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Mathematical Analysis of Gaseous Reduction of Fe_2O_3-MnO_2-SiO_2 Mixed Oxides

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ABSTRACT:*Low grade iron ores are often contaminated with relatively high percentage of different impurity gangue minerals. The iron ores contaminated with manganese oxide and silica are hardly reducible and consume more energy in the integrated steel plant. Therefore it is important to estimate and predict the influence of manganese oxide, silica and temperature on the reduction yield of iron oxide using mathematical model approach. In the current study, a* 2^3 *(three-parameters, two-levels)factorial design is applied on the gaseous reduction experimental data of mixed oxides (*Fe_2O_3*-*MnO_2*-*SiO_2*) to build a linear regression model. The calculations have been performed using Matlab program. The developed mathematical model indicated that* SiO_2 *and temperature have positive effect on the reduction yield of iron oxide. On the other hand,* MnO_2 *exhibited the highest negative impact on the reduction yield of iron oxide followed by the interaction coefficient of* MnO_2*,* SiO_2 *and temperature. The results of the developed mathematical model are fitted to the experimental reduction data of mixed oxides.*

Keywords:*factorial design; modelling; mixed oxides; gaseous reduction; mathematical analysis*

I. Introduction

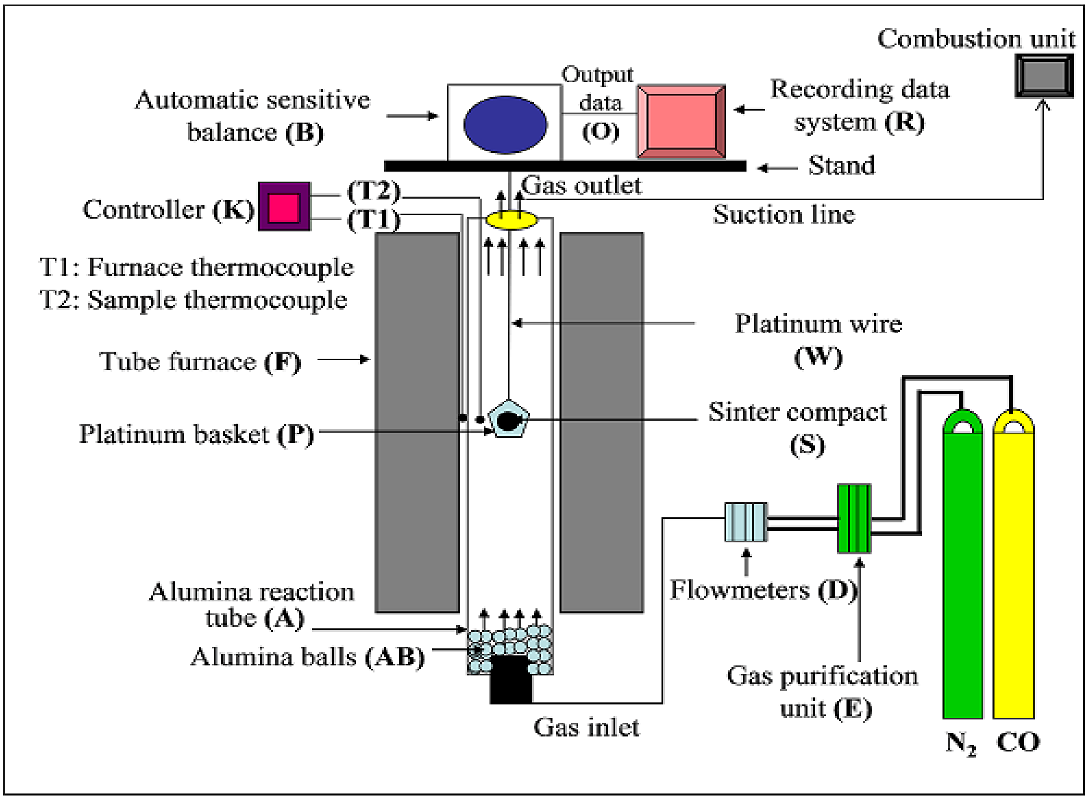
The quality of iron ore is a crucial factor that affecting the product quality, production rate and total cost of final steel product. The intensive mining of iron ores over many decades has resulted in a depletion of the high grade resources and increased its prices in the global market. Recently, utilization of low and medium grade iron ores became necessary in steel industry to overcome the shortage of the high grade ores. In addition, intensive work is being done to efficiently recycle iron waste materials which are generated in the integrated steel plants. The Egyptian iron ores are classified as low to medium grade ores which are often contaminated with relatively high percentage of manganese oxide (0.03-13.7%) and silica (0.07-13.7%) beside many other residues [1]. These impurities have a negative impact on the steel production rate and resulted in high energy consumption, low product quality and high production cost. Intensive experimental work has been carried out to evaluate the reduction kinetics and mechanism of SiO_2-MnO_2 doped Fe_2O_3 compacts [2-5]. The reduction rate was enhanced by SiO_2 at the beginning while reduction retardation appeared at the final stages. This was attributed to the formation of low reducible fayalite (Fe_2SiO_4) phase. In MnO_2-SiO_2-doped Fe_2O_3 compacts, the reduction rate was greatly hindered due to the formation of hard reducible fayalitemanganoan [$(Fe,Mn)_2SiO_4$]. The behavior of manganese oxides during magnetizing reduction of Baharia iron ore by CO/CO_2 gas mixtures at 600-1000^oC was studied [6]. It was found that the reduction of $MnO_2$is stopped at MnO phase. The reduction of mixed iron-manganese oxides in H_2 and CO atmosphere indicated that the presence of manganese oxide has slowed the reduction rate of iron oxide [7]. The kinetics of hydrogen reduction of iron-manganese mixed oxides and the carbothermic reduction of $MnFe_2O_4$have been investigated [8]. The reduction with H_2 in absence of carbon led to the formation of an intermediate product between MnO and FeO at 507^oC which has been finally reduced to MnO and metallic Fe. In presence of carbon, methane was formed at 800^oC and decomposed at about 1050^oC to nascent carbon which accelerated the reduction process. The reduction behaviour of high manganese iron ore (9.9 wt.% MnO_2) by H_2 at 800-1000^oC has been investigated [9]. The reduction rate was increased with temperature and decreased as the time proceeded.

