

Research article

# Empirical analysis of limit of desulphurization of iron ore based on multi-factorial process variables

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## Abstract

Desulphurization of iron ore was carried out using an oxidant; powdered potassium chlorate (KClO<sub>3</sub>) of mass-input range (5-12g) and temperature range (500-800°C). The limit of desulphurization was evaluated considering the initial ore sulphur content and removed sulphur concentration. Investigation on the process analysis and mechanism of the desulphurization process revealed that oxygen gas from the decomposition of KClO<sub>3</sub> interacted with sulphur through molecular combination within the Gas Evolution Temperature Range (GETR); 375-502°C. Sulphur transformation into vapour within this temperature range was observed to facilitate easy reaction with oxygen gas to form SO<sub>2</sub>, A limit of desulphurization; 92.22% was experimentally achieved following successful reduction of the initial ore sulphur content to 0.007 % using 12g of KClO<sub>3</sub> at a treatment temperature of 800°C. A model was derived and used as a tool for empirical analysis of limit of desulphurization based on treatment temperature, mass-input of KClO<sub>3</sub>, sulphur loss-sulphur initial ratio. Deviational analysis indicates that the derived model gives best-fit process analysis with a deviation range of just 0.65-8.82%, from experimental results and invariably an operational confidence level range 91.18-99.35%. The deviation range corresponds to limit of desulphurization range: 31.4019-86.6128%, treatment temperature range: 600-800°C, KClO<sub>3</sub> mass-input range: 7-12g and range of sulphur loss-sulphur initial ratio: 0.3444-0.5556. Hence, the derived model can exclusive, be significantly and viably operational within these process conditions.

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Keywords: Desulphurization, temperature, KClO3 mass-input, sulphur loss

## 1. Introduction

High demands for defect-free engineering structures have necessitated various researches aimed at reducing the sulphur content of the iron oxide ore to a generally admissible level. Failure of steel put in service in very hot shortness or environment has been attributed to the presence of a membrane of high concentration of sulphur as iron sulphide in steel crystals [1].

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Application of lime in various desulphurization methods has been as a mix (lime/magnesium mix) or a lone addition [2]. The researchers observed that usage of "fluidized" lime and magnesium not only decreases explosion risks associated with usage of calcium carbide but reduces the cost of the treatment compared to the process of using calcium carbide.

Nwoye [3] showed significant success made on desulphurization of iron ore using varying mass-inputs of powdered potassium chlorate ( $KClO_3$ ) and treatment temperatures. In this research,  $KClO_3$  acts as an oxidant.

So many models [6-8] have been derived for analysis of the removed sulphur concentration during desulphurization of iron oxide ore using powdered potassium chlorate ( $KClO_3$ ) as oxidant. Some of these models show significant dependence of sulphur removal on the treatment temperature [5]. The validity of the model;

$$\% S = 0.1011(LogT)^{-1} \tag{1}$$

was strongly rooted in the expression  $[(T)^{\gamma\% S}] = \alpha/k_n$  where both sides of the relationship are correspondingly almost equal. Similarly Nwoye [6] derived a model for prediction of the concentration of removed sulphur based on the treatment temperature. The model is expressed as;

$$\% S = 0.0745 (LogT)^{-1} \tag{2}$$

In both cases the temperatures are in degree celsius. Furthermore, an empirical analysis of sulphur removal based on the mass-input of KClO<sub>3</sub> was carried out by Nwoye *et al* [5]. Based on the model derived for analysis, the researchers found sulphur removal to be inversely proportional to the logarithm of the mass-input of the KClO<sub>3</sub> used as oxidant. In the model expressed as;

$$%S = 0.0415(LogY)^{-1}$$
(3)

the validity of the model was rooted on the core expression  $k_n[(\gamma)^{\mu\%S}] = T/\alpha$  where both sides of the expression are correspondingly almost equal. A very similar model [8], expressed as;

$$\% S = 0.0357 (Log\alpha)^{-1}$$
<sup>(4)</sup>

shows that the concentration of removed sulphur is also inversely proportional to the logarithm of the mass-input of the KClO<sub>3</sub> used as oxidant. The model also gives a single series factor predictive analysis of sulphur removal based on the quantity of potassium chlorate added during the process.

