

Microwave Production of Manganese from Manganese (IV) Oxide using Postconsumer Polypropylene as Reductant*

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Dankwah, J. R., Adjei-Kyeremeh, F., Dwumah, B. K., Dankwah, J. B. and Koshy, P. (2015), "Microwave Production of Manganese from Manganese (IV) Oxide using Postconsumer Polypropylene as Reductant", *Ghana Mining Journal*, Vol. 15, No. 2, pp. 65 - 72.

Abstract

This work investigates the production of manganese metal from MnO_2 by microwave irradiation using postconsumer polypropylene (PP) as reductant. Reagent grade MnO_2 was first calcined to Mn_3O_4 followed by reduction with pulverised PP in a domestic microwave oven (Pioneer, Model PM-25 L, 1000 W, 2.45 GHz) in a recorded temperature range 900-1200 °C. Calcined and reduced products were characterised by XRD, XRF and SEM/EDX. The results showed that microwave irradiation is effective at calcining MnO_2 to Mn_3O_4 , evidenced by the complete disappearance of peaks of MnO_2 and appearance of peaks of Mn_3O_4 after 40 minutes. SEM/EDX analysis revealed that calcined Mn_3O_4 was first transformed to MnO and later to manganese metal. The range of temperature achieved in the microwave oven was below the equilibrium temperature for MnO reduction by solid carbon and accordingly solid carbon produced from PP cannot be wholly responsible for the production of manganese metal. It is therefore concluded that the production of manganese metal observed in this investigation was effected with CH_4 (generated from the thermal decomposition of PP) and solid amorphous carbon as the predominant reductants, with solid amorphous carbon providing the heat energy required for the reduction. The reduction of manganese oxides using postconsumer plastics as reductants is therefore a potential route for diverting plastics from landfill sites as well as decrease the amount of expensive metallurgical coke currently used in the ferromanganese process.

Keywords: Microwave irradiation, Polypropylene, Calcination, Reduction, Manganese

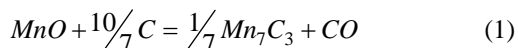
1 Introduction

Only 18.1 % of PP was recycled in Australia in the period July 2011- June 2012 (Anon., 2012). The rest was landfilled or dumped illegally. Novel and inexpensive ways for recycling plastics are currently being explored by various researchers across the globe (Matsuda *et al.*, 2008; Ueki *et al.*, 2008; Murakami *et al.*, 2009; Murakami and Kasai, 2011; Dankwah *et al.*, 2011; Dankwah *et al.*, 2012; Dankwah *et al.*, 2013; Dankwah and Koshy, 2014a). The ferromanganese industry is currently faced with the challenge of 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). A greater portion of coke and electrical energy consumption occurs in the upper part of the ferromanganese furnace (pre-reduction zone) where the highly endothermic Boudouard reaction is active (Abdel Halim *et al.*, 2011). Previous investigations (Dankwah *et al.*, 2011; Dankwah *et al.*, 2012; Dankwah *et al.*, 2013; Dankwah and Koshy, 2014a and Dankwah *et al.*, 2015) have demonstrated that waste plastics can be utilised as reductants for iron oxide reduction and also in the pre-reduction step (upper part) of the ferromanganese process (Dankwah and Asamoah, 2013; Dankwah and Koshy, 2014b) through the

release of highly reducing gaseous species (CO , H_2 and CH_4) into the reaction environment.

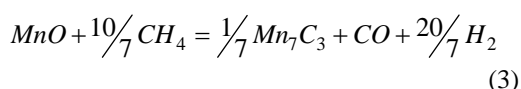
The pre-reduction of manganese ores by carbon has been investigated by Abdel Halim *et al.*, (2011), Ishak and Tangstad (2007), and Gao *et al.*, (2012). The pre-reduction of higher manganese oxides to MnO using HDPE and the effect of HDPE addition on the pre-reduction of Mn_3O_4 to MnO by metallurgical coke were investigated by Dankwah and Asamoah (2013) and Dankwah and Koshy (2014b), respectively. 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. However, little or no information exists in the literature on the exclusive utilisation of postconsumer plastics on the production of manganese metal and manganese

carbides from manganese oxides/ores. Thermodynamically, while it is impracticable to use hydrogen alone as reductant for MnO reduction at normal operating temperatures, the reduction of MnO by carbon requires a higher equilibrium temperature (1340 °C) compared to MnO reduction by methane (928 °C) (Ostrovski *et al.*, 2004; Knacke *et al.*, 1991).