The previous survey summarized various investigations which have been conducted experimentally to estimate the effect of manganese oxide and silica on the reduction rate of iron oxides. However, the effective magnitude of these oxides on the reduction rate of iron oxides at different temperature is still required. Moreover, the influence of the interaction of different parameters on the reduction yield of iron oxide is difficult to be experimentally evaluated. The statistical analysis is able to precisely estimate the effect of each individual parameter and its interaction with the other parameters on the reduction yield of iron oxide. A regression model has been developed based on factorial design approach. The factorial design has several advantages such as prediction of process yield and process performance and it can be also modified for further applications in the iron and steel making processes [10-15]. In the current study, a 2^3 (three-parameters, two-levels) factorial design is used to investigate the impact of MnO_2, SiO_2 and temperature on the reduction yield of iron oxide. The interaction coefficient between mixed oxides and temperature is statistically analysed.

II. Materials and Methodology

A2^3factorial design has been applied on the experimental data of pure $Fe_2O_3$and its mixture with MnO_2 and SiO_2. The reduction was conducted with CO at various temperatures [3]. Chemically high grades (> 99.5%) Fe_2O_3, MnO_2 and SiO_2 powders (< 50 µm) were used to eliminate the influence of any other impurities that are usually associated with iron ores. The additions of MnO_2 and/or SiO_2 to Fe_2O_3 were 6.0 and 7.5 wt.% respectively. These values have been used to simulate the average of MnO_2 and SiO_2 percentage in the Egyptian iron ores. The powders are mixed in a ball mill for 8.0 hours to attain a complete homogeneous mixture. The oxides mixtures are moistened with 6 wt.% naphtha and then pressed in a cylindrical mould at 10 kN. The dry compacts (9.0 mm diameter, 14 mm height) are gradually heated in a muffle furnace (10 K/min) up to 1200^{o}C and kept at this temperature for 6.0 hours. After firing, the compacts are gradually cooled down to room temperature to avoid the thermal shock. Pure Fe_2O_3 and mixed oxides (Fe_2O_3-MnO_2-SiO_2) compacts were isothermally reduced with pure CO gas (1.0 l/min) at 800-1100^{o}C. The reduction trial shave been conducted in tube furnace as can be schematically shown in Figure 1. The system consists of an automatic sensitive balance (B) equipped with a vertical tube furnace (F). An alumina reaction tube (A) was fitted inside the furnace. The output of the balance (O) is connected to the recording system (R) for continuous measurement of weight loss during the reduction as a function of time until the compact weight becomes constant. The flow rate of CO gas is adjusted using flow meter (D). The actual temperatures of the furnace (T1) and sample (T2) are measured with controller (K).Purified N_2 at constant flow rate of 1.0 l/min is introduced in the reaction tube during the heating up of the furnace to the predetermined temperature. At the applied temperature, the compact (S) is placed in a platinum basket (P) and is suspended from the balance arm by a platinum wire (W). The compact is positioned in the middle hot zone of the tube furnace. After soaking the sample at the ambient temperature for 10 min, CO gas is applied for the reduction of sinter. When the sample weight becomes constant, the reducing gas is stopped and pure N_2purged in the reaction tube then the reduced compact is pulled up to the cooled zone of the reaction tube and quickly dropped out in a conical flask containing acetone to prevent the re-oxidation.

Figure 1. Schematic diagram of the reduction system



The experimental reduction behaviour of pure Fe_2O_3 and mixed oxides are discussed elsewhere [3]. In the current study, a 2^3factorial design is applied on the experimental data of the reduction trails. The reduction yield of pure Fe_2O_3 and mixed oxides compacts after 20 min at 800°C and 900°C is given in Table 1. The reduction yield is determined depending on the reducible oxygen in the sample as given in Eq. 1.

$$Reduction\ yield\ (\%) = \frac{W_1 - W_2}{W_o} * 100 \quad (1)$$

where: W_1 is the weight of the sample before reduction, W_2 is the weight of sample after reduction and W_o is theweight of reducible oxygen.

Table 1.Conditions of experiments and reduction yield of iron oxide

Trail No.	MnO_2, wt. %	SiO_2, wt. %	Fe_2O_3, wt. %	Temperature, °C	Reduction yield of iron oxide, %	
					1st	2nd
1	0	0	Rest	800	39.9	40.1
2	6	0	Rest	800	33.5	33.6
3	0	7.5	Rest	800	44.9	45.1
4	6	7.5	Rest	800	34.8	35.2
5	0	0	Rest	900	41.5	41.7
6	6	0	Rest	900	37.7	37.9
7	0	7.5	Rest	900	48.0	48.2
8	6	7.5	Rest	900	33.4	33.6

The experiments have been conducted two times under the same conditions and the reduction yield has been calculated. A 2^3 factorial design is applied to evaluate the effect of MnO_2, SiO_2 and temperature on the reduction yield. Mathematical analysis of MnO_2, SiO_2, temperature and their interactions effect on the reduction of iron oxide is addressed. Regression model is formulated based on the factorial design approach to calculate the reduction yield in terms of MnO_2, SiO_2 and applied temperature.

III. Results and Discussion

3.1. Definition of the controlling parameters

The controlling parameters which are considered in the applied 2^3 factorial design are including the effect of MnO_2 and SiO_2 as well as the effect of temperature on reduction process of iron oxide.

By convention, the effect of a factor was donated by a capital Latin letter. Thus "A" refers to the effect of MnO_2, "B" refers to the effect of SiO_2, "C" refers to the effect of reduction temperature, "AB" refers to the interaction effect of MnO_2 and SiO_2, "AC" refers to interaction effect of MnO_2 and temperature, "BC" refers to the interaction effect of SiO_2 and temperature, while "ABC" refers to the interaction effect of MnO_2, SiO_2 and temperature. The low and high levels of A, B and C are denoted by "–"and "+" respectively. The eight treatment combinations in the design are usually represented by lowercase letters. The high level of any factor in the treatment combination is denoted by the corresponding lowercase letter (a, b, c, ab, ac, bc and abc), while the low level of any factor will be indicated by the absence of its corresponding letter. Thus, "a" represents the treatment combination of A at high level when B and C at low levels, "b" represents B at high level when A and B at low levels, "ab" represents A and B factors at the high levels when C at low level, "abc" represents A, B and C factors at the high levels, finally (1) is used to denote all factors at low level. The average effect of any factor can be defined as the change in response which is produced by changing the factor level divided by the averaged levels of the other factors. The symbols (1), a, b, ab, c, ac, bc, abc represent the total of all 2 replicates which have been taken at the treatment combination i.e. (n=2).

3.2 Mathematical formulations

Mathematical formulations are used to estimate the effect of different parameters on the reduction yield. The effect of A at low level of B and C is [a-(1)]/n, the effect of A at high level of B and C is [abc-bc]/n, the effect of A at low level of B and high level of C is [ac-c]/n and the effect of A at high level of B and low level of C is [ab-b]/n. The main effect of A is the average values of its effect at low and high levels of B and C as given in Eq. 2.

$$A = \frac{1}{4n}[(a + ab + ac + abc) - ((1) + bc + c + b)] \quad (2)$$

The average effect of B and C can be calculated by the same manner of A as given in Eqs. 3 and 4 respectively.

$$B = \frac{1}{4n}[(b + ab + bc + abc) - ((1) + a + ac + c)] \quad (3)$$

$$C = \frac{1}{4n}[(c + ac + bc + abc) - ((1) + a + b + ab)] \quad (4)$$

The binary interaction effects of the factors A and B represented in AB is one half of the difference between the averages effect of factor A at the two levels of factor B. The average effect of factor A at high level of factor B is [(abc-bc)+(ab-b)]/2n, and the average effect of factor A at low level of factor B is [(ac-c)+(a-(1))]/2n. Based on this the binary interaction AB effect can be given in Eq. 5.

$$AB = \frac{1}{4n}\left[\left(abc + ab + c + (1)\right) - \left(bc + b + ac + a\right)\right] \quad (5)$$

In similar way, the average binary interaction effect of AC and BC can be calculated as give in Eqs. 6 and 7 respectively.

$$AC = \frac{1}{4n}\left[\left(abc + ac + b + (1)\right) - \left(ab + c + bc + a\right)\right] \quad (6)$$

$$BC = \frac{1}{4n}\left[\left(abc + bc + a + (1)\right) - \left(ac + b + ab + c\right)\right] \quad (7)$$

The ternary interaction effects of factor A, B, and C represented in ABC is defined as the average difference between binary interaction effects ABat two different levels of C as given in Eqs. 8 and 9.

$$ABC = \frac{1}{4n}[(abc - bc) - (ac - c) - (ab - b) + (a - (1))] \quad (8)$$

$$ABC = \frac{1}{4n}[(abc + c + b + a) - (bc + ac + ab + (1))] \quad (9)$$

3.3 Application of factorial design

Plus and minus signs that can be developed from the contrasts are summarized in Table 2. The high level is referred by plus sign and low level is referred by minus sign. The signs of identity element are plus.

Table 2. Algebraic signs for calculating effects in the 2^3 Design

Treatment combination	Factorial Effect							
	I	A	B	AB	C	AC	BC	ABC
(1)	+	-	-	+	-	+	+	-
a	+	+	-	-	-	-	+	+
b	+	-	+	-	-	+	-	+
ab	+	+	+	+	-	-	-	-
c	+	-	-	+	+	-	-	+
ac	+	+	-	-	+	+	-	-
bc	+	-	+	-	+	-	+	-
abc	+	+	+	+	+	+	+	+

Sum of squares (SS) for the effect of parameters in 2^3factorial design with n replicates is SS=(contrast)^2/8n. The total sum of squares (SS_T) has (abcn-1) degree of freedom while the error sum of squares (SS_E) has abc(n-1) degree of freedom. It can be calculated by using Eqs. 10 and 11 respectively. The avearge effect of the different parameters and their interactions are calculated and given in Table 3.Thesum of squares, mean square and the absolute magnitude effect is given in Table 4.

$$SS_T = \sum_{i=1}^{2}\sum_{j=1}^{2}\sum_{k=1}^{2}\sum_{n=1}^{n} y_{ijk}^2 - \frac{y_{...}{}^2}{4n} \quad (10)$$

$$SS_E = SS_T - SS_A - SS_B - SS_C - SS_{AB} - SS_{AC} - SS_{BC} - SS_{ABC} \quad (11)$$

Table 3. Values of the average effect of all parameters

Trail No.	Effective Parameter	Variant	Average of reduction yield, %	No. of applied equation	Average effect of parameters
1	(1)	(1)	40	---	---
2	A (MnO_2%)	a	33.55	2	-8.7125
3	B (SiO_2%)	b	45	3	2.1625
4	AB (MnO_2% & SiO_2%)	ab	35	4	1.8625

5	C (Temperature)	c	41.6	5	-3.5875
6	AC (MnO_2& Temperature)	ac	37.8	6	-0.4875
7	BC (SiO_2% & Temperature	bc	48.1	7	-1.0625
8	ABC (MnO_2% & SiO_2% & Temperature)	abc	33.5	9	-1.8125

Table 4. Analysis of variances

Source of Variance	**Average Effect**	**Sum of Square (SS)**	**Degree of Freedom**	**Mean Square MS**	**F_o (magnitude effect)**
A (MnO_2%)	-8.7125	303.6306	1	303.6306	11849
B (SiO_2%)	2.1625	18.70563	1	18.70563	729.9756
C (Temp.)	1.8625	13.87563	1	13.87563	541.4878
AB	-3.5875	51.48063	1	51.48063	2009
AC	-0.4875	0.950625	1	0.950625	37.09756
BC	-1.0625	4.515625	1	4.515625	176.2195
ABC	-1.8125	13.14063	1	13.14063	512.8049
Error	-----	0.205	8	0.025625	---
Total	-----	406.5044	15	---	---

From the previous calculations, the magnitude and direction of the factors can be examined to determine which variable is important and more effective. The main effect of A is negative with large magnitude. This means that the higher MnO_2% in the oxide mixture, the lower reduction yield is. Also, it is clear that the main effect of B is positive with relatively large magnitude comparedwith the magnitude of C. This means that SiO_2 has a significant positive effect on reduction yield of iron oxide. Temperature has positive effect with small magnitude on the reduction yield compared to that of SiO_2. On the other hand, the effect of the interaction combination of AB (MnO_2 and SiO_2) is negative with relatively large magnitude. This means that the increasing both of SiO_2 and MnO_2in the mixed oxide mixture affects negatively the reduction yield. The interaction combination of BC (SiO_2 with temperature) and AC (MnO_2 with temperature) are negative with relatively small magnitude. This indicates that SiO_2 with temperature and MnO_2 with temperature are retarded the reduction process of iron oxide for a certain extent. Finally, the interaction combination of the three factors ABC (MnO_2, SiO_2, and temperature) is negative with relatively high magnitude. This indicates that the high levels of MnO_2, SiO_2 and temperature are retarded the reduction rate of iron oxide.

