Saeed Ghali, Elsayed A. Mousa

**Analysis of the reduction yield of synthetic iron oxide sinter reduced by H₂ at 900-1100°C using factorial design approach**

The factorial design approach can be used to precisely estimate the effect of different parameters on the reduction process of iron oxide. In the current study, a $2^4$ factorial design was used to significantly calculate the magnitude impact of manganese oxide and silica on the reduction yield of iron oxide which was reduced with $H_2$ at 900-1100°C. A regression model was built on the experimental reduction results of pure iron oxide and iron oxide doped with either MnO$_2$ (mass content of 6%) and/or SiO$_2$ (mass content of 7.5%) at 900°C and 1100°C. The developed mathematical model was used to predict the reduction yield as a function of four parameters including MnO$_2$ (mass content of 0-6%), SiO$_2$ (mass content of 0-7.5%), reduction time (1.0-10 min) and temperature (900-1100°C). In addition, the effect of the interaction combination of different parameters (MnO$_2$, SiO$_2$, time, and temperature) on the reduction yield was estimated. The results showed that the reduction time has the highest positive effect on the reduction yield of iron oxide sinter followed by the applied temperature and then SiO$_2$ addition. On the other hand, MnO$_2$ exhibited the highest negative effect on the reduction yield of iron oxide followed by the combination effect of SiO$_2$ with time and temperature. The interaction combination effect of MnO$_2$ with temperature or MnO$_2$-SiO$_2$ with time and temperature on the reduction yield was very small. The regression model was applied to theoretically estimate the reduction yield of pure and doped iron oxide at 900-1100°C. The obtained values of the derived model are in a good agreement with the experimental results under different conditions.

1. **Introduction**

The depletion of high-grade iron ore reserves allows the world to face the iron and steel workers to utilize the intermediate- and low-grade iron ore in order to meet the growing demand on the international iron ore market [1]. The Egyptian iron ores are often contaminated with various impurities which affect negatively the production yield and energy consumption in the blast furnace [2,3]. The influence of manganese oxide and silica on the reduction behavior of iron oxide compacts with either CO or $H_2$ was intensively studied [3-6]. A catastrophic swelling (up to 405%) was accompanied the reduction of MnO$_2$-doped iron oxide compacts with CO due to the formation of metallic iron whiskers and plates while the presence of SiO$_2$ hindered such phenomenon through increasing the compact strength [3]. In addition, both of SiO$_2$ and MnO$_2$ exhibited unsteady influence on the reduction rate and mechanism of iron oxide due to the formation of hard reducible phases such as manganese ferrite (Mn$_2$FeO$_4$), iron manganese oxide (FeMnO$_4$) and a ferrite manganooan (Fe$_{3}$Mn$_{2}$O$_{9}$). The reduction behavior of high manganese iron ore (mass content of 9.9% MnO$_2$) by $H_2$ at 800-1000°C was investigated [7]. The reduction rate was increased with temperature and decreased as the time proceeded. The investigation carried out on the effect of barite (BaSiO$_4$) on the mineralogical composition of iron ore sinter indicated that the optimum BaO content in the ore blend is 2.5% mass content which is able to form eutectic and needle structures of barium ferrite solid solution [8]. The effect of Al$_2$O$_3$ on the reduction of iron ore sinter was studied [9]. The rate of reduction was increased with Al$_2$O$_3$ content up to 2.5% mass content which was attributed to the formation of high reducible silico-ferrite of calcium and aluminium phase in the applied sinter. The effect of CaO/SiO$_2$ on the reduction rate of wüstite sinter reducibility using low and high potential reducing gas is examined [10]. The highest reduction rate was exhibited by basic sinter (CaO/SiO$_2$ = 2.0) which was attributed to the formation of high reducible calcium ferrite while acidic sinter (CaO/SiO$_2$ = 0.5) was exhibited the lowest reducibility due to the formation of fayalite and ferroharzburgite. The effect of SiO$_2$ and/or Al$_2$O$_3$ on the reduction of wüstite was studied [11]. The Al$_2$O$_3$ was found to decrease the reduction rate in the all range of applied temperature; 670-930°C. On the other hand the simultaneous dissolution of SiO$_2$ and Al$_2$O$_3$ in FeO was accelerated the reduction process of FeO.