The aim of this work is to carry out an empirical analysis of limit of desulphurization based on multi-factorial process variables used: treatment temperature, KClO<sub>3</sub> mass-input and sulphur loss-sulphur initial ratio. The essence of this work is to ascertain the possible extent of desulphurization for any set of input process parameters of treatment temperature and KClO<sub>3</sub> mass-input used during the desulphurization process.

#### 2. Materials and Method

Agbaja (Nigeria) iron ore concentrate used for this work was obtained from National Metallurgical Development Centre (NMDC) Jos. This concentrate was dried in air (under atmospheric condition) and used in the as-received condition with particle size; 150 µm. A weighed quantity of the dried iron ore concentrate was mixed with different proportions of powdered KClO3 (obtained from Fisher Scientific Company Fair Lawn, New Jerry, USA) as weighed with a triple beam balance at NMDC laboratory. Iron crucibles were filled with the sample mixtures of 5g of KClO<sub>3</sub> and 50g of ore concentrate. These samples in the crucibles were then heated to a temperature of  $500^{\circ}$ C in a Gallen kamp Hot pot electric furnace at NMDC Laboratory for 5 minutes and thereafter were emptied on white steel pans for observation. The experiment was repeated using varied combination of mass-input of KClO<sub>3</sub>, i.e. (5, 7, 9, 10, 12g) and treatment temperature i.e. (500, 600, 700, and 800°C), while the mass-input of the ore was kept constant. Weighed quantities of the sample mixtures for each experiment set were taken (after being heated) for chemical analysis (to determine percentage sulphur removal) using wet analysis method. The average of the sulphur removed concentration determined in each experiment set was taken as the precise result. It is important to state that treatment temperature range was chosen to prevent the melting of the ore during the process.

Limit of desulphurization was calculated from the expression;

$$D_L = \left(\frac{S_o - S_F}{S_o}\right) \times 100$$
(5)

Where:  $D_L$  is the limit of desulphurization (%) (Percentage of sulphur removed),  $S_F$  is the sulphur content of the ore after treatment and  $S_0$  is the initial sulphur content of the ore (before treatment).

#### 2.1. Model Formulation

Experimental data obtained from the highlighted research work were used for the model derivation. Computational analysis of these data shown in Table 2 gave rise to Table 3 which indicate that;

$$K_{e} D_{L} -1 \approx N\alpha^{2} + N_{e} \alpha + S\gamma^{2} - S_{e} \gamma + K\beta$$
(6)

Introducing the values of K, Ke, N, Ne, S, and Se into Eq. 6

$$4053 \times 10^{-5} D_{L} - 1 = 7.9203 \times 10^{-3} \alpha^{2} + 12.61 \times 10^{-3} \alpha + 5404 \times 10^{-9} \gamma^{2} - 4.3574 \times 10^{-3} \gamma + 1351 \times 10^{-3} \beta$$
(7)

$$4053 \times 10^{-5} D_{\rm L} = 7.9203 \times 10^{-3} \alpha^2 + 12.61 \times 10^{-3} \alpha + 5404 \times 10^{-9} \gamma^2 - 4.3574 \times 10^{-3} \gamma + 1351 \times 10^{-3} \beta + 1$$
(8)

$$D_L = \frac{7.9203 \times 10^{-3} \,\alpha^2 + 12.61 \times 10^{-3} \,\alpha + 5404 \times 10^{-9} \,\gamma^2 - 4.3574 \times 10^{-3} \,\gamma + 1351 \times 10^{-3} \,\beta + 1}{4053 \times 10^{-5}} \tag{9}$$

$$D_{\rm L} = 0.1954 \,\alpha^2 + 0.3111 \alpha + 1.3333 \times 10^4 \,\gamma^2 - 0.1075 \,\gamma + 33.3333 \beta + 24.6708 \tag{10}$$

Where;  $D_L$  is the limit of desulphurization (%), ( $\gamma$ ) is the treatment temperature (°C),  $\beta$  is the Sulphur loss-Sulphur initial ratio ( $S_{Loss}/S_o$ ), N is 7.9203x10<sup>-3</sup>; Equalizing constant (determined using C-NIKBRAN [3],  $\alpha$  is the mass-input of KClO<sub>3</sub> (g),  $N_e$  is 12.61x10<sup>-3</sup>; equalizing constant (determined using C-NIKBRAN [3], S is 5404x10<sup>-9</sup>; equalizing constant (determined using C-NIKBRAN [3],  $S_e$  is 4.3574x10<sup>-3</sup>; equalizing constant (determined using C-NIKBRAN [3], K is 1351x10<sup>-3</sup>; equalizing constant (determined using C-NIKBRAN [3],  $K_e$  is 4.3574x10<sup>-3</sup>; equalizing constant (determined using C-NIKBRAN [3],  $S_e$  is 4.3574x10<sup>-3</sup>; equalizing constant (determined using C-NIKBRAN [3],  $K_e$  is 4.3574x10<sup>-3</sup>; equalizing constant (determined using C-NIKBRAN [3],  $K_e$  is 4.3574x10<sup>-3</sup>; equalizing constant (determined using C-NIKBRAN [3],  $K_e$  is 4.3574x10<sup>-3</sup>; equalizing constant (determined using C-NIKBRAN [3],  $K_e$  is 4.3574x10<sup>-3</sup>; equalizing constant (determined using C-NIKBRAN [3],  $K_e$  is 4.3574x10<sup>-3</sup>; equalizing constant (determined using C-NIKBRAN [3],  $K_e$  is 4.3574x10<sup>-3</sup>; equalizing constant (determined using C-NIKBRAN [3],  $K_e$  is 4.3574x10<sup>-3</sup>; equalizing constant (determined using C-NIKBRAN [3],  $K_e$  is 4.3574x10<sup>-5</sup>; equalizing constant (determined using C-NIKBRAN [3].

# 2.2. Boundary and Initial Condition

Consider iron ore (in a furnace) mixed with potassium chlorate (oxidant). The furnace atmosphere is not contaminated i.e. free of unwanted gases and dusts. Initially, atmospheric levels of oxygen are assumed just before the decomposition of KClO<sub>3</sub> (due to air in the furnace). Mass of iron oxide ore: (50g), treatment time: 300 sec., treatment temperature range: 500-800°C, ore grain size; 150  $\mu$ m, and mass of KClO<sub>3</sub>; (5-12g) were also used.

The boundary conditions are: furnace oxygen atmosphere due to decomposition of  $KClO_3$  (since the furnace was air-tight closed) at the top and bottom of the ore particles interacting with the gas phase. At the bottom of the particles, a zero gradient for the gas scalar are assumed and also for the gas phase at the top of the particles.

# 3. Results and Discussion

Table 1 shows the result of chemical analysis carried out on the beneficiated iron ore concentrate. The table shows that the percentage of sulphur present in the asbeneficiated ore is 0.09%.

## Table 1

Result of chemical analysis of iron ore used					
Element/Compound Fe S SiO <sub>2</sub> Al <sub>2</sub> C				Al <sub>2</sub> O <sub>3</sub>	
Unit (%)	56.2	0.09	15.91	5.82	

In [2], oxygen gas from the decomposition of  $KClO_3$  attacked the ore in a gas-solid reaction, thereby removing (through oxidation) the sulphur present in the ore in the form of  $SO_2$  Eqs. 11 and 12 show this.

$$2\text{KClO}_{3(s)} \rightarrow 2\text{KCl}_{(s)} + 3\text{O}_{2(g)} \tag{11}$$

$$S_{(s)} \xrightarrow{\text{heat}} S_{(g)} + O_{2(g)} \rightarrow SO_{2(g)}$$
(12)

Result of the desulphurization process in Table 2 shows that at a constant treatment temperature, the concentration of removed sulphur increases with an increase in the mass-input of KClO<sub>3</sub>. This translates into increase in  $S_{Loss}/S_0$  values and invariably increases in the limit of desulphurization with increase in the mass-input of KClO<sub>3</sub>, even though the treatment temperature is constant.