The contrast coefficient has been used to estimate the effect of the individual and interacted parameters as summerized in Table 5. The contrast coefficient is either (+1) or (-1) referring to the maximum and minimum level of the parameter respectively.

Table 5. Contrast coefficients of effects

Effects	**(1)**	**a**	**b**	**ab**	**c**	**ac**	**bc**	**abc**
A	-1	+1	-1	+1	-1	+1	-1	+1
B	-1	-1	+1	+1	-1	-1	+1	+1
AB	+1	-1	-1	+1	+1	-1	-1	+1
C	-1	-1	-1	-1	+1	+1	+1	+1
AC	+1	-1	+1	-1	-1	+1	-1	+1
BC	+1	+1	-1	-1	-1	-1	+1	+1
ABC	-1	+1	+1	-1	+1	-1	-1	+1

The results of the experimental trails can be expressed in terms of regression model as given in Eq. 12.

$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 + \beta_{123} x_1 x_2 x_3 + \epsilon$(12)

Where: x_1,x_2 and x_3 are coded variables that represent the MnO_2%, SiO_2% and temperature respectively and $\beta_s'$are regression coefficients.β_0 is the intercept which is the grand average of all 16 observations (i.e.β_0=39.31875), the regression coefficientsβ_1, β_2 and $\beta_3$are one-half the corresponding factors A, B, and C respectively (β_1= -4.35625, β_2= 1.08125 &β_3= 0.93125), the regression coefficients β_{12}, β_{13}, β_{23} and $\beta_{123}$are one-half the corresponding factors AB, AC, BC and ABC respectively which are (β_{12}= -1.79375, β_{13}= -0.24375, β_{23}= -0.53125 &β_{123}=-.0.90625) and ϵ is the residual (the difference between observed and fitted point of the design). Based on this values, Eq. 12 can be modified as given in Eq. 13.

$Reduction\ yield = 39.31875 - 4.35625x_1 + 1.08125x_2 + 0.93125x_3 - 1.79375x_1x_2 - 0.224375x_1x_3 - 0.53125x_2x_3 - 0.90625x_1x_2x_3 + \epsilon$ (13)

To calculate the predicted reduction yield and to estimate the residual (ϵ) at (1), a, b, c, ab, ac, bc and abc; the sign of coded variables have been taken from Table 2. The resuts are presented in Table 6. The average residence is ± 0.10625 which can be neglicted.

Table 6. Actual and predicted reduction yield at different conditions

Variable	Predicted reduction yield, %	Actual reduction yield, %		Residence (ϵ)		The residence variation
		1st	2nd	1st	2nd	
(1)	40.002	39.9	40.100	-0.102	0.098	± 0.1
a	33.548	33.5	33.600	-0.048	0.052	± 0.05
b	45.002	44.9	45.100	-0.102	0.098	± 0.1
ab	34.998	34.8	35.200	-0.198	0.202	± 0.2
c	41.598	41.5	41.700	-0.098	0.102	± 0.1
ac	37.802	37.7	37.900	-0.102	0.098	± 0.1
bc	48.098	48	48.200	-0.098	0.102	± 0.1
abc	33.502	33.4	33.600	-0.102	0.098	± 0.1

The relation between the natural and coded variables can be defined as follow: the coded variable is equal to [(natural variable – 1/2(variable at high level + variable at low level)) / 1/2(variable at high level + variable at low level)]. Based on this concept the reduction yield can be predicted in terms of MnO_2 and SiO_2 concentration with temperature as given in Eq. 14.

$Reduction\ yield =$
$35.2144 + 0.0066T - 0.9483[SiO_2\%] - 7.2798[MnO_2\%] + 1.215[MnO_2\%][SiO_2\%] + 0.0075T[MnO_2\%] + 0.002T[SiO_2\%] - 0.0016T[MnO_2\%][SiO_2\%]$
(14)

where; T is the temperature in ^{o}C

This equation covering the concentration of MnO_2 and SiO_2form 0 to 6.0% and from 0 to 7.5% respectively within temperature range 800-900^{o}C. This full regression formulation indicates that the reduction yield is not only dependent on the individual parameter (MnO_2, SiO_2, or temperature) but also on the mutual interactions between all of these parameters.

3.4 Validation of regression model

In order to verify the previously described regression model, the reduction yield has been calculated using the coded and the actual variables of mixed oxides. The results are compared to that of experimetal results as can be seen in Figure 2. It can be seen that the calculated reduction yield is very close to that of the experimental values. The maximum reduction yield is obtained at the highest concentration of SiO_2 (7.5% wt.%) at 900°C while the lowest reduction is demonstrated at the highest MnO_2 level (6.0 wt.%) at 800°C.

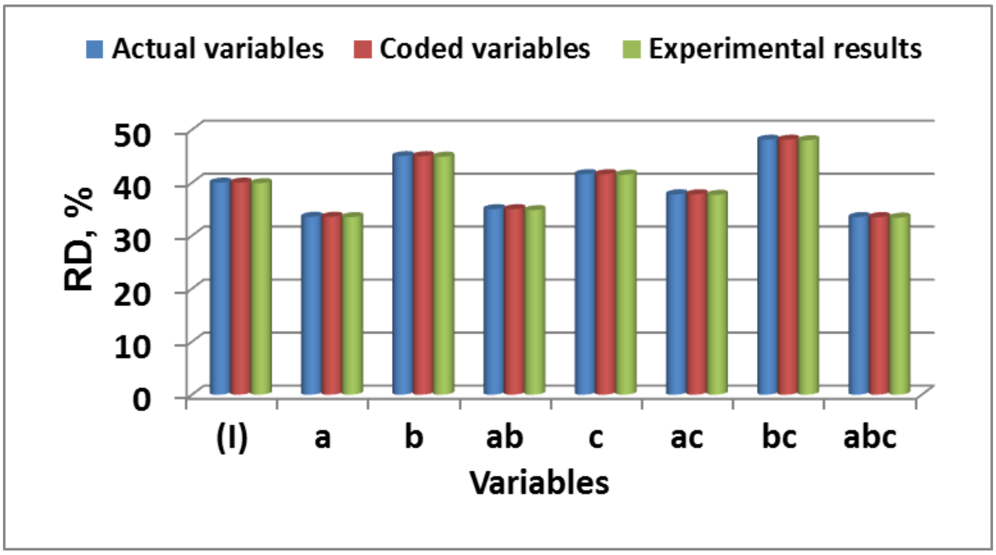


Figure 2. Actual and predicted reduction yield by using actual and coded variables

The effect of MnO_2 and SiO_2 on the reduction yield of Fe_2O_3 at 800°C and 900°C is given in Figure 3 and Figure 4 respectively. In both cases, the maximum reduction yiled is exhibited by Fe_2O_3 mixed with 7.5 wt.% SiO_2. On the other hand, the iron oxide mixed with 6 wt.% MnO_2 showed the lowest reduction yield at 800°C. At 900°C, the lowest reduction yield at 900°C is demonstrated by the iron oxide mixed with 6 wt.% MnO_2 and 7.5 wt.% SiO_2.

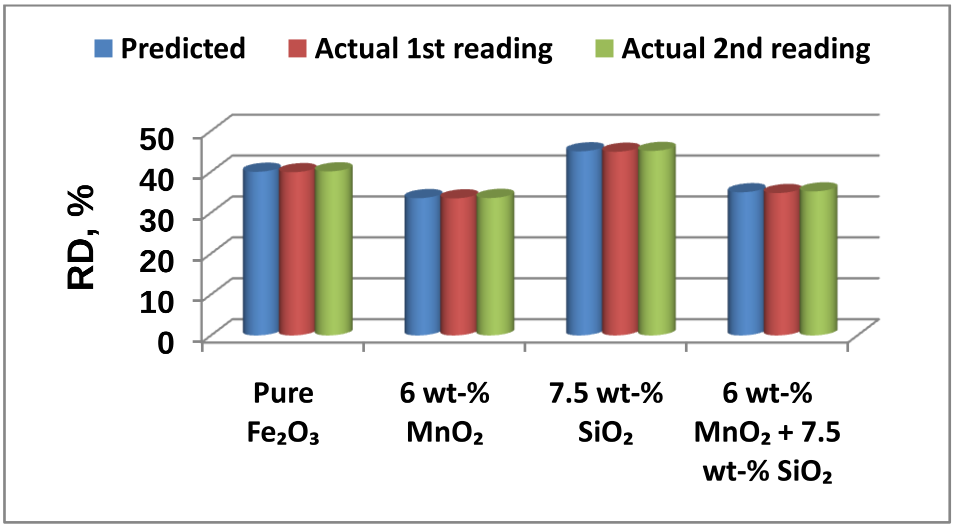


Figure 3. Actual and predicted reduction yield at 800°C

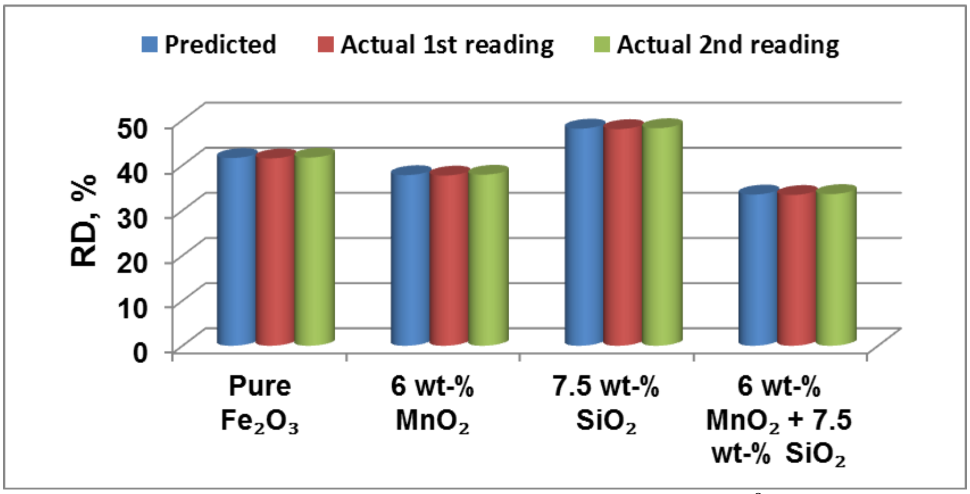


Figure 4. Actual and predicted reduction yield at 900°C

In order to examine the efficiency of the derived model, the mathematical formula which is given in Eq. 12 has been applied at different percentages of MnO_2 (0, 2, 4, and 6 wt-%), SiO_2 (0, 2.5, 5, and 7.5 wt-%) and mixture of SiO_2 (7,5 wt-%) with (0, 2, 4, and 6 wt-%) MnO_2. The results are compared to that obtained by the experimental work at 800 and 900°C as shwon in Figures 5 and 6 respectively. It can be seen that, in many cases, the calculated values of reduction yield are in a good agreement with that obtained by the experimental trails. This reveals that the factorial design is very usefull approach for staistaical analysis of the factors that affecting the reduction yield of iron oxide. The developed regerssion model is able to predict the reduction yield of iron oxide as a function of chemical composition and temperaure which makes it very effective in saving time and production cost.

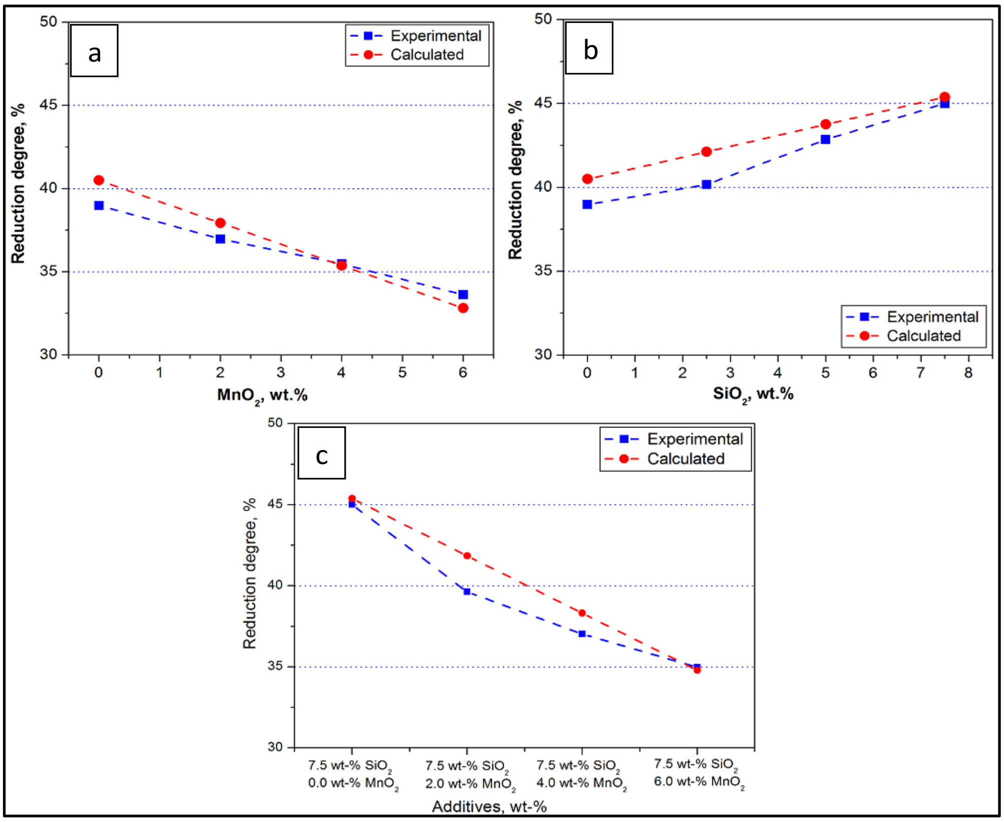


Figure 5. Comparison between the experimental and calculated reduction yield at 800°C under the influence of (a) MnO_2 (b) SiO_2 (c) $MnO_2 + SiO_2$