The previous survey summarized some studies which were experimentally carried out to estimate the effect of some impurities on the reduction rate of iron oxides however the effective magnitude of these impurities individually or collectively on the reduction yield is still not clear. It was demonstrated that the factorial design approach has several advantages in the prediction of process yield, process performance and estimation of key parameters that controlling the overall process [12-14]. In the current study, a $2^4$ factorial design is used to precisely estimate the individual and collective impacts of MnO$_2$, SiO$_2$, temperature and time on the reduction yield of iron oxide. The factorial design is built up on synthetic iron oxide sinter doped with MnO$_2$ (mass content of 6%) and SiO$_2$ (mass content of 7.5%) which was reduced with $H_2$ gas at 900°C and 1100°C at 1.0 and 10 min [4]. The derived model is verified with different percentage of MnO$_2$ (mass content of 2, 4 and 6%) and / or SiO$_2$ (mass content of 2.5, 5.0 and 7.5%) at 900°C, 1000°C and 1100°C in the range of 1.0-10 min. The doping amounts of either MnO$_2$ or SiO$_2$ were selected to simulate the average content of these impurities in the Egyptian iron ores.

2. **Experimental Procedure**

A $2^4$ factorial design is derived based on the experimental data of pure FeO$_2$, MnO$_2$ (mass content of 6%)-doped FeO$_2$, SiO$_2$ (mass content of 7.5%)-doped FeO$_2$ and [MnO$_2$ (mass content of 6%)+SiO$_2$(mass content of 7.5%)]-doped FeO$_2$
compacts that were reduced with H₂ gas at 900°C and 1100°C [4]. Chemically high grades (mass content of around 99.5%) Fe₂O₃, MnO, and SiO₂ powders (< 50 μm) were used to prepare a synthetic sinter in order to eliminate the influence of the other impurities that are usually associated with iron ores. The powders were mixed well in a ball mill for 8.0 hours to guarantee the full homogeneity of the mixtures. Pure Fe₂O₃ and Fe₃O₄ doped with either MnO₃ and/or SiO₂ moistened with naphtha (mass content of around 6%) were pressed in a cylindrical mould at 10 kN. The dry compacts (9.0 mm diameter, 14 mm height) were gradually heated in a muffle furnace (10 K/min) up to 1200°C and kept at this temperature for 6.0 hours. After firing, the compacts were gradually cooled down to room temperature in the furnace to avoid the thermal shock and kept dry in a desiccator. Pure and doped compacts were isothermally reduced with pure H₂ gas (1.0 L/min) at 900-1100°C. The reduction process was took place in vertical tube furnace and the oxygen weight loss from the reduction process was continuously recorded as a function of time. The reduction system was described elsewhere [10]. The reduction yield of pure Fe₂O₃ and doped iron oxide compacts after 1.0 and 10 min at 900°C and 1100°C is given in Table 1. Each trail was repeated twice under the same conditions in order to confirm the results reproducibility. A 2⁴ factorial design is used to estimate the individual and combination effect of MnO₂, SiO₂ additions, time and temperature on reduction yield. Mathematical analysis clarifies the effect of MnO₂, SiO₂ time and temperatures and their interactions on the reduction yield were deduced. The formulated regression model is applied on iron oxide compacts that doped with MnO₂ (mass content of 2, 4 and 6%), SiO₂ (mass content of 2.5, 5 and 7.5%) and MnO₂ (mass content of 2 and 4%) with SiO₂ (mass content of 7.5%) to precisely estimate the reduction yield.