The Gibbs free energy change for equation (1) is given by equation (2) (Knacke *et al.*, 1991).

$$\Delta G^\circ = 257.75 - 0.1598T \text{ J mol}^{-1} \quad (2)$$



$$\Delta G^\circ = 377.68 - 0.3144T \text{ J mol}^{-1} \quad (4)$$

For the reduction of MnO by methane gas, the equilibrium constant for equation (3) is about 8.46 at 1000°C, 113.80 at 1100 °C and 1075 at 1200°C, suggesting a high extent of reduction of MnO to manganese carbide by CH₄ in the temperature range (1000-1200°C) utilised for this investigation. Since the thermal decomposition of waste polymers at high temperatures generates large volumes of CH₄ and H₂, the reduction of compacts of MnO-polymer mixtures is possible, especially in a microwave oven where heating of materials starts from the core to the outer periphery. Anacleto *et al.*, (2004) observed that the presence of CH₄ 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 hydrogen on the reduction of manganese oxides has been studied in other works (De Bruijn *et al.*, 1980; Kononov *et al.*, 2008).

On microwave heating technology and its application to minerals processing, Walkiewicz *et al.*, (1988) investigated the microwave heating characteristics of selected minerals and compounds. They observed highest temperatures for the metal oxides NiO (1305 °C after 6.25 min), MnO₂ (1287 °C after 6.0 min), F₃O₄ (1258 °C after 2.75 min), Co₂O₃ (1290 °C after 3.0 min) and WO₃ (1270 °C after 6.0 min). They also observed that amorphous carbon sample heated from an ambient temperature of 29 °C to 1170 °C during the first 30 sec and to 1283 °C after 1 min. These conditions make the calcination of MnO₂ to Mn₃O₄ and the subsequent reduction of Mn₃O₄ to MnO and later to Mn or manganese carbides ideal. It appears the heat required to calcine MnO₂ to Mn₃O₄ can be provided by the heating process of MnO₂ in the microwave. However, the reduction of MnO to Mn

or any of the carbides will require the excellent heating capability of amorphous carbon (1283 °C) owing to the poor heating capability of MnO (113 °C).

Accordingly, this investigation is aimed at the exclusive utilisation of postconsumer PP (a producer of C, H₂ and CH₄) on the production of manganese metal and manganese carbides from manganese oxides through the crucible-reduction approach. This represents the final stage (MnO Mn/Mn₇C₃/Mn₂₆C₇ transformation) of the overall utilisation of postconsumer plastics in the ferromanganese process after a successful demonstration of postconsumer plastics utilisation in the pre-reduction step (MnO₂ Mn₂O₃/Mn₃O₄ MnO) (Dankwah and Asamoah, 2013; Dankwah and Koshy, 2014b).

2 Materials and Methods Used

2.1 Materials

Pulverised reagent grade manganese (IV) oxide (-75 μm) obtained from SD-Fine Chem. Ltd. (Mumbai) was used as the oxide. Its XRD and XRF are shown in Fig. 1 and Table 1, respectively.

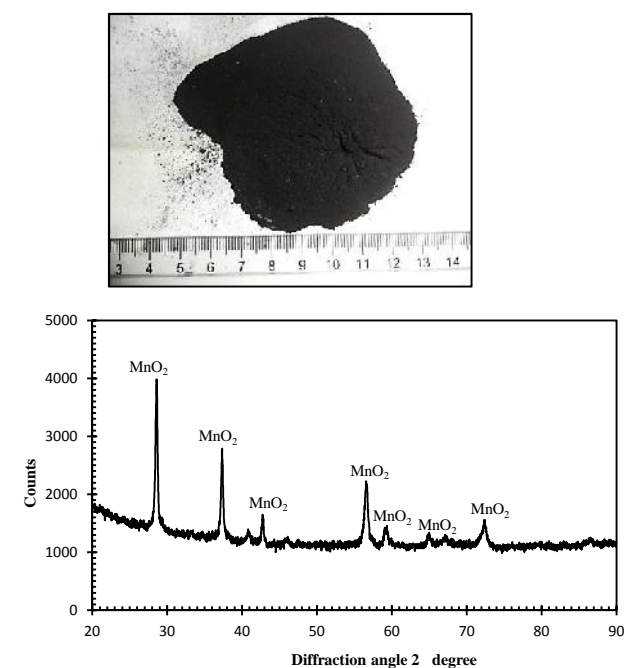


Fig. 1 Pulverised Reagent Grade MnO₂ along with its XRD

Table 1 XRF of Pulverised Reagent Grade MnO₂ used for this Investigation

Component	SiO ₂	Al ₂ O ₃	MgO	MnO ₂	Na ₂ O	K ₂ O
wt %	1.57	0.67	0.42	77.85	0.30	0.38
Component	P ₂ O ₅	CaO	Fe ₂ O ₃	Other oxides		LOI
wt %	0.26	1.25	3.64	0.99		12.93

