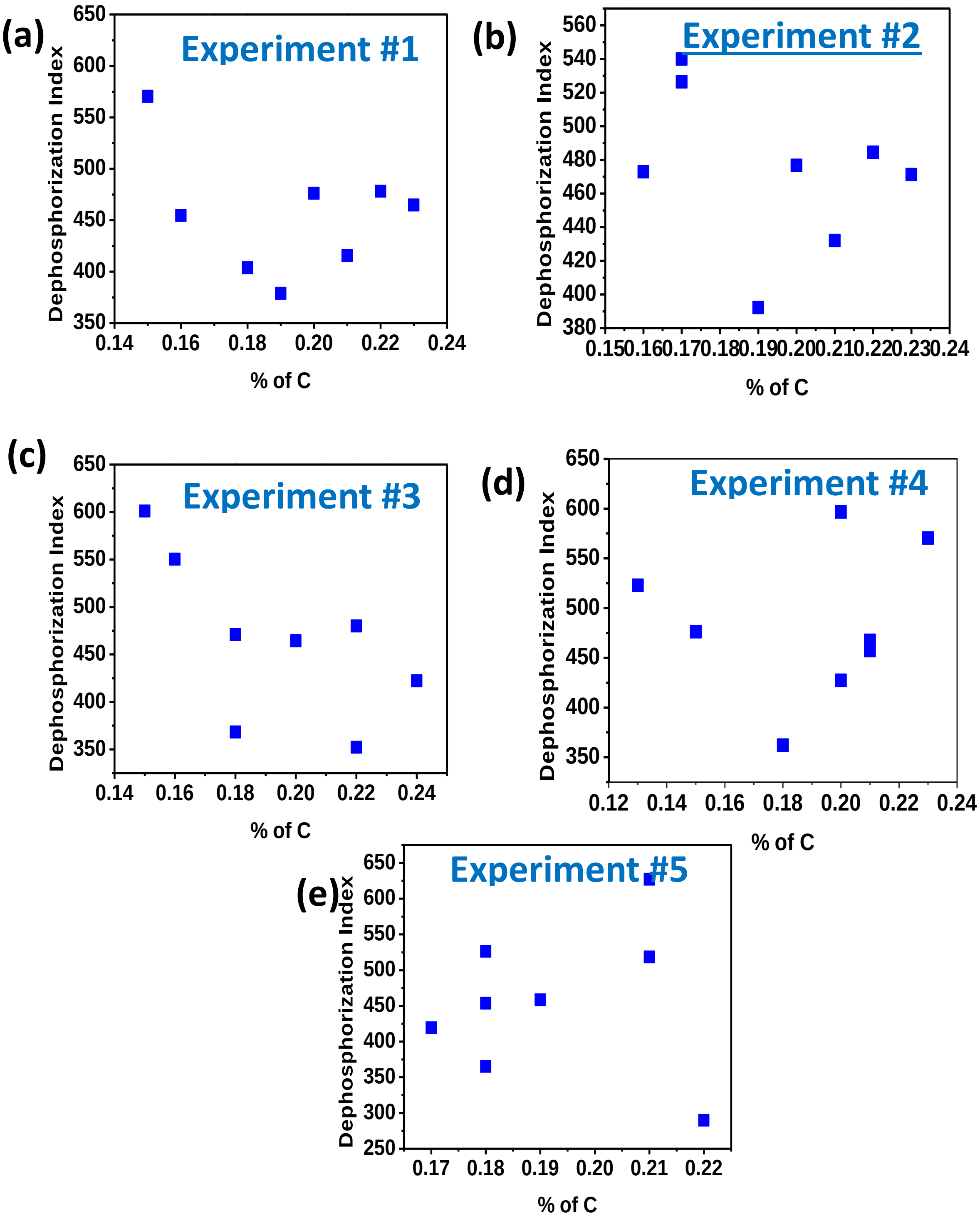

Fig. 8: Graph shows the variation of dephosphorization index and variations are ranges between 290- 627.06.

Fig. 7 and 8 shows the degree of dephosphorization and the dephosphorization index. These were calculated using the formulas mentioned earlier. The highest degree of dephosphorization and dephosphorization index were achieved 85% and 627.06 respectively and this has been achieved by maintaining the desired condition for removal of phosphorous in the bath. It was possible only by maintaining high basicity and high amount of FeO content in the bath. The variations in the values observed is due to the variation in the chemical compositions in the raw steel. The initial P is not constant and the slag prepared has quite good variation in the FeO and CaO contents.

Finally, it could be understood that the values obtained by the Taguchi method are in line with the experimentations conducted in the lab scale. This confirms that one can use the Taguchi method to design the experiment and use the optimization to obtain the values. This kind of approach will not only reduce the costly trails in plants but also provide the industries to play with the parameters prior to the study and provide deep understanding on the concepts occurring in the steel bath.

4. Conclusions

Dephosphorization of steel through induction and LRF route was studied using the experimental part as well as the design of experiment was verified by Taguchi method. Following conclusions were drawn through the theoretical as well as experimental trails:

1. By utilizing Induction and LRF route, the project cost can be decreased as compared to the traditional Arc furnace route. Further can have the advantages of less start up time as compared to the primary steel making route and less foot print generation as compared to the BOF and Arc furnace plants.
2. Based on analysis of Mean of Mean plot of Taguchi method, it was observed that the minimum value of mean of mean was achieved for dephosphorization of steel by keeping degree of dephosphorization, dephosphorization index and V-ratio as 0.82,570.45 and 2.327 respectively as the process was customized for lower is better.

3. Based on analysis of SN ratio, it was observed that optimum factor levels for dephosphorization of steel were obtained at 0.82, 570.45 and 2.327 degree of dephosphorization, dephosphorization index and V-ratio respectively.
4. The obtained maximum degree of dephosphorization using the induction furnace route was 85.09%.
5. The work provides a new insight of effective dephosphorization through LRF route with minimum capital investment. The highest dephosphorization index achieved in this route was 627.06.

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