

Calcination Behaviour of Nsuta Pyrolusite Ore in the Presence and Absence of End-of-Life Polystyrene

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Abstract

Pyrolusite ore from Ghana Manganese Company usually has a manganese composition ranging from 40 – 50 wt%. This work sought to investigate the calcination behaviour of Nsuta pyrolusite ore in the absence and presence of polystyrene. Pulverised samples of the ore with labels A, B, C and D with an initial manganese composition of 42 wt% were placed in fireclay crucibles and calcined in the absence of polystyrene in a gas fired furnace for 30, 40, 50 and 60 min, respectively, and the maximum temperature in the fireclay crucible was measured using a digital thermocouple. The samples were then calcined in the presence of polystyrene at the same duration and firing conditions. The change in colour, maximum temperature in the crucible and percent weight loss were recorded after calcination. It was observed that samples changed from greyish black to brown and this transformation was accompanied by a weight loss of 13-14.2% and 10.8-14.8%, in the absence and presence of polystyrene, respectively. Maximum recorded temperature in the fireclay crucible was in the range 584-611 °C and 732-756 °C for calcination in the absence and presence of polystyrene, respectively. These measured temperatures were above the thermodynamic equilibrium temperature (~500 °C) required for the transformation MnO_2 - Mn_2O_3 - Mn_3O_4 . Therefore, it can be concluded that, calcination of pyrolusite ore above 500 °C would increase its manganese composition.

Keywords: Calcination, Polystyrene, Pyrolusite, Weight loss, MnO_2 , Mn_2O_3 , Mn_3O_4

1 Introduction

Ghana Manganese Company is a Manganese mining company at Nsuta-Tarkwa. The company mines two major Manganese ores which are Rhodochrosite and Pyrolusite. The pyrolusite contains a higher percentage of manganese than the rhodochrosite giving it a higher value. GMC mines and sell the manganese ore to a foreign company to extract the manganese mineral from the ore. The value of the ore depends on the amount or percentage of Manganese in the ore. This implies that the higher the manganese content, the higher the value of the ore. Results of gravimetric analysis from the mine showed that the percentage of the MnO_2 in the total ore is 66% with a 42% Manganese in the ore. There have been arguments on the need to add value to the ore before shipping to create jobs. This has resulted in the search for effective ways of upgrading the ore. For

pyrometallurgical processing of the ore, the higher manganese ores, which are typically unstable at high temperatures must be transformed into a more stable form, typically from MnO_2 to Mn_3O_4 by calcination and further to MnO by reduction using C, CO, H_2 , CH_4 or waste polymers as reductants (Dankwah and Asamoah, 2013; Dankwah and Koshy, 2013). Dankwah *et al.* (2015) investigated the microwave production of manganese from MnO_2 using end-of-life polypropylene as reductant. They observed that manganese metal with a composition of up to 92.94 wt. % could be produced using pulverised carbonaceous material generated from end-of-life PP. No reducing agent was required in the transition $MnO_2 \rightarrow Mn_2O_3 \rightarrow Mn_3O_4$. However, CH_4 , H_2 , and C (from PP) were required for the subsequent transition $Mn_3O_4 \rightarrow MnO \rightarrow Mn$. For the electrolytic production of manganese metal from manganese ores $MnSO_4$ is typically used as the electrolyte, a situation that

requires that the ore must be leached using H_2SO_4 as lixiviant. Solubility of the ore in H_2SO_4 is therefore a requirement. Accordingly, the ore must be transformed from the insoluble Mn^{4+} state (as in MnO_2) to the soluble Mn^{2+} state (as in MnO). The transformation from MnO_2 to MnO involves both calcination and reduction using suitable reductants. Alternative reductants are needed owing to rising cost of electrical energy and high grade metallurgical coke, along with environmental concerns associated with production of metallurgical coke and sintered ores (Ishak and Tangstad, 2007; Abdel Halim *et al.*, 2011; Dankwah and Asamoah, 2013; Dankwah and Koshy, 2014).