3. Results and Discussion
3.1. Definition of the controlling parameters
The controlling parameters which are considered in the current applied 2⁴ factorial design are including the effect of MnO₂, SiO₂, reduction time and applied temperature. By convention, the effect of each factor was donated by a Latin letter. Thus “A” refers to the effect of MnO₂; “B” refers to the effect of SiO₂; “C” refers to the effect of time; “D” refers to the effect of applied temperature; “AB” refers to the interaction effect of MnO₂ with SiO₂; “AC” and “BC” refer to the interaction effect of MnO₂ and SiO₂ with reduction time respectively; “AD” and “BD” refer to the interaction effect of MnO₂ and SiO₂ with temperature respectively; “CD” refers to the interaction effect of time with temperature; “ABC” refers to the interaction effect of MnO₂, SiO₂ with time; “ABD” refers to the interaction effect of MnO₂, SiO₂ with temperature; “ACD” refers to the interaction effect of MnO₂, time with temperature; “BCD” refers to the interaction effect of SiO₂, time with temperature; “ABCD” refers to the interaction effect of all the parameters including MnO₂, SiO₂, time with temperature. The low and high levels of A, B, C and D are denoted by “−” and “+” respectively.

<table>
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<tr>
<th>Trial No.</th>
<th>MnO₂ (mass content of %)</th>
<th>SiO₂ (mass content of %)</th>
<th>Fe₂O₃ (mass content of %)</th>
<th>Time, min</th>
<th>Temperature, °C</th>
<th>Reduction yield of iron oxide, (mass content of %)</th>
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Table 1: Conditions of experiments and reduction yield of iron oxide

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3.2 Development of mathematical formulations

Mathematical formulations are used to estimate the effect of different parameters on the reduction yield. The effect of A at low levels of B, C and D is \((a-1)/n\), the effect of A at high levels of B, C and D is \([abc-bcd]/n\), the effect of A at low level of B and high levels of C and D is \([ac-cd]/n\), the effect of A at high level of C and low levels of B and D is \([ab-d]/n\), the effect of A at high level of C and low levels of B and D is \([ac-c]/n\), the effect of A at low level of D and high levels of B and C is \([abc-c]/n\), the effect of A at high level of B and low levels of C and D is \([ab-c]/n\), and the effect of A at high level of D and low levels of B and C is \([ad-d]/n\). The main effect of A is the average quantities of its effect at low and high levels of B and C. In similar way, the average main effect of one, two, three or four factors and their interactions can be calculated (dividing its contrast by 16 i.e. number of experiments) according to factorial design of 2^4. The effect of all parameters on the reduction yield and its interaction are given in Eqs. 1-15 (see appendix 1).

3.3 Application and validation of regression model

The plus and minus signs which can be developed from the contrasts of the effective factors are given in Table 2. The high level is referred by (+) sign and low level is referred by (-) sign.

<table>
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<th>Source of Variance</th>
<th>Average effect</th>
<th>Sum of Square (SS)</th>
<th>Degree of Freedom</th>
<th>Mean Square (MS)</th>
<th>F_0 (magnitude effect)</th>
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Table 3: Analysis of variances

Two replicates is SS= (Contrast)^2/16n. The total sum of squares (SST) has abcd-1 degrees of freedom and the error sum of squares (SSE) has abcd(n-1) degrees of freedom. Table 3 summarizes the main effects of variables, sum of squares and the mean square.

Based on this data, the magnitude and direction of the factors can be examined to determine which variable is relatively more effective compared to the others. The reaction time (C) exhibited the highest positive effect on the reduction yield followed by the applied temperature (D) followed by the interaction effect of time with temperature (CD) and finally the effect of SiO_2 (B). On the other hand, the highest negative effect on the reduction yield was revealed by MnO_2 (A) followed by the interaction effect of SiO_2-time-temperature (BCD), MnO_2-time (AC) and then SiO_2-temperature (BD). The remaining interaction parameters have a slightly negative effect on the reduction yield of iron oxide sinter. Based on the previous values, the parameters with relatively high and positive magnitude will enhance the reduction process of iron oxide at the applied time interval while the parameters with negative magnitude will retard the reduction process and consequently affect negatively the reduction yield.

The contrast coefficients which are used in the calculations are summarized in Table 2. Algebraic signs represent the contrast constants for the 2^4 factorial design.
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<th>b</th>
<th>ab</th>
<th>c</th>
<th>ac</th>
<th>bc</th>
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Table 4: Contrast coefficients of effects

The results of the experiment can be expressed in terms of a regression model given in Eq. 16, where \( x_1, x_2, x_3, \) and \( x_4 \) are coded variables that represent the MnO\(_2\)% and SiO\(_2\)%.

\[
\text{Reduction yield} = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_4 x_4 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{14} x_1 x_4 + \\
\beta_{23} x_2 x_3 + \beta_{24} x_2 x_4 + \beta_{34} x_3 x_4 + \beta_{123} x_1 x_2 x_3 + \beta_{124} x_1 x_2 x_4 + \beta_{134} x_1 x_3 x_4 + \beta_{234} x_2 x_3 x_4 + \epsilon \tag{16}
\]

The relation between the natural variable and the coded variable is given as follow:

The coded variable is equal to \([(\text{natural variable} - 1/2(\text{variable at high level} + \text{variable at low level})) / 1/2(\text{variable at high level} - \text{variable at low level})] \) for each variable. Consequently, the reduction yield can be predicted as a function of MnO\(_2\) (mass content in %), SiO\(_2\) (mass content in %), reduction time and applied temperature as given in Eq. 17.

\[
\text{Reduction yield} = 10.174 + 1.1080296[\text{SiO}_2\%] + t - 2.5392296[\text{SiO}_2\%] - 11.412 t + t - 0.0110223T - 0.001329457[\text{MnO}_2\%][\text{SiO}_2\%] -
0.0922222[\text{MnO}_2\%] * t - 0.169777778[\text{MnO}_2\%] +
0.0029457[\text{SiO}_2\%] + 0.01946222T - t -
0.008987654[\text{MnO}_2\%][\text{SiO}_2\%] * t -
0.0011105197[\text{SiO}_2\%] * t \tag{17}
\]

The derived mathematical model represented in Eq. 18 was applied to calculate the reduction yield of iron oxide sinter under the influence of different percentages of MnO\(_2\) (mass content of 2, 4, and 6%), SiO\(_2\) (mass content of 2.5, 5, 7.5%), reduction time range (1.0-10 min) and applied temperatures (900, 1000 and 1100\(^\circ\)C). The calculated values are compared to the results that was recorded by the experimental work as can be seen in Fig. 1 (a-i). It can be seen that the calculated values of reduction yield based on the application of regression model are close and in a good agreement to the average values of the experimtal results. Based on the previous
findings, it can be concluded that the factorial design is a very useful approach to precisely predict the reduction yield as a function of chemical composition, reduction time and applied temperature.

4. Conclusions

A 2^4 factorial design is built on the experimental data of pure Fe₃O₄, MnO₂ (mass content of 6%), SiO₂ (mass content of 7.5%) and MnO₂ (mass content of 6%) with SiO₂ (mass content of 7.5%) which were reduced with pure H₂ at temperature 900°C and 1100°C. The derived regression model was applied on synthetic iron oxide sinters including MnO₂ (mass content of 2.4 and 6%), SiO₂ (mass content of 2.5, 5 and 7.5%), MnO₂ (mass content of 2% and 6%) with SiO₂ (mass content of 7.5%). In order to estimate the efficiency of the derived mathematical regression model, the calculated values of reduction yield

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<th>Trail No.</th>
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Table 5: Experimental and calculated reduction yield of iron oxide under the influence of different parameters

(a) Pure Fe₃O₄, (b) MnO₂ (mass content of 2%)–Fe₃O₄, (c) MnO₂ (mass content of 4%)–Fe₃O₄, (d) MnO₂ (mass content of 6%)–Fe₃O₄, (e) SiO₂ (mass content of 2.5%)–Fe₃O₄, (f) SiO₂ (mass content of 5%)–Fe₃O₄, (g) SiO₂ (mass content of 7.5%)–Fe₃O₄, (h) MnO₂ (mass content of 2%)–SiO₂, (i) MnO₂ (mass content of 6%)–SiO₂

Fig. 1: Comparison between the calculated and experimental reduction yield.