*Other oxides: P₂O₅, BaO, TiO₂, V₂O₅, NiO, CuO, SrO, ZrO₂, HfO₂ and PbO

Postconsumer PP (obtained from waste buckets collected from Tarkwa, Ghana and pulverised to -75 μm , after a heat treatment and embrittlement process) was used exclusively as reductant (Fig. 2). Its XRD patterns are shown in Fig. 3 and they are identical to the diffraction patterns of virgin polypropylene.



Fig. 2 Pulverised Postconsumer PP obtained from Waste Plastic Buckets

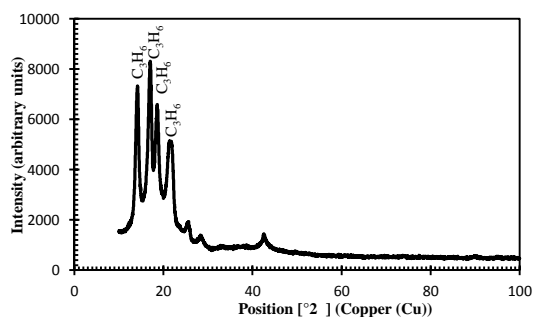


Fig. 3 XRD Patterns of Pulverised Carbonaceous Material Generated from PP

2.2 Experimental Procedure

2.2.1 Calcination of MnO_2 to Mn_3O_4

About 50 g of pulverised (-75 μm) reagent grade MnO_2 was placed in a fireclay crucible and calcined for 60 minutes in a domestic microwave oven (Pioneer, Model PM-25 L, 1000 W, 2.45 GHz), Fig. 4. The calcined sample was quickly withdrawn from the microwave oven and the temperature was recorded using a K-type thermocouple; the calcined and sintered sample was then allowed to cool and its mass measured and recorded using an electronic balance.

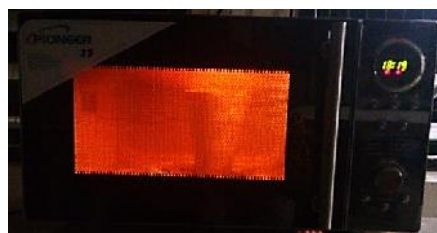


Fig. 4 Calcination Process in a Domestic Microwave Oven

This procedure was repeated three times after which the calcined and sintered samples were mixed, crushed, ground and sieved to -75 μm .

2.1.2 Reduction of Mn_3O_4 to MnO and Mn

About 20 g of the pulverised calcined Mn_3O_4 was mixed with pulverised PP at a C/O ratio of 1.50. The mixture was bottle-rolled for about 4 hours to ensure a thoroughly uniform mixture. The mixture was then emptied into a fireclay crucible followed by microwave irradiation for 40 minutes. The reduced mass was quickly withdrawn from the oven and its temperature determined using a K-type thermocouple. It was then allowed to cool sufficiently and samples were submitted for XRD and SEM/EDX analyses.

3 Results and Discussion

3.1 Calcination of MnO_2 to Mn_3O_4

The product of the calcination process (Mn_3O_4) along with its XRD is shown in Fig 5, whilst Table 2 gives the composition by XRF analysis.

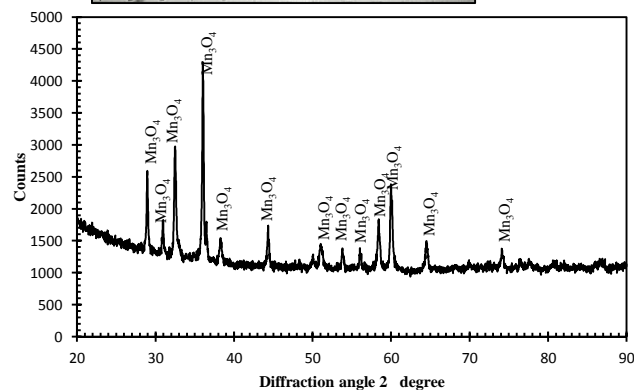


Fig. 5 Mn_3O_4 obtained after Calcination of MnO_2 along with its XRD Patterns

The measured weights before and after calcination are shown in Table 3. As indicated in Fig 5, the calcination process was complete, evidenced by the complete disappearance of peaks of MnO_2 and the subsequent appearance of sharp peaks that correspond to Mn_3O_4 . Stoichiometrically, when 50.0 g of pure MnO_2 undergoes complete calcination, 43.87 g of Mn_3O_4 is produced,

assuming no losses. From Table 3, the mass of calcined product obtained was slightly below the stoichiometric value of 43.87 g; the slight discrepancy could be attributed to loss of volatiles and sintering due to calcination, which resulted in part of the material sticking to the crucible.

Table 2 XRF of Pulverised Mn₃O₄ obtained after Calcination of MnO₂

Component	SiO ₂	Al ₂ O ₃	MgO	Mn ₃ O ₄	Na ₂ O	K ₂ O
wt %	3.95	1.35	0.53	82.14	0.46	0.48
Component	CaO	Fe ₂ O ₃	NiO	Other oxides	LOI	SUM
wt %	1.66	5.04	1.70	1.80	weight gain	99.14

*Other oxides: BaO, TiO₂, V₂O₅, P₂O₅, Cr₂O₃, CuO, SrO, ZrO₂, HfO₂ and PbO

Table 3 Weight of Manganese Oxide before and after Calcination

Sample	Weight before Calcination (g)	Weight after Calcination (g)
Stoichiometrically	50	43.87
A	50	42.28
B	50	42.83
C	50	40.58
D	50	42.49

The SEM/EDX analyses of two regions from the semi-reduced mass are reported in Figs 6 and 7. Table 4 shows the %Mn of the various oxides of manganese, along with the O/Mn atomic ratios.

Table 4 (%Mn) and O/Mn Ratios for Various Oxides of Manganese

Oxide	%Mn	O/Mn
MnO ₂	63.2	2.0
Mn ₂ O ₃	69.6	1.5
Mn ₃ O ₄	72.0	1.33
MnO	77.4	1.0

The elemental composition in regions 1 and 2 are shown in Tables 5 and 6, respectively.

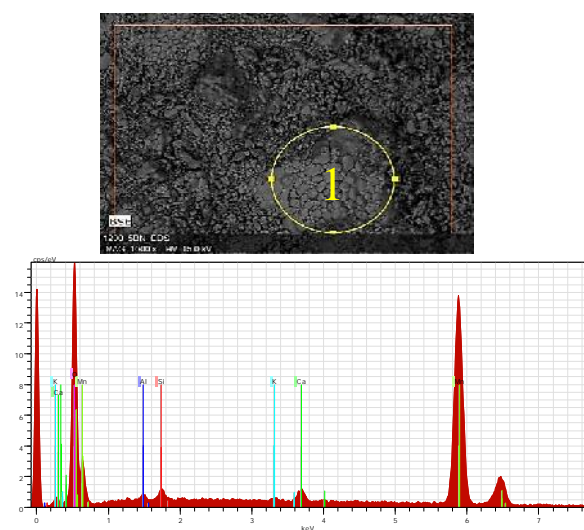


Fig. 6 SEM/EDX Analysis of Region 1 from Calcined Manganese Oxide (MnO₂ Mn₃O₄)

Table 5 Elemental Analysis of Region 1

Element (K-Series)	Weight %	Atomic %
Mn	73.11	45.04
O	25.17	53.25
Ca	0.72	0.61
Si	0.40	0.48
Al	0.25	0.31
K	0.36	0.31
SUM	100.00	100.00

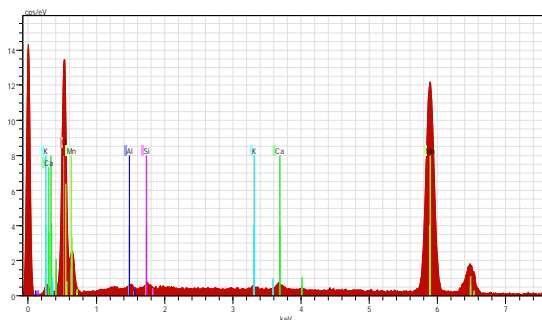
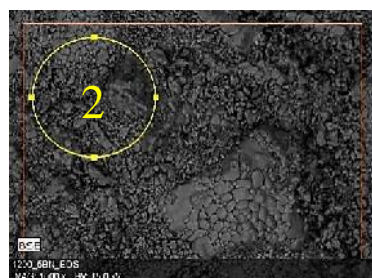


Fig. 7 SEM/EDX Analysis of Region 2 from Calcined Manganese Oxide (MnO₂ Mn₃O₄)

Table 6 Elemental Analysis of Region 2

Element (K-Series)	Weight %	Atomic %
Mn	70.71	42.62
O	26.33	54.49
Ca	1.50	1.24
Si	0.81	0.95
Al	0.36	0.44
K	0.30	0.25
SUM	100.00	100.00

The composition of Mn in the two regions were 70.71 wt % and 73.11 wt %, and an O/Mn atomic ratio greater than 1 but less than 1.5, suggesting that manganese existed predominantly as the oxide, Mn₃O₄ (which has a manganese content of 72.03 wt %) along with some MnO. The low silicon content gives an indication of the absence of a slag zone.

3.2 Reduction of Mn₃O₄ to MnO and Mn

The reduced mass obtained after reduction of Mn₃O₄ by postconsumer PP in the microwave is shown in Fig. 8.



Fig. 8 Material Produced after Reduction of Mn₃O₄ by Postconsumer PP

The morphology of the reduced mass as analysed by SEM is shown in Fig. 9, which shows that the oxide became sintered after the reduction process.

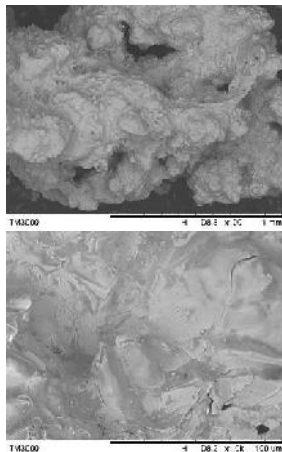


Fig. 9 SEM Micrograph of a Sample of the Reduced Mass

The SEM/EDS of the sample after 10 min of reduction is shown in Fig. 10 and the elemental analysis (mass %) of region 1 is shown in Fig. 11. Region 1 consists of the elements Mn (84.3%) and O (15.7%) only, with an O/Mn atomic ratio (0.64) well below 1. Accordingly, this region is a mixture of metallic Mn and unreduced MnO. It shows that after 10 min of reduction, calcined Mn₃O₄ is completely transformed to MnO and Mn.

After about 40 minutes of microwave irradiation the reduced mass was analysed for evidence of MnO or Mn/Mn₇C₃/Mn₂₆C₇ formation. The SEM is shown in Fig. 12 whilst Fig. 13 shows the EDX of a region. The composition of the major elements in this region is shown in Table 7.

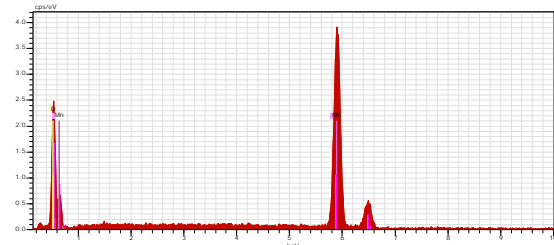
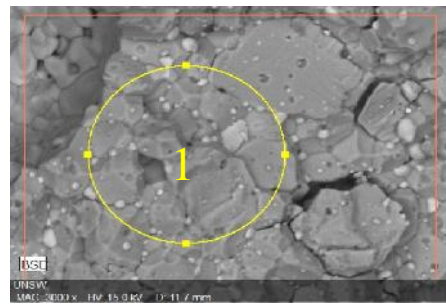


Fig. 10 SEM/EDX of the Reduced Mass after 10 mins of Reduction

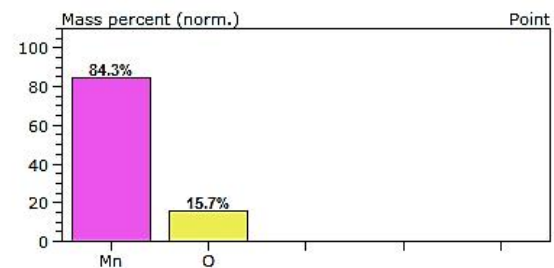


Fig. 11 Elemental Analysis (Mass Percent) of Region 1

From Fig. 13 and Table 7, it is seen that the oxide is almost essentially reduced to the metal with the manganese content increasing from about 72 wt% after calcination to about 93 wt%, while the oxygen content decreased from about 26 wt% to 4.4%. In the same region a low O/Mn ratio of 0.16 is observed along with an extremely low Si content, indicating a low slag activity. The maximum temperature recorded in the transformation step of MnO to the metal was about 1197 °C. At this temperature the thermodynamic conversion of MnO to the metal or carbide is not possible, suggesting that solid carbon alone could not have effected that transformation, since the equilibrium temperature for the carbide formation is about 1340 °C. It shows that methane (equilibrium reducing temperature 928 °C) (or possibly another hydrocarbon) could be a component of the reducing agents. Previous researchers (Nishioka *et al.*, 2007; Dankwah and Koshy, 2014b) observed that large amounts of CH₄ and H₂ are generated along with some solid C, when polypropylene is heated above 1000 °C. Accordingly, the most likely reductant responsible for the formation of manganese metal from MnO in this investigation is CH₄ or a combination of C, CO or H₂, since reduction of the oxide by C, CO or H₂ alone is not likely at the

temperatures recorded in the microwave oven. Of course, as elucidated earlier, solid amorphous carbon is still important for the reduction of MnO to Mn or manganese carbide. This is because of the low heating capability of MnO which can only get to a maximum of 113 °C. This temperature is reasonably below the equilibrium temperature for the reduction of MnO by even CH₄.

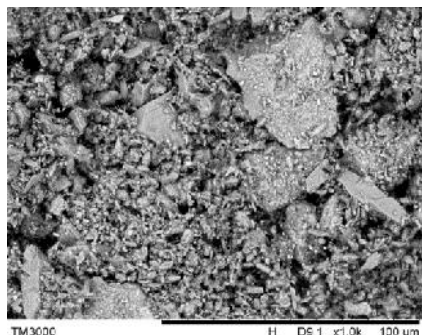


Fig. 12 SEM of the Reduced Mass after 30 mins of Reduction

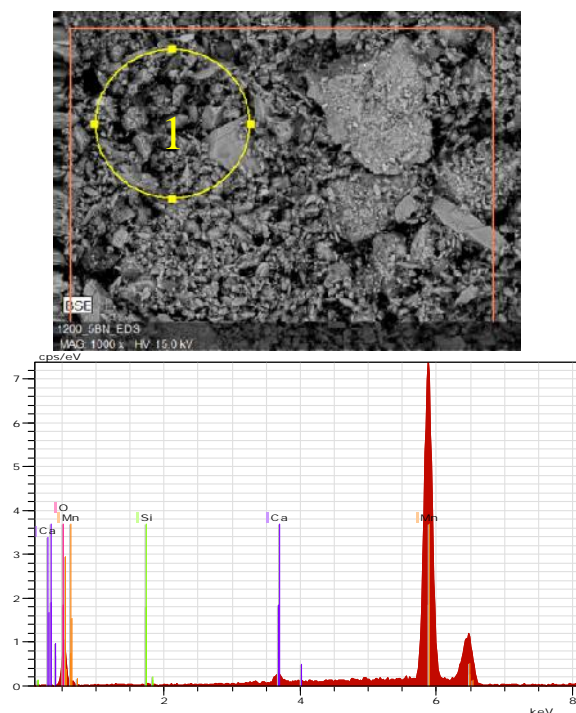


Fig. 13 SEM/EDX of Region 1

Table 7 Elemental Analysis of Region 1

Element	Series	Weight %	Atomic %
Mn	K	92.94	82.86
O	K	4.44	13.60
Ca	K	1.98	2.42
Si	K	0.64	1.12
Total		100.00	100.00

Kononov *et al.*,(2008) showed that greater rates and extents in the reduction of MnO were achievable with the presence of H₂ in comparison to reduction under the inert gases He and Ar between 1200-1300°C.

4 Conclusions

The production of manganese from MnO₂ has been investigated using carbonaceous material prepared from postconsumer PP. Major findings of the investigation are:

- (i) Manganese can be successfully produced from MnO₂ using carbonaceous materials generated from postconsumer PP.
- (ii) The reduction process followed the sequence MnO₂ Mn₃O₄ MnO Mn
- (iii) The last transformation step MnO Mn required both solid carbon and CH₄ as reductants.
- (iv) The Ferromanganese Process is another promising avenue for dealing with postconsumer plastics like PP

Acknowledgements

Part of the analyses for the investigation was conducted at the School of Materials Science and Engineering and the Analytical Centre, School of Chemical Science and Engineering, UNSW, Sydney, Australia. The authors are grateful to the various authorising bodies for the assistance received.

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