The pre-reduction of manganese ores by carbon has been investigated by Abdel Halim *et al.* (2011), Ishak and Tangstad (2007), and Gao *et al.* (2012). Kor (1978) investigated the thermal decomposition (calcination) of Mn_2O_3 and reduction of Mn_3O_4 by C and CO. The carbothermic reduction of manganese ores and pure manganese oxide has been investigated by various researchers (Holta and Olsen, 1986; Eric and Burucu, 1992; Ostrovski and Webb, 1995; Akdogan and Eric, 1993; Akdogan and Eric, 1994; Akdogan and Hurman, 1995; Peretyagin and Pavlov, 2003; Kononov *et al.*, 2007; Kumar *et al.*, 2007; Kononov *et al.*, 2009a; Kononov *et al.*, 2009b). The reduction of manganese oxides with CO have been investigated by Kor *et al.* (1978); Berg and Olsen (2000) and Gao *et al.* (2012). The isothermal reduction behaviour of Fe_2O_3 - MnO composite materials with solid carbon was investigated by Abdel Halim (2007). Using the sessile drop approach, Blagus *et al.* (2013) investigated the interfacial reactions between coke/HDPE blends and high carbon ferromanganese slag. End-of-life plastics generate high amounts of the reductants CH_4 , H_2 , C, CO at high temperatures. Anacleto *et al.* (2004) observed that the presence of CH_4 with MnO allowed for greater extents and rates of reduction in comparison to reduction under hydrogen or carbon monoxide between 1000-1200°C. The impact of H_2 on the reduction of manganese oxides has been studied in other works (De Bruijn *et al.*, 1980; Kononov *et al.*, 2008)

The calcination and/or reduction of manganese oxides using end-of-life plastics has been investigated before (Dankwah and Asamoah, 2013; Dankwah and Koshy, 2014; Blagus *et al.*, 2013.).

In most of these investigations, however, reagent grade material or synthetic slag was the source of manganese oxide. Application of this concept to naturally occurring oxides of manganese would provide fundamental understanding on the behaviour of these oxides in the presence of end-of-life polymers. This is a major focus of this work.

2 Materials and Methods Used

2.1 Materials

Naturally occurring pyrolusite ore (Fig. 1) from Nsuta was used for this experimental investigation as a source of MnO_2 , whilst samples of end-of-life polystyrene (PS) collected from MELCOM supermarket, Tarkwa were used as the source of reductant. Samples of PS were broken down into crumbs and pulverised carbonaceous materials ($\sim 180 \mu\text{m}$) were prepared (Fig. 3) through a heating-quenching sequence in a manner described elsewhere (Dankwah *et al.*, 2015).



Fig. 1 Sample of Nsuta Pyrolusite Ore used for the Investigation

Samples of the pulverised pyrolusite ore (Fig. 2) were characterised by XRF, SEM/EDS and XRD at the Mark Wainwright Analytical Centre, School of Chemical Science and Engineering, UNSW-Sydney, Australia.



Fig. 2 Sample of Pulverised Nsuta Pyrolusite ore used for the Investigation



Fig. 3 Sample of Pulverised Carbonaceous Material Prepared from End-of-Life PS

2.2 Experimental Procedure

2.2.1 Calcination of Nsuta Pyrolusite Ore

Pulverised samples (80% passing $-75\ \mu\text{m}$) of about 50 g each of the Nsuta pyrolusite ore were placed in fireclay crucible and calcined for 30, 40, 50 and 60 minutes in a gas fired furnace in the absence of end-of-life polymer (PS). The temperature of the process was recorded continuously using a digital thermocouple and the maximum temperature was noted; the calcined sample was then allowed to cool and its mass measured and recorded using an electronic balance. This procedure was repeated three times after which the calcined samples were crushed, ground and sieved to 80% passing $-75\ \mu\text{m}$. The percent weight loss was recorded for each experimental run.

Next, the process was repeated in the presence of 10.0 g of pulverised samples of PS for 30, 40, 50 and 60 minutes.

3 Results and Discussion

3.1 Characterisation of Nsuta Pyrolusite Ore

3.1.1 XRF Analysis of Pulverised Pyrolusite Ore before Calcination

Results of analysis by XRF of pulverised samples of Nsuta pyrolusite ore are shown in Table 1.

Table 1 XRF of Pulverised Pyrolusite Ore before Calcination

Component	SiO ₂	Al ₂ O ₃	MgO	MnO	K ₂ O
wt %	12.58	2.21	0.24	67.08	0.55
Component	CaO	Fe ₂ O ₃	P ₂ O ₅	Other oxides*	LOI
wt %	0.47	2.62	0.19	0.40	13.67

*Other oxides: NiO, BaO, ZrO₂, Cr₂O₃, Na₂O, ZnO, CuO, TiO₂

The major component, as would be expected from an oxide ore, is MnO at 67.08 wt. % (equivalent to 51.95 wt. % Mn). This is a 'high' grade fraction obtained at the mine site from an initial 'low' grade ore of composition 45.19 wt. % (or 35.00 wt. % Mn) using spiral concentration; this upgrade was at the expense of SiO₂, whose concentration decreased from 22.25 wt. % to the current 12.58 wt. % shown in Table 1. Parameters that are of interest to manganese smelters in the Ferromanganese industry are the Mn/Fe ratio, limitations on the alumina and silica contents and the phosphorus content. High contents of alumina and silica result in excessive slag formation in the furnace; this significantly increase electricity consumption. Accordingly, the ore in its present form is suitable for the production of silicomanganese (SiMn) alloy owing to its high SiO₂ content. For use as raw material for the production of ferromanganese alloy, the SiO₂ needs further reduction in composition to below 10.00 wt. %.

The phosphorous content is a key parameter and in the selection of manganese ore, Ores with less than 0.1% P₂O₅ are at a premium. From Table 1, the composition of P₂O₅ (0.19 wt. %) is almost twice the limiting value required for the ore to be placed at a premium.

3.1.2 SEM/EDS Analysis of Pulverised Pyrolusite Ore before Calcination

The results from characterisation of the Nsuta oxide ore are shown in Fig. 4 for Region 1 in the SEM micrograph with the elemental analysis displayed in Table 2. As would be expected and indicated in Table 2, the major element is Mn whose normalized composition of 56.95 wt. % is not very different from 51.95 wt. % Mn estimated from the XRF results in Table 1.

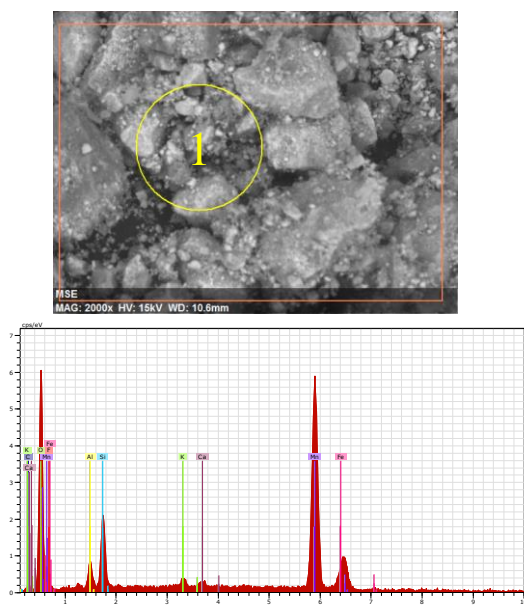


Fig. 4 SEM/EDS Analysis of Nsuta Pyrolusite Ore

Table 2 Elemental Analyses of Region 1

Element	Atomic No.	Normalized wt %	Atomic %
Manganese	25	56.95	32.34
Oxygen	8	23.25	45.33
Silicon	14	8.10	9.00
Iron	26	4.52	2.53
Aluminium	13	2.80	3.24
Carbon	6	2.02	5.25
Potassium	19	1.12	0.89
Calcium	20	0.72	0.56
Fluorine	9	0.53	0.86
TOTAL:		100	100

The Mn/Fe weight and atomic ratios are 12.6 and 12.8, respectively.

