Application of Microwave Technology to the Production of Iron Nuggets from the Sheini Iron Ore using Mixed Plastics Waste as Reductants*

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Abstract

Disposal of waste plastics is currently a huge global problem, especially in the least developed countries where proven avenues for recycling such wastes are non-existent or not well documented. In this work the potential for producing high grade iron nuggets from the Sheini iron ore in the Northern Region of Ghana is investigated using carbonaceous materials generated from mixed plastics waste (MPW) as reductants. Carbonaceous materials were generated from MPW by pulverising an embrittled mixture of waste pure water sachets (PWS), waste polypropylene (PP), waste polystyrene (PS), waste low density polyethylene (LDPE) and waste polyethylene terephthalate (PET) obtained through a melting-quenching sequence. Two variants of MPW (MPW 1 and MPW 2) were used along with the individual polymers as reductants. MPW 1 was comprised of 40 wt % PWS, 30 wt % PP, 20 wt % LDPE and 10 wt % PS while MPW 2 consisted of 40 wt % PWS, 30 wt % PP, 20 wt % LDPE and 10 wt % PET. The generated carbonaceous materials were characterised by XRD, SEM/EDS and FTIR analyses. Reduction studies were then conducted on composite pellets of the ore containing MPW 1, MPW 2 and the individual plastics. Reaction products were characterised by SEM/EDS and XRD and the extent of reduction after 40 min was determined for MPW 1, MPW 2 and the individual polymers. XRD and FTIR analyses showed that individual polymers retained their chemical structure in the generated carbonaceous materials after the melting-quenching process. It was further observed that iron nuggets can be produced from the Sheini iron ore using MPW 1, MPW 2 and the individual waste polymers. The measured extent of reduction ranged from about 68.7% to about 91.24%, with the highest attained for PET and the blend containing PET.

Keywords: Reduction; Sheini Iron Ore; Waste Pure Water Sachets; Extent of Reduction.

1 Introduction

The Sheini iron ore is a low grade ore in the Northern Region of Ghana. In its current form it cannot not be exploited economically because of its location, relatively high phosphorus content and iron content, which ranges averagely from about 47 to 50 wt % Fe (Kesse and Banson, 1975).

Waste polymer reduction technology is an emerging area in Metallurgy where carbonaceous materials generated from waste polymers are used in place of or in combination with other carbonaceous materials as reductants for metal oxides reduction (Dankwah et al., 2016). Countries like Ghana boast of commercial quantities of iron ores but lack the relevant source of carbonaceous materials like metallurgical coke and natural gas conventionally used for iron oxide reduction. For such Countries, mixed plastics waste may offer a readily available alternative to high grade metallurgical coke and/or natural gas as reductant for iron making. In particular, the use of MPWs as reductants eliminates the need to sort out various plastics from municipal solid waste and, accordingly, cut down cost. Currently there is no

information on the exploitation of the Sheini iron ore for iron production.

End-of-life plastics like PWS, PP, LDPE, PS, EPS and PET are readily available in Ghana (Dankwah 2015). Currently disposal of these polymers is a huge problem and is predominantly through the landfill option. However, a significant portion is disposed of illegally resulting in choked drains. Incineration of these polymers at landfill sites results in the generation of dioxins, furans and aromatic polycyclic hydrocarbons (PAHs) (Dankwah et al., 2016). One promising route to finding a fitting solution to the plastics waste pandemonium is the utilisation of postconsumer plastics as reductants or as a source of energy in iron and steelmaking and is currently gaining the attention of various researchers (Matsuda et al., 2006; Nishioka et al., 2007; Matsuda et al., 2008; Ueki et al., 2008; Dankwah et al., 2011; Kongkarat et al., 2011; Murakami et al., 2009; Murakami and Kasai, 2011; Dankwah et al., 2012; Dankwah et al., 2013a; Dankwah and Koshy, 2014; Dhunna et al., 2014; Mansuri et al., 2014; Dankwah et al., 2015a; Dankwah et al., 2015b; Dankwah et al., 2016). However, the focus of existing research in this area

has been on the utilisation of waste polymers and or their blends with metallurgical coke, graphite, or biomass as reductants for the production of metallic iron from reagent grade iron oxides or electric arc furnace (EAF) slags containing iron oxide. For previous single plastics-biomass blends, researchers investigated the reduction of reagent grade iron oxide with HDPE-PNS (Dankwah, 2013b), HDPE-CNS (Dankwah, 2014a) and HDPE-SD (Dankwah 2014b) as reductants. Successful production of premium grade metallic iron using various blends of plastics-biomass blends as reductants was reported. A major challenge in this direction was the need to sort out individual plastics from municipal solid waste, regardless of whether the individual plastics were to be used alone or in combination with various forms of biomass or metallurgical coke. This is because the sorting out process is expensive and highly labour intensive.

Metal oxide reduction using microwave technology is an emerging phenomenon and it is aimed at addressing part of the environmental issues associated with the conventional production of metals in the Blast furnace (Dankwah *et al.*, 2016). The key factor for application of microwaves in the iron industry is its high potential for an essential reduction of carbon dioxide emission (Takayama, 2007). It relies on the ability of either the metal oxide or the carbonaceous reductant or both to absorb microwave energy and to heat rapidly to temperatures sufficient for the reduction of the metal oxide (Dankwah *et al.*, 2016).

The reduction of iron oxides using microwave energy as heating source has been previously investigated. Ishizaki et al. (2006) investigated the reduction of magnetite ore-coal composite pellets in a nitrogen gas atmosphere using the microwave approach. By means of XRD analysis they observed that the reduction of magnetite commenced at about 800°C and got to completion (with pig iron formation) at about 1350 °C. They further observed that the heating rate of pellets was independent of their mass but dependent on applied power because of self-heating. The selectivity of microwave absorption in the reduction of Fe₃O₄ with carbon black in mixed powder was investigated by Ishizaki and Nagata (2007). Standish and Huang (1991) had earlier observed that the carbothermic reduction of both magnetite concentrates and hematite fines could be and rapidly carried out with satisfactorily microwave heating. Zhong et al., (1996) investigated the reduction of a low-silica taconite concentrate by coke or coal using an industrial microwave furnace. Mourao et al., (2001) investigated the carbothermic reduction of composite pellets containing a hematite iron ore, coke or charcoal as carbonaceous material. Nagata *et al.*, (2004) showed that pig iron was obtained from magnetite ore–coal composite pellets by microwave heating. Chen *et al.*, (2003) observed the metallization rate of iron ore concentrates containing coal can be increased through microwave heating with carbothermal reduction. Using microwave irradiation, Morita *et al.*, (2002) were able to recover about 70 wt. % of iron and 25 wt. % of phosphorous from factory steel-making slag by carbothermal reduction. More data is needed in the literature on the use of mixed plastics waste as reductants in the production of metallic iron from naturally occurring iron ores in a microwave.

Accordingly, in this work, the potential for utilising MPW as reductant for the production of metallic iron from the Shieni iron ore is investigated through the microwave approach.

2 Resources and Methods Used

2.1 Materials

Mixed plastics waste consisting of PWS, LDPE, PP, PS and PET were collected from the surroundings of the University of Mines and Technology (UMaT), Tarkwa. These were shredded using a pair of scissors and rinsed with water in a plastic container to remove most of its associated debris. The cleaned MPW were thereafter air dried for two days. Pulverised carbonaceous materials were then prepared from the MPW in a manner as described in the work by Dankwah et al., 2015. Two variants of MPW (MPW 1 and MPW 2) were used along with the individual polymers as reductants. MPW 1 was comprised of 40 wt % PWS, 30 wt % PP, 20 wt % LDPE and 10 wt % PET while MPW 2 consisted of 40 wt % PWS, 30 wt % PP, 20 wt % LDPE and 10 wt % PS. Pulverised Shieni iron ore was used as the source of iron oxide.

2.2 Experimental Procedure

For pellet formation, pulverised sample of iron ore was mixed with appropriate amounts of the carbonaceous reductant with about 1 g of wheat flour as binder. Pellets were prepared into paste for moulding, by adding water and stirring with plastic spoon to homogenise the mixtures. Pellets were cured and dried at room temperature for four days to remove physically held water in the pellets. The dry weights of the pellets were taken after curing using an electronic balance.

The dried pellets (Fig. 1) were placed into a fire clay crucible and positioned at the central location of the microwave (Fig. 2) to ensure maximum concentration of the radiation emitted.



Fig. 1 Composite Pellet of Pulverised Samples of Shieni Iron Ore, PP and Flour.



Fig. 2 Reduction Process in a Domestic Microwave Oven.

After 40 minutes of firing, the crucible was taken out and quenched in air to stop any further reactions. The weights of the reduced iron pellets were recorded.

3 Results and Discussion

3.1 Carbonaceous Materials

Fig. 3 shows the XRD and FTIR of samples of pulverised carbonaceous materials obtained from pure water sachets after the heat treatment process detailed in the work of Dankwah *et al.*, 2015b. As illustrated in Fig. 3, peaks of C_2H_4 corresponding to semi crystalline HDPE are observed in the XRD (Dankwah *et al.*, 2015b).



Fig. 3 a) XRD and b) FTIR of Pulverised Carbonaceous Material Generated from PWS

From the FTIR, very prominent peaks are observable at the wavenumbers 2914 and 2849 cm⁻¹, corresponding characteristically to =C-H stretch in alkenes of polyethylene. The third prominent peak at 1463 cm⁻¹ is due to C-H bend in alkanes of polyethylene. Although the final major peak at 719 cm⁻¹ is characteristic of C-H "oop" of aromatics, the minor prominent peak at 1719 cm⁻¹ is due to C=O stretch of saturated aliphatic, which is also characteristic of polyethylene. It means that the heat treatment process does not affect the structure of the original polymer from which the sachet was formed from.

The XRD diffractogram and the FTIR of samples of pulverised carbonaceous materials generated from waste PP bucket are shown in Fig. 4a and 4b, respectively. The XRD diffractogram shown in Fig. 4a is dominated by distinct peaks of C_3H_6 , suggesting monomers of propene, which can emanate from the polymer polypropylene. From the FTIR of Fig. 4b, a weak peak at 2950 cm⁻¹ combines with very prominent peaks at wavenumbers 2916 and 2849 cm⁻¹, corresponding characteristically to =C-H stretch in alkenes of polypropylene. The next two major peaks at 1462 and 1375 cm⁻¹ correspond to C-H bend and C-H rock, respectively, of alkanes. In this case, again, the structure of the polypropylene molecule does not appear to be significantly affected by the heat treatment process, although several minor peaks are observed at 997, 992, 841, 719 and 464 cm⁻¹.



Fig. 4 a) XRD and b) FTIR of Pulverised Carbonaceous Material Generated from Waste PP Bucket

The XRD in Fig. 5a suggests that the take-awayfood container was manufactured from an amorphous polymer. The FTIR in Fig. 5b is consistent with an aromatic polymer evidenced by the peaks at 3025 cm⁻¹ (C-H stretch for Aromatics), 1600 cm⁻¹ (C=C stretch (in-ring) for Aromatics), 1492 cm⁻¹ (C-C stretch (in-ring) for Aromatics) and 753 and 695 cm⁻¹ (C-H "oop" in Aromatics). However, the peaks at 2919 cm⁻¹ (=C-H stretch for Alkanes), 1451 cm⁻¹ (C-H bond in Alkanes) suggest an aliphatic hydrocarbon.



Fig. 5 a) XRD and b) FTIR of Pulverised Carbonaceous Material Generated from Waste PS

The structure of polystyrene is shown in Fig. 6 and it demonstrates that the styrene monomer consists of the aromatic benzene ring and the aliphatic ethylene molecule, confirming the peaks observed in the FTIR.



Fig. 6 Structure of Polystyrene (PS)

Fig. 7 shows the XRD diffractogram and FTIR of pulverised carbonaceous material generated from the Voltic bottles. There are no well-defined peaks in the diffractogram of Fig. 7a, suggesting that the original polymer is amorphous. Although peaks corresponding to C-H stretch for Aromatics are missing, the peak at 1577 cm⁻¹ on one hand and those at 1505 cm⁻¹ and 1409 cm⁻¹ correspond to C=C stretch (in-ring) for Aromatics, respectively. Additionally, the multiple peaks at 872, 792, 724, 698 cm⁻¹ are all due to C-H "oop" for Aromatics. Fig. 7b also shows that PET has peaks that are similar to polyethylene molecules, at 2917, 2849 cm⁻¹ as a result of =C-H stretch for Alkanes and

1716 cm⁻¹, due to C=O stretch for saturated aliphatic. Two other peaks at 1372, 1341 cm⁻¹ correspond to C-H rock for Alkanes. It shows then that the pulverised carbonaceous material generated from PET is a copolymer of an aliphatic polymer and an aromatic monomer as illustrated in Fig. 8. This again suggests that the heat-treatment process did not have any significant effect on the structure of the polymers used for the investigation.



Fig. 7 a) XRD and b) FTIR of Pulverised Carbonaceous Material Generated from Waste PET Bottles



Fig. 8 Structure of Polyethylene Terephthalate (PET)

The FTIR in Fig. 9 shows that the structure of LDPE is similar to that of pure water sachets. Major peaks occur at 2915 and 2848 cm⁻¹ (=C-H stretch for Alkanes), 1463 and 1472 cm⁻¹ (C-H bend in Alkanes), similar to what was observed for PWS.



Fig. 9 FTIR of Pulverised Carbonaceous Material Generated from Waste LDPE

The compositions of the two blends (MPW 1 and MPW 2) which were used along with the individual polymers are shown in Table 1. The FTIRs of carbonaceous materials generated from MPW 1 and MPW 2 are shown in Fig. 10. The peaks in the FTIRs of the two blends do not appear to show any marked difference, evidenced in the compositions displayed in Table 1. The reason is that 90 wt % of the individual aliphatic polymers in the blend is similar with the only difference being the extra 10 wt % of the aromatic polymers PS and PET for MPW 1 and MPW 2, respectively.



Fig. 10 FTIR of Pulverised Carbonaceous Material Generated from a) Blend S1 and b) Blend S2

 Table 1 Blend Composition of Reductants utilised for the Investigation

Sample ID	Carbonaceous Material	Composition (%)	
	PWS	40	
MPW 1	PP	30	
	LDPE	20	
	PS	10	
	PWS	40	
MPW 2	PP	30	
	LDPE	20	
	PET	10	
S3	PWS	100	
S 4	PP	100	
S5	LDPE	100	
S 6	PS	100	
S 7	PET	100	

3.2 The Nature of the Sheini Iron Ore

The composition (XRF) of the Sheini iron ore utilised for the investigation is shown in Table 2. The major component is Fe_2O_3 with an average composition of about 64.5 wt%. This shows that

the Sheini iron ore is a low to medium grade hematite ore.

Table 2 Chemical	Composition	(XRF)	of	Sheini
Iron Ore				

Component	Composition (wt %)		
-	Undersize	Oversize	
Fe ₂ O ₃	64.666	64.483	
SiO ₂	9.901	10.204	
CaO	0.039	0.044	
Mn_3O_4	0.060	0.062	
Al_2O_3	10.157	10.092	
TiO ₂	1.595	1.604	
Na ₂ O	0.136	0.127	
K ₂ O	0.011	0.018	
MgO	0.083	0.073	
P_2O_5	0.889	0.826	
Cr_2O_3	0.053	0.031	
SO ₃	0.124	0.134	
LOI	12.322	12.319	
TOTAL	100.036	100.017	

The SiO₂ content of the ore (9.901 wt % and 10.092 wt % for undersize and oversize, respectively) is significant, unlike in the situation for reagent grade Fe₂O₃. Silica requires a much higher temperature for reduction than Fe₂O₃ does. Accordingly, the presence of large amounts of SiO₂ in the ore would have a negative impact on the reduction of Fe₂O₃.

The Sheini iron ore is similar to the Agbaja iron ore from Nigeria; pretty high average phosphorus contents of 0.858 wt % and 1.403 wt %, respectively. Although the Agbaja iron ore has a relatively high phosphorous content no traces of phosphorus were detected in the metal that was produced after reduction of the ore by carbonaceous materials generated from waste thermosetting electrical sockets (Dankwah *et al.*, 2016).

The SEM/EDS analyses of the Sheini iron ore before reduction are illustrated in Figs. 11-14 for four Regions 1, 2, 3, and 4, respectively. Regions 1, 2, 3 and 4 show iron contents of 47.30 wt %, 50.11 wt %, 47.89 wt % and 47.58 wt %, respectively. These values agree with the observation by Kesse and Banson (1975) that the ore is low grade. A major difference between the Sheini and the Akpafu-Todzi iron ores is that the Sheini ore has slightly higher iron content; another difference is that the Al₂O₃ content is higher than the SiO₂ content for the Sheini iron ore. The reverse is true for the Akpafu-Todzi iron ore.



Fig 11 SEM/EDS Analysis of the Sheini Iron Ore (Region 1)



Fig. 12 SEM/EDS Analysis of the Sheini Iron Ore (Region 2)



Fig 13 SEM/EDS Analysis of the Sheini Iron Ore (Region 3)



Fig. 14 SEM/EDS Analysis of the Shieni Iron Ore (Region 4) The ore was also characterised by XRD. The diffraction patterns are shown in Fig 15. The diffraction patterns correspond to those of Fe_2O_3 along with minor peaks of SiO_2 .



Fig 15 XRD of Sheini Iron Ore

3.3 Reduction of the Sheini Iron Ore by MPW

Fig. 16 shows the nature of metal produced from the reduction of the Sheini iron ore by carbonaceous materials generated from MPW. Several micro-spherical nuggets of metallic iron are seen distributed unevenly in a lump of oblong solid mass. This gives an indication of localised melting in areas where the spheres are located in the solid mass. The level of reduction achieved is high, judging from the fact that reduction was done in an oxygen atmosphere (no flow of inert gas).



Fig. 16 Reduced Sheini Ore

3.4 Extent of Reduction of Shieni Iron Ore by MPW and the individual Polymers

The extent of reduction of the ore by carbonaceous materials generated from various polymers was measured for seven different reductants. The results are shown in Table 3 and compared with the reduction of the Itakpe-Kogi iron ore by five of the reductants under identical experimental conditions.

Table	3	Observed	Extent	of	Reduction	for
	var	ious Experi	iments			

Reductant	Blend Composition	Extent of Reduction (%)	
	-	Sheini	Itakