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Gaseous-state desulphurization of Agbaja iron ore concentrate

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Abstract

Desulphurization of Agbaja Iron ore concentrate using solid potassium trioxochlorate (v) ($KCI0_3$) as oxidant has been carried out. The concentrate was treated at a temperature range 500 – 800^0C. The results of the investigation reveal that simultaneous increase in both the percentage of the oxidant added and treatment temperature used give the ideal conditions for increased desulphurization efficiency. This translates into high desulphurization efficiency when both oxidant concentration and treatment temperature are high.

Keywords: Gaseous-state; Desulphurization; Agbaja iron ore; Concentrate

1. Introduction

Agbaja is the largest known Nigerian iron ore deposit estimated at 1250 metric tonnes of ore reserve. It consists of oolitic and pisolitic structures rich in iron oxides, in a matrix that is predominantly clay. The principal constituent mineral is goethite, with minor hematite, maghemite, siderite, quartz, kaolinite pyrite and an average of 0.09%S. Uwadiele (1984). Desulphurization mainly takes place at the metal-slag interface, but the behaviour of sulphur in the bulk of metal is also of great importance. In the presence of oxygen, sulphur can form oxysulphides, which have a low melting point lntergranular sulphur-rich layers of the metal has been found to soften on heating the ingots before rolling or forging, hence the bonds between the grains are destroyed and cracks can form during plastic working. This defect is called red shortness. Edneral (1979). If manganese is present in steel, it solidifies with the formation of manganous sulphide MnS ((m,p 1620°C) and complex sulphides, this prevents red shortness of the metal in hot working but impair its mechanical properties, since brittle manganous sulphides are located at grain boundaries. Thus, it is therefore required in all cases to minimize the content of sulphur in the metal. Furthermore, there is need to control the sulphur content of the pig iron required for steel making to a value below 0.04 wt% in order to obtain a high grade steel for Engineering works. The present work is an attempt to desulphurize Agbaja iron ore concentrate (using $KClO_3$) as a pretreatment process prior to pig iron production so as to reduce the overall sulphur content of the produced steel and therefore eliminate red shortness.

2. Experimental

Agbaja iron ore concentrate used for this work was obtained from NMDC Jos. This concentrate was used in the as-received condition with particle size- 7µm. The dried concentrate as beneficiated was mixed in different proportions with solid $KClO_3$ powder obtained from Fisher scientific company, Fair Lawn, New Jersey, U.S.A) and weighed with a triple beam balance at NMDC laboratory. Iron crucibles were filled with the same mixtures of varying masses of $KClO_3$ and 50g of ore concentrate. The samples in the crucibles were then heated and held at a temperature range of 500-800°C in a Gallenkamp Hotpot electric furnace at NMDC lab. for 5 minutes and thereafter were emptied on a white steel pan for observation. It is important to state that this temperature range was chosen to prevent melting of the ore during the process. The experiment was repeated three times in each case and the average values token.. A weighed quantity of the pyrometallurgically treated ore concentrate was taken in each case for chemical analysis (to determine percentage sulphur) using wet chemical analysis method. The percentage sulphur loss following the pyrometallurgical treatment of the as received concentrate is given as

$$S_L = \%\, S_{AR} - \%S_R \qquad (1)$$

while the desulphurization efficiency of the treatment process is obtained by

$$E_D = 100(\%S_L/S_{AR}) \qquad (2)$$

where, % S_L= percentage sulphur loss resulting from the treatment of the ore, % S_{AR} = percentage sulphur in the as-received ore concentrate, %S_R = percentage sulphur remaining in the concentrate after pyrometallurgical treatment, E_D = desulphurization efficiency of the treatment process %.