3.1.3 XRD Analysis of Pulverised Pyrolusite Ore before Calcination

The results of analysis by XRD of the Nsuta pyrolusite ore are summarised in the XRD diffractogram illustrated in Fig. 5. As shown in Fig. 5, the diffractogram of the Nsuta Pyrolusite ore consists of a mixture of Pyrolusite and Nsutite along with crystalline peaks of quartz. The presence of Nsutite, evidenced by several peaks in the XRD diffractogram in Fig. 5, is in consonance with the observation by Zwicker *et al.* (1962) who studied the nature of Nsutite ores and Ali and Amankwah (2013) who recovered Nsutite from tailings material at Ghana Manganese Company Limited at Nsuta. Zwicker *et al.* (1962) observed that Nsutite was a widespread, hydrated,

manganese oxide mineral of the composition: $Mn_{1-x}^{4+}Mn_x^{2+}O_{2-2x}(OH)_{2x}$; where $x = 0.06-0.07$ for analysed material from Ghana, Greece, and Mexico; and $x = 0.16$ for a Manganoan variety from Ghana.

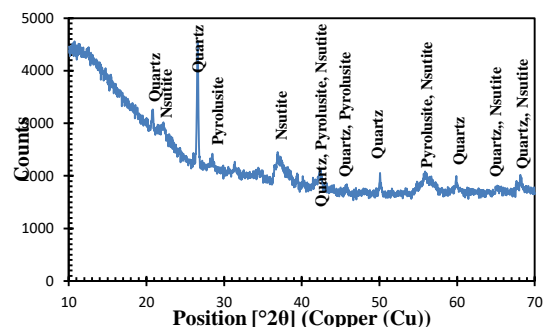
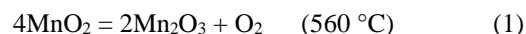


Fig. 5 XRD Diffractogram of Nsuta Pyrolusite Ore

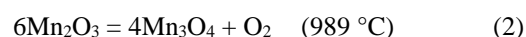
3.2 Results of Calcination of Nsuta Pyrolusite Ore

3.2.1 Calcination of Nsuta Pyrolusite Ore in the absence of PS

Table 3 shows some measured parameters for the calcination of Nsuta Pyrolusite in the absence of the end-of-life polymer. It is clear from Table 3 that the measured temperature in the crucible generally increases with time from 584 °C after 30 min to 611 °C after 60 min. A portion of the stability areas of the various oxides in the Mn-O system is shown in Fig. 6. For an oxygen partial pressure of 1 atm, MnO_2 is stable up to a temperature around 500 °C, (Knacke *et al.*, 1991), beyond which the following transition occurs in accordance with Equation(1):



Above 980 °C, Mn_2O_3 spontaneously transforms into Mn_3O_4 , as illustrated in Equation (2).



For the range of temperatures displayed in Table 3, it can be speculated provisionally that the most likely calcination product of manganese is Mn_2O_3 . The appearance of the calcined sample is shown in Fig. 7, which displays a reddish-brown mass typical of Mn_3O_4 .

Table 3 Measured Parameters for Calcination in the Absence of PS

Sample	Time (min)	Initial Mass(g)	Final Mass(g)	Temperature (°C)
A	30	50	43.5	584
B	40	50	43.2	602
C	50	50	43.0	600
D	60	50	42.9	611

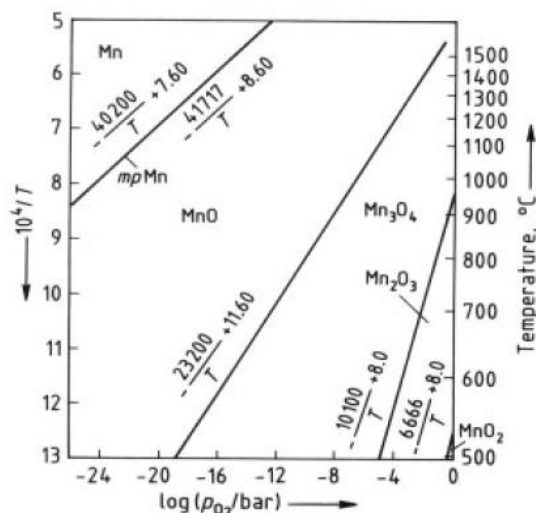


Fig. 6 Stability Areas: Manganese-Oxygen (Knacke *et al.*, 1991)



Fig. 7 Appearance of Nsuta Pyrolusite Ore after 60 min Calcination in the Absence of PS

The calcined ore was characterised by XRF. The results are shown in Table 4.