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are compared to that obtained experimentally under different conditions. The reduction yield can be calculated based on the regression Eq. 19.

The formulated factorial design demonstrated very successfully the effect of individual and combined parameters on the reduction yield of iron oxide sinter. The reduction time showed the highest positive effect on the reduction yield followed by the applied temperature then the interaction effect of time with temperature and finally the effect of SiO₂ addition. On the other hand, MnO₂ exhibited the highest negative effect on the reduction yield followed by the interaction effect of SiO₂ with time and temperature. The interaction effect of MnO₂ with time or SiO₂ with temperature was resulted in a relatively small negative effect on the reduction yield while the remaining interaction parameters revealed very small negative effect on the reduction yield and it can be neglected.

The predicted reduction yield using the developed regression model is found to be in a good agreement with the experimental data under different conditions which reflected the efficiency of the factorial design in the deduction of the influence of various variables on the reduction process of iron oxides.

5. References

Author’s note

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Appendix 1

\[ A = \frac{1}{8n} \left[ (abcd + abc + abd + acd + ab + ac + ad + a) - (bcd + bc + bd + cd + b + c + d + 1) \right] \]  

(1)

\[ B = \frac{1}{8n} \left[ (abcd + abc + abd + bcd + ab + bc + bd + b) - (acd + ac + ad + cd + a + c + d + 1) \right] \]  

(2)

\[ C = \frac{1}{8n} \left[ (abcd + abc + cbd + acd + cb + ac + cd + c) - (abd + ab + bd + ad + b + a + c + d + 1) \right] \]  

(3)

\[ D = \frac{1}{8n} \left[ (abcd + bcd + abd + acd + bd + cd + ad + d) - (abc + bc + ab + ac + b + c + a + 1) \right] \]  

(4)

\[ AB = \frac{1}{8n} \left[ (abcd + acd + bcd + cd + ab + a + b + 1) - (abc + ac + bc + c + abd + ad + bd + d) \right] \]  

(5)

\[ AC = \frac{1}{8n} \left[ (abcd + abd + bcd + bd + ac + a + c + 1) - (abc + ab + bc + b + acd + ad + cd + d) \right] \]  

(6)

\[ AD = \frac{1}{8n} \left[ (abcd + abc + bcd + bc + ad + a + d + 1) - (acd + ac + cd + c + abd + ab + bd + b) \right] \]  

(7)

\[ BC = \frac{1}{8n} \left[ (abcd + abd + acd + ad + bc + b + c + 1) - (abc + ab + ac + a + bcd + bd + cd + d) \right] \]  

(8)

\[ BD = \frac{1}{8n} \left[ (abcd + abc + acd + ac + bd + b + d + 1) - (bcd + bc + cd + c + abd + ab + ad + a) \right] \]  

(9)

\[ CD = \frac{1}{8n} \left[ (abcd + abc + abd + ab + cd + c + d + 1) - (acd + ac + ad + a + bcd + bc + bd + b) \right] \]  

(10)

\[ ABC = \frac{1}{8n} \left[ (abcd + abc + ad + cd + bd + a + b + c) - (abd + acd + bcd + ab + ac + bc + d + 1) \right] \]  

(11)

\[ ABD = \frac{1}{8n} \left[ (abcd + abd + ac + cd + bc + a + b + d) - (abc + acd + bcd + ab + ad + bd + c + 1) \right] \]  

(12)

\[ ACD = \frac{1}{8n} \left[ (abcd + acd + ab + bd + bc + a + c + d) - (abc + abd + bcd + ac + ad + cd + b + 1) \right] \]  

(13)

\[ BCD = \frac{1}{8n} \left[ (abcd + bcd + ab + ad + ac + b + c + d) - (abc + abd + acd + bc + bd + cd + a + 1) \right] \]  

(14)

\[ ABCD = \frac{1}{8n} \left[ (abcd + ab + ac + ad + bc + bd + cd + 1) - (abc + abd + bcd + acd + a + b + c + d + 1) \right] \]  

(15)

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