3. Results and discussion

The results of this study as shown in Tables 2-15 has revealed profoundly, the significant effects of $KCI0_3$ addition (as oxidant) and temperature on the desulphurization of Agbaja iron ore concentrate. It is believed that the initial gas evolution was as a result of the decomposition of $KCIO_3$ i.e.

$$2KClO_{3\,(s)} \longrightarrow 2KCl_{(s)} + 3O_3 \qquad (3)$$

the oxygen so liberated is believed to have combined with sulphur in the iron ore to produce $S0_2$ in the equation

$$S_{(s)} \xrightarrow{\text{Heat}} S_{(g)} + O_{2(g)} \xrightarrow{375\text{-}5020C} SO_{2(g)} \qquad (4)$$

It is pertinent to note that sulphur boil and turn to vapour at a temperature of between 400-450°C, this corresponds to the Gas Evolution temperature Range (GETR). In this work, the GETR was observed to be between 375°C and 502°C following preliminary experiments carried out. It is therefore believed that it is within and above this temperature that sulphur vapour can combine with oxygen to liberate SO_2 as in Eq. 2. Tables 2-5 indicate that increase in the quantity of oxidant added increases the percentage of sulphur loss the initial S content. This indicates that oxygen is indispensably required for desulphurization to take place. This also agrees with the role of oxygen during desulphurization reaction in the steel making blast furnace, where very high activity of CaO in the slag is needed, from where oxygen is produced. Tables 6-8 also indicate that increase in the treatment temperature resulted in increase in the percentage sulphur loss.

Considering the varying quantities of $KCIO_3$ used, the GETR for sulphur in this work and the large temperature difference between GETR for sulphur in this work and the maximum treatment temperature used, it is suggested that simultaneously high oxidant concentration and high treatment temperature are the most ideal desulphurization condition. This is confirmed in Tables 5 and 8 where the maximum quantity of oxidant (15g) used at maximum treatment temperature (800°C) gave the percentage final sulphur in the concentrate to be in traces. In other words almost sulphur free since the percentage of sulphur present is too minute to be quantified. Tables 9-15 indicate that the desulphurization efficiency increase with simultaneous increase in quantity of oxidant added and treatment temperature used. Based on the foregoing, it is the view of the researcher that high oxidant ($KCIO_3$) concentration and high treatment temperature are require for high desulphurization efficiency in pyrometallurgical treatment of Agbaja iron ore concentrate. This agrees with the blast furnace conventional desulphurization conditions which includes high CaO activity (from where 0_2 is given out) and high metal temperature.

Conclusion

Following the results of this work, it is therefore concluded that pyrometallurgical desulphurization of Agbaja iron ore concentrate essentially requires simultaneously high oxidant concentration and high treatment temperature. This invariably ensures high desulphurization efficiency. This is so when both the main oxidizing agent (O_2) and sulphur are in the gaseous state.

Acknowledgment

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Table 1

Result of chemical analysis of the as-received agbaja iron ore concentrate

Element/compound	Fe	S	SiO_2	Al_2O_3
Unit (%)	56.2	0.09	15.91	5.82

Table 2

Effect of oxidant ($KClO_3$) on the sulphur content of the pyrometallurgically treated agbaja iron ore concentrate

$KClO_3$(g)	Remnant % sulphur	% sulphur Loss	Treatment Temperature (^{0}C)
5	0.080	0.010	500
7	0.068	0.022	500
9	0.062	0.028	500
10	0.060	0.030	500
12	0.038	0.052	500
15	0.020	0.070	500

Table 3
Effect of oxidant ($KClO_3$) on the sulphur content of the pyrometallurgically treated agbaja iron ore concentrate

$KClO_3$(g)	Remnant % sulphur	% Sulphur loss	Treatment temperature (^{0}C)
5	0.070	0.020	600
7	0.059	0.031	600
9	0.054	0.036	600
10	0.050	0.040	600
12	0.031	0.059	600
15	0.010	0.080	600

Table 4
Effect of oxidant ($KClO_3$) on the sulphur content of the pyrometallurgically treated agbaja iron ore concentrate

$KClO_3$(g)	Remnant % sulphur	% Sulphur loss	Treatment temperature (^{0}C)
5	0.060	0.030	700
7	0.050	0.040	700
9	0.044	0.046	700
10	0.040	0.050	700
12	0.026	0.064	700
15	0.010	0.080	700

Table 5
Effect of oxidant ($KClO_3$) on the sulphur content of the pyrometallurgically treated agbaja iron ore concentrate