Table 4 XRF of Calcined Pyrolusite Ore in the absence of PS

Component	SiO ₂	Al ₂ O ₃	MgO	MnO	K ₂ O
wt %	15.331	4.067	-	73.631	0.514
Component	CaO	Fe ₂ O ₃	P ₂ O ₅	TiO ₂	Other oxides*
wt %	0.359	5.623	0.148	0.109	0.218

*Other oxides: NiO, SrO, BaO, ZrO₂, Cr₂O₃, Na₂O, ZnO, CuO, V₂O₅

Calcination, in the absence of the polymer, results in an improvement in the MnO content of the ore from 67.08 wt. % to 73.63 wt. %.

3.2.2 Calcination of Nsuta Pyrolusite Ore in the Presence of PS

Table 5 shows some measured parameters for the calcination of Nsuta Pyrolusite in the presence of the end-of-life polymer. It is clear from Table 5 that the measured temperature in the crucible generally increases with time from 732 °C after 30 min to 756 °C after 60 min. Temperature levels are higher (> 100 °C) in the presence of PS. The presence of PS is thus expected to enhance the overall calcination process from MnO₂ to Mn₃O₄. The product obtained after calcination in the presence of PS is shown in Fig. 8; the XRF of the pulverised sample is shown in Table 6.

Table 5 Measured Parameters for Calcination in the Presence of PS

Sample	Time (min)	Initial Mass(g)	Final Mass(g)	Temperature (°C)
A	30	50	44.6	732
B	40	50	43.4	743
C	50	50	42.8	744
D	60	50	42.9	756



Fig. 8 Appearance of Nsuta Pyrolusite Ore after 60 min Calcination in the Presence of PS

The composition of MnO in the calcined ore in the presence of PS is observed from Table 6 to be 74.14 wt. %. Although the presence of the polymer results in an increase in the temperature in the furnace, only a marginal increase (< 1 wt. %) in the content of MnO is observed. This should come as no surprise judging from Tables 3 and 5, where no appreciable difference in mass of the calcined values could be observed.

Of course, since the MnO content is still higher in the presence of the polymer, calcination of manganese ores could serve as another avenue for diverting millions of tonnes of end-of-life plastics from landfill sites to conserve landfill space.

Table 6 XRF of Calcined Pyrolusite Ore in the presence of PS

Component	SiO ₂	Al ₂ O ₃	MgO	MnO	K ₂ O
wt %	15.329	3.791	-	74.144	0.460
Component	CaO	Fe ₂ O ₃	P ₂ O ₅	TiO ₂	Other oxides*
wt %	0.353	5.500	0.094	0.125	0.204

*Other oxides: NiO, SrO, BaO, ZrO₂, Cr₂O₃, Na₂O, ZnO, CuO, V₂O₅

4 Conclusions and Recommendations

The calcination behaviour of Nsuta pyrolusite ore has been investigated in the presence and absence of end-of-life PS. The calcined products were characterised by XRF analysis and temperature measurements in the presence and absence of the polymer were compared. It is concluded that:

- i. The Nsuta pyrolusite ore is a blend of pyrolusite, Nsutite and quartz:
- ii. The ore can be calcined effectively to Mn₃O₄ and this transformation is accompanied by an increase in the content of Mn in the calcined product:
- iii. The calcined product is unaffected by the presence of PS during the calcination process:
- iv. Calcination of manganese ores is a potential route for diverting the millions of tonnes of end-of-life plastics from landfill sites.

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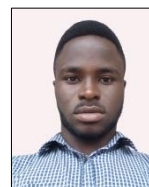
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