# 3.1. Model Validation

The validity of the model is strongly rooted in Eq. 7 (core model equation) where both sides of the equation are approximately equal. Table 3 also agrees with Eq. 7 following the values of 4053 x 10<sup>-5</sup> D<sub>L</sub> – 1 and 7.9203 x 10<sup>-3</sup>  $\alpha^2$  + 12.61 x 10<sup>-3</sup>  $\alpha$  + 5404 x 10<sup>-9</sup>  $\gamma^2$  – 4.3574 x 10<sup>-3</sup>  $\gamma$  + 1351 x 10<sup>-3</sup>  $\beta$  evaluated from the experimental results in Table 2.

## Table 2

Variation of limit of desulphurization with treatment temperature, mass-input of KClO<sub>3</sub> and  $S_{Loss}/S_0$ 

Temperature	Mass of KClO <sub>3</sub>	SLoss	So	SLoss /So	DL
(°C)	(g)	(%)	(%)		(%)
500	5	0.01	0.09	0.11	11.11
600	7	0.03	0.09	0.34	34.44
700	9	0.05	0.09	0.51	51.11
700	10	0.05	0.09	0.56	55.56
800	12	0.08	0.09	0.92	92.22

Furthermore, the derived model was validated by comparing the limits of desulphurization predicted by the model and that obtained from the experiment. This was done using various evaluative techniques such as computational, statistical, graphical and deviational analysis.

#### Table 3

Variation of  $4053x10^{-5}$  D<sub>L</sub>-1 with  $7.9203x10^{-3}$   $\alpha^2$  +  $12.61x10^{-3}$   $\alpha$  +  $5404x10^{-9}$   $\gamma^2$  -  $4.3574x10^{-3}$   $\gamma$  +  $1351x10^{-3}$ B

4053x10 <sup>-5</sup> D <sub>L</sub> -1	$\begin{array}{l} 7.9203 x 10^{-3}  \alpha^2 + 12.61 x 10^{-3}  \alpha + 5404 x 10^{-9}  \gamma^2 \\ -  4.3574 x 10^{-3}  \gamma + 1351 x 10^{-3}  \beta \end{array}$
- 0.5497	- 0.42
0.3959	0.27
1.0715	1.04
1.2518	1.27
2.7377	2.51

## **3.2. Computational Analysis**

Computational analysis of the experimental and model limit of desulphurization was carried out to ascertain the degree of validity of the derived model. This was done by comparing limits of desulphurization per unit rise in treatment temperature and per unit mass-input of KClO<sub>3</sub> evaluated from predicted results with those from actual experimental results.

Limit of desulphurization per unit mass-input of KClO3  $D_{L^{\mathsf{M}}}$  (%/g) was calculated from the equation;

$$D_L M = \frac{D_L}{M} \tag{13}$$

Therefore, a plot of the limit of desulphurization against mass-input of  $KClO_3$  as in Fig. 1 using experimental results in Table 2 gives a slope, S at points (5, 11.11) and (10, 55.56) following their substitution into the mathematical expression;

$$D_L M = \frac{\Delta D_L}{\Delta M} \tag{14}$$

Eq. 14 is detailed as:

$$D_L M = \frac{D_{L2} - D_{L1}}{M_2 - M_1} \tag{15}$$

where;  $\Delta D_L$  is the variation in limits of desulphurization between  $D_{L2}$  and  $D_{L1}$  at two mass-input values of KClO<sub>3</sub> M<sub>2</sub> and M<sub>1</sub>, respectively. Considering the points (5, 11.11) and (10, 55.56) for (M<sub>1</sub>, D<sub>L1</sub>) and (M<sub>2</sub>, D<sub>L2</sub>) respectively, and substituting them into Eq. 15 gives the slope as 8.89 %/g which is the limit of desulphurization per mass-input of KClO<sub>3</sub> during the actual desulphurization process. A plot of the limit of desulphurization against mass-input of KClO<sub>3</sub> (as in Fig. 2) using derived model-predicted results gives a slope: 8.3053 %/g on substituting the points (5, 14.3971) and (10, 55.9235) for (M<sub>1</sub>, D<sub>L1</sub>) and (M<sub>2</sub>, D<sub>L2</sub>) respectively into Eq. 15. This is the model-predicted limit of desulphurization per mass-input of KClO<sub>3</sub>.



Fig. 1 Coefficient of determination between limit of desulphurization and mass-input of KClO<sub>3</sub> as obtained from the experiment



Fig. 2 Coefficient of determination between limit of desulphurization and mass-input of KClO<sub>3</sub> as obtained from derived model

Similarly, a plot of the limit of desulphurization against mass-input of KClO<sub>3</sub> (as in Fig. 3) using regression model-predicted results gives a slope: 8.89 %/g on substituting the points (5, 11) and (10, 55.56) for ( $M_1$ ,  $D_{L1}$ ) and ( $M_2$ ,  $D_{L2}$ ) respectively into Eq. 15. This is the regression model-predicted limit of desulphurization per mass-input of KClO<sub>3</sub>.



Fig. 3 Coefficient of determination between limit of desulphurization and mass-input of KClO<sub>3</sub> as obtained from regression model

Limit of desulphurization per unit rise in treatment temperature  $D_{L^{T}}$  (%/°C) was calculated from the equation;

$$D_L T = \frac{D_L}{T} \tag{16}$$

Therefore, a plot of the limit of desulphurization against treatment temperature as in Fig. 4 using experimental results in Table 2 gives a slope, S at points (500, 11.11) and (700, 55.56) following their substitution into the mathematical expression;

$$D_L T = \frac{\Delta D_L}{\Delta T} \tag{17}$$

Eq. 17 is detailed as:

$$D_L T = \frac{D_{L2} - D_{L2}}{T_2 - T_1} \tag{18}$$

where;  $\Delta D_L$  is the changing in limit of desulphurization  $D_{L2}$  and  $D_{L1}$  at two treatment temperature values  $T_2$  and  $T_1$ . Considering the points (500, 11.11) and (700, 55.56) for ( $T_1$ ,  $D_{L1}$ ) and ( $T_2$ ,  $D_{L2}$ ) respectively, and substituting them into Eq. 18 gives the slope as 0.2223 %/°C which is the limit of desulphurization per unit rise in treatment temperature during the actual desulphurization process. Furthermore, a plot of the limit of desulphurization against treatment temperature (as in Fig. 5) using derived modelpredicted results gives a slope: 0.2076 %/°C on substituting the points (500, 14.3971) and (700, 55.9235) for ( $T_1$ ,  $D_{L1}$ ) and ( $T_2$ ,  $D_{L2}$ ) respectively into Eq. 18. This is the modelpredicted limit of desulphurization per unit rise in treatment temperature. Also a plot of the limit of desulphurization against treatment temperature (as in Fig. 6) using regression model-predicted results gives a slope: 0.2223 %/°C on substituting the points (500, 11.11) and (700, 55.56) for  $(T_1, D_{L1})$  and  $(T_2, D_{L2})$  respectively into Eq. 18. This is the regression model-predicted limit of desulphurization per unit rise in treatment temperature.



Fig. 4 Coefficient of determination between limit of desulphurization and treatment temperature as obtained from the experiment



Fig. 5 Coefficient of determination between limit of desulphurization and treatment temperature as obtained from derived model



Fig. 6 Coefficient of determination between limit of desulphurization and treatment temperature as obtained from regression model

Limit of desulphurization per unit  $S_{Loss}/S_0 DL^{SS}$  was calculated from the equation;

$$D_L SS = D_L / T \tag{19}$$

Therefore, a plot of the limit of desulphurization against  $S_{Loss}/S_0$  as in Fig. 7 using experimental results in Table 2 gives a slope, S at points (0.1111, 11.11) and (0.5556, 55.56) following their substitution into the mathematical expression;

$$D_L ss = \frac{\Delta D_L}{\Delta S_{Loos} / S_O}$$
(20)

Eq. 20 is detailed as:

$$D_L ss = \frac{D_{L2} - D_{L1}}{(S_{LOSS}/S_O) - S_{LOSS}/S_O}$$
(21)

Where;  $\Delta D_L$  is changing in limit of desulphurization  $D_{L2}$  and  $D_{L1}$  at two sulphur losssulphur initial values ( $S_{Loss}/S_0$ )<sub>2</sub>, ( $S_{Loss}/S_0$ )<sub>1</sub>. Considering the points (0.1111 11.11) and (0.5556, 55.56) for (( $S_{Loss}/S_0$ )<sub>1</sub>,  $D_{L1}$ ) and (( $S_{Loss}/S_0$ )<sub>2</sub>,  $D_{L2}$ ) respectively, and substituting them into Eq. 21 gives the slope as 100% which is the limit of desulphurization per unit  $S_{Loss}/S_0$  during the actual desulphurization process.



Fig. 7 Coefficient of determination between limit of desulphurization and sulphur losssulphur initial ratio as obtained from the experiment

Similarly, a plot of the limit of desulphurization against  $S_{Loss}/S_0$  (as in Fig. 8) using derived model-predicted results gives a slope: 93.4227% on substituting the points (0.1111, 14.3971) and (0.5556, 55.9235) for (( $S_{Loss}/S_0$ )<sub>1</sub>,  $D_{L1}$ ) and (( $S_{Loss}/S_0$ )<sub>2</sub>,  $D_{L2}$ ) respectively into Eq. 21. This is the model-predicted limit of desulphurization per unit  $S_{Loss}/S_0$ .



Fig. 8 Coefficient of determination between limit of desulphurization and sulphur losssulphur initial ratio as obtained from the derived model



Fig. 9 Coefficient of determination between limit of desulphurization and sulphur losssulphur initial ratio as obtained from regression model

Furthermore, a plot of the limit of desulphurization against  $S_{Loss}/S_0$  (as in Fig. 9) using regression model-predicted results gives a slope: 100% on substituting the points (0.1111 11.11) and (0.5556, 55.56) for ( $(S_{Loss}/S_0)_1$ ,  $D_{L1}$ ) and ( $(S_{Loss}/S_0)_2$ ,  $D_{L2}$ ) respectively into Eq. 21. This is the regression model-predicted limit of desulphurization per unit  $S_{Loss}/S_0$ .

A comparison of these three sets of values for limits of desulphurization (per unit massinput of KClO<sub>3</sub>, per unit treatment temperature, and per unit ( $S_{Loss}/S_0$ ) also shows proximate agreement and a high degree of validity of the derived model.

## 3.3. Statistical Analysis

## Standard Error

The standard error (STEYX) in predicting the limit of desulphurization (using results from the derived model, regression model and experiment) for each value of the treatment temperature and mass-input of  $KClO_3$  is indicated in Table 4. The standard error was evaluated using Microsoft Excel version 2003.

#### Table 4

Comparison of the standard errors (%) evaluated from D-Model, R-Model predicted, and ExD results based on treatment temperature and mass-input of KClO<sub>3</sub>.

Analysis	Based on treatment temperature		Base	Based on mass-input of KClO <sub>3</sub>		
	ExD	D-Model	<b>R-Model</b>	ExD	D-Model	<b>R-Model</b>
STEYX	6.9305	5.9295	$2.9959 \times 10^{-5}$	6.5489	4.7067	$1.8501\times10^{\text{-5}}$

#### Correlation

The correlations between limit of desulphurization and mass-input of KClO<sub>3</sub>, limit of desulphurization and treatment temperature as well as the limit of desulphurization and  $S_{Loss}/S_0$  as obtained from the derived model, regression model and experiment considering the coefficient of determination  $R^2$  from Figs. 1-6 was calculated using Microsoft Excel version 2003.

$$R = \sqrt{R^2}$$
(22)

The evaluations correlations are shown in Tables 5 and 6. These evaluated results indicate that the derived model predictions are significantly reliable and hence valid considering its proximate agreement with results from regression model and actual experiment.

## Table 5

Comparison of the correlation evaluated from D-Model, R-Model predicted, and ExD results based on treatment temperature and mass-input of KClO<sub>3</sub>

Analysis	Based on treatment temperature			Based or	n mass-input	cof KClO3
	ExD	D-Model	<b>R-Model</b>	ExD	D-Model	<b>R-Model</b>
CORREL	0.9886	0.9961	0.9886	0.9932	0.9969	0.9932

## Table 6

Comparison of the correlation evaluated from D-Model, R-Model predicted, and ExD results based on sulphur loss - sulphur initial ratio

Analysis	Based on treatment temperature		Based on SLoss/So
	ExD	D-Model	R-Model
CORREL	1.0000	0.9972	1.0000

## 3.4. Graphical Analysis

Comparative graphical analysis of Figs. 10-12 shows very close alignment of the curves from model-predicted limit of desulphurization (MoD) and that of the experiment (ExD). The degree of alignment of these curves is indicative of the proximate agreement between both experimental and model-predicted limit of desulphurization.

## 3.5. Comparison of derived model with standard model

The validity of the derived model was further verified through application of the regression model (Least Square Method, ReG) in predicting the trend of the experimental results. Comparative analysis of Figs. 13-15 shows very close alignment of curves and significantly similar trend of data point's distribution for experimental (ExD), derived

model-predicted (MoD), and regression model predicted (ReG) results of limits of desulphurization.



**Fig. 10** Comparison of the limits of desulphurization (relative to mass-input of KClO<sub>3</sub>) as obtained from experiment and derived model



Fig. 11 Comparison of the limits of desulphurization (relative to treatment temperature) as obtained from experiment and derived model



Fig. 12 Comparison of the limits of desulphurization (relative to sulphur loss-sulphur initial ratio) as obtained from experiment and derived model



**Fig. 13** Comparison of the limits of desulphurization (relative to mass-input of KClO<sub>3</sub>) as obtained from derived model, regression model and experiment



Fig. 14 Comparison of the limits of desulphurization (relative to treatment temperature) as obtained from derived model, regression model and experiment



Fig. 15 Comparison of the limits of desulphurization (relative to sulphur loss-sulphur initial ratio) as obtained from derived model, regression model and experiment

# 3.6. Deviational Analysis

Analysis of limit of desulphurization from the experiment and derived model revealed deviations on the part of the model-predicted values relative to the values obtained from the experiment. This is attributed to the fact that the surface properties of the iron ore and the physiochemical interactions between the ore and the oxidant which were found to have played vital roles during the process were not considered during the model formulation. This necessitated the introduction of correction factor to bring the model-predicted limit of desulphurization to those of the corresponding experimental values.

Deviation (*Dn*) of model-predicted limit of desulphurization from that of the experiment is given by;

$$Dn = \left(\frac{PL - EL}{EL}\right) \times 100$$
(23)

Where; *PL* is limit of desulphurization as predicted by derived model (%) and *EL* is limit of desulphurization as obtained from the experiment (%)

Correction factor (*Cr*) is the negative of the deviation i.e.

$$Cr = -Dn \tag{24}$$

Therefore;

$$Cr = -\left(\frac{PL - EL}{EL}\right) \times 100\tag{25}$$

Introduction of the corresponding values of Cr from Eq. 25 into the derived model gives exactly the limit of desulphurization as obtained from the experiment.



Fig. 16 Variation of model-predicted limits of desulphurization with associated deviation from experimental results (relative to mass-input of KClO<sub>3</sub>)

Deviational analysis (from Figs. 16-18) indicates that the derived model gives best-fit process analysis following deviation range of just +0.65 to -8.82%, from experimental results and invariably an operational confidence level range 91.18-99.35%. The deviation range corresponds to the limit of desulphurization range: 31.4019-86.6128%, treatment

temperature range: 600-800°C, KClO<sub>3</sub> mass-input range: 7-12g and range of sulphur loss-sulphur initial ratio: 0.3444-0.5556. This implies that the derived model can exclusive, be significantly and viably operational within these process conditions.



Fig. 17 Variation of model-predicted limits of desulphurization with associated deviation from experimental results (relative to treatment temperature)



Fig. 18 Variation of model-predicted limits of desulphurization with associated deviation from experimental results (relative to sulphur loss-sulphur initial ratio)

Comparative analysis of Figs. 16-21 indicates that the orientation of the curve in Figs. 19-21 is opposite that of the deviation of model-predicted limit of desulphurization (Figs. 16-18). This is because correction factor is the negative of the deviation as shown in Eqs. 24 and 25.

Correction factor (from Figs. 19-21) indicates that the derived model gives best-fit process analysis within correction factor range of -0.65 to +8.82% added to the model-predicted limit of desulphurization. This also invariably gives an operational confidence level range 91.18-99.35%. The correction factor also corresponds to limit of desulphurization range: 31.4019-86.6128%, treatment temperature range: 600-800°C, KClO<sub>3</sub> mass-input range: 7-12g and range of sulphur loss-sulphur initial ratio: 0.3444-0.5556.



Fig. 19 Variation of model-predicted removed sulphur concentration with associated correction factor (relative to treatment temperature)



Fig. 20 Variation of model-predicted removed sulphur concentration with associated correction factor (relative to treatment temperature)

It is believed that the correction factor takes care of the effects of surface properties of the iron ore and the physiochemical interactions between the ore and the oxidant which were found to have played vital roles during the process were not considered during the model formulation.

It is important to state that the deviation of model predicted results from that of the experiment is just the magnitude of the value. The associated sign preceding the value signifies that the deviation is a deficit (negative sign) or surplus (positive sign).



Fig. 21 Variation of model-predicted removed sulphur concentration with associated correction factor (relative to treatment temperature)

# 4. Conclusions

From the results and discussion above the following conclusion can be made:

- Sulphur transformation into vapour within this temperature range facilitated easy reaction with oxygen gas to form SO<sub>2</sub>.
- A limit of desulphurization as 92.22% was experimentally achieved following successful reduction of the initial ore sulphur content to 0.007% using 12g of KClO<sub>3</sub> at a treatment temperature of 800°C.
- The derived model functioned as a tool for empirical analysis of the limit of desulphurization based on treatment temperature, mass-input of KClO<sub>3</sub> and sulphur loss-sulphur initial ratio.
- The validity of the three-factorial model was found to be rooted on the expression 4053.3748x10<sup>-5</sup> S<sub>R</sub>-1= 7.9203x10<sup>-3</sup>  $\alpha^2$  + 12.61x10<sup>-3</sup>  $\alpha$  + 5404.3646x10<sup>-5</sup>  $\gamma^2$  4.3574x10<sup>-3</sup>  $\gamma$  + 1351.1236x10<sup>-3</sup>  $\beta$  where both sides of the expression are correspondingly approximately equal.
- Statistical analysis of the derived model-predicted, regression model-predicted and experimental limits of desulphurization for each value of treatment temperature and mass-inputs of KClO<sub>3</sub> considered shows standard errors of 5.9295, 2.9959x10<sup>-5</sup> & 6.9305% and 4.7067, 1.8501x10<sup>-5</sup> & 6.5489% respectively.

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