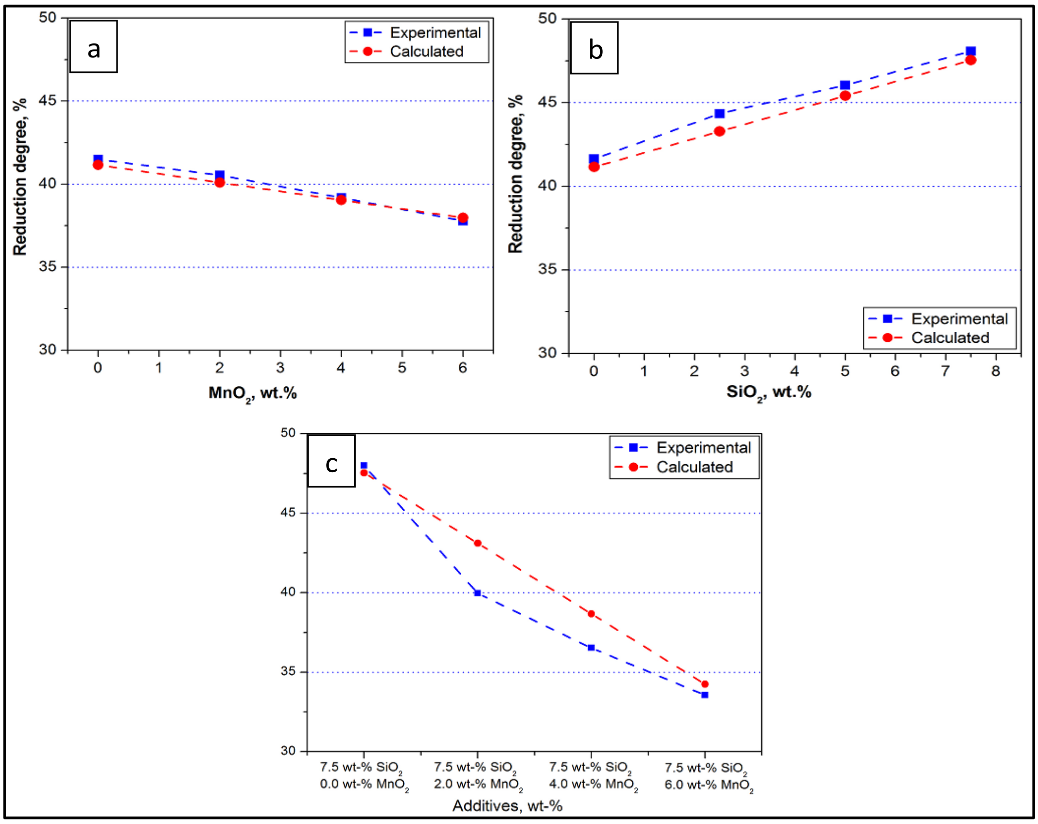


Figure 6. Comparison between the experimental and calculated reduction yield at 900°C under the influence of (a) MnO_2 (b) SiO_2 (c) $MnO_2 + SiO_2$

IV. Conlcusions

A 2^3 (three-parameters, two-levels) factorial experimental design has been implemented to analyse the reduction data of iron oxide compacts mixed with 6.0 wt.% MnO_2, 7.5 wt.% SiO_2 and 6.0% MnO_2+7.5% MnO_2.The reduction process of pure Fe_2O_3 and mixed oxides (Fe_2O_3-MnO_2-SiO_2) was carried out by CO at 800-900°C and the reduction degree was considered at 20 minutes. The main finding can be summarized in the following points:

(1) A regression model is developed based on an experimental design approach to calculate the reduction yield: $Reduction\ yield = 35.2144 + 0.0066T - 0.9483[SiO_2\%] - 7.2798[MnO_2\%] + 1.215[MnO_2\%][SiO_2\%] + 0.0075T[MnO_2\%] + 0.002T[SiO_2\%] - 0.0016T[MnO_2\%][SiO_2\%]$

(2) The SiO_2 content in the oxide mixtures showed the highest positive effect on the reduction yield while MnO_2 exhibited the highest negative effect on the reduction of iron oxide.

(3) The interaction combination of the three parameters (MnO_2, SiO_2, and temperature) was demonstrated relatively high negative impact on the reduction yield of iron oxide.

(4) The calculated values of reduction yield using the developed regression model were found in a good agreement with that obtained by experimental trails. This confirms that the factorial design can be successfully implemented to evaluate the effect of different parameters on the production yield in the metallurgical processes.

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