$KClO_3$(g)	Remnant % sulphur	% Sulphur loss	Treatment temperature (^{0}C)
5	0.040	0.050	800
7	0.026	0.064	800
9	0.018	0.072	800
10	0.010	0.080	800
12	0.007	0.083	800
15	****	****	800

Table 6:
Effect of treatment temperature on the sulphur content of the pyrometallurgically treated agbaja iron ore concentrate using 5g $KClO_3$

Treatment temperature (^{0}C)	Remnant % sulphur	% Sulphur loss
500	0.080	0.010
550	0.077	0.013
600	0.070	0.020
650	0.066	0.024
700	0.060	0.030
750	0.053	0.037
800	0.040	0.050

Table 7
Effect of treatment temperature on the sulphur content of the pyrometallurgically treated agbaja iron ore concentrate using 10g $KClO_3$

Treatment temperature (^{0}C)	Remnant % sulphur	% Sulphur loss
500	0.060	0.030
550	0.055	0.035
600	0.050	0.040
650	0.047	0.043
700	0.040	0.050
750	0.028	0.062
800	0.010	0.080

Table 8
Effect of treatment temperature on the sulphur content of the pyrometallurgically treated agbaja iron ore concentrate using 15g $KClO_3$

Treatment temperature (^{0}C)	Remnant % sulphur	% Sulphur loss
500	0.020	0.070
550	0.017	0.073
600	0.010	0.080
650	0.010	0.080
700	0.010	0.080
750	0.007	0.083
800	****	****

Table 9
Effect of oxidant ($KClO_3$) on the desulphurization efficiency (treatment temperature – 500^{0}C)

$KClO_3$(g)	% Sulphur loss	Desulphurization efficiency(%)
5	0.010	11.11
7	0.022	24.44
9	0.028	31.11
10	0.030	33.33
12	0.052	57.78
15	0.070	77.78

Table 10
Effect of oxidant ($KClO_3$) on the desulphurization efficiency (treatment temperature – 600^{o}C)

$KClO_3$(g)	% Sulphur loss	Desulphurization efficiency(%)
5	0.020	22.22
7	0.031	34.44
9	0.036	40.00
10	0.040	44.44
12	0.059	65.56
15	0.080	88.89

Table 11
Effect of oxidant ($KClO_3$) on the desulphurization efficiency (treatment temperature – 700^0C)

$KClO_3$(g)	% Sulphur loss	Desulphurization efficiency(%)
5	0.030	33.33
7	0.040	44.44
9	0.046	51.11
10	0.050	55.56
12	0.064	71.11
15	0.080	88.89

Table 12
Effect of oxidant ($KClO_3$) on the desulphurization efficiency (treatment temperature – 800^0c)

$KClO_3$(g)	% Sulphur loss	Desulphurization efficiency(%)
5	0.050	55.66
7	0.064	71.11
9	0.072	80.00
10	0.080	88.89
12	0.083	92.22
15	****	> 92.22

Table 13
Effect of treatment temperature on the desulphurization efficiency (mass of oxidant = 5g)

Treatment temperature (^0C)	% Sulphur loss	Desulphurization efficiency (%)
500	0.010	11.11
550	0.013	14.44
600	0.020	22.22
650	0.024	26.67
700	0.030	33.33
750	0.037	41.11
800	0.050	55.56

Table 14
Effect of treatment temperature on the desulphurization efficiency (mass of oxidant = 10g)

Treatment temperature (^0C)	% Sulphur loss	Desulphurization efficiency (%)
500	0.030	33.33
550	0.035	38.89
600	0.040	44.44
650	0.043	47.78
700	0.050	55.56
750	0.062	68.89
800	0.080	88.89

Table 15
Effect of treatment temperature on the desulphurization efficiency (mass of oxidant = 15g)

Treatment temperature (^0C)	% Sulphur loss	Desulphurization efficiency (%)
500	0.070	77.78
550	0.073	81.11
600	0.080	88.89
650	0.080	88.89
700	0.080	88.89
750	0.083	92.22
800	****	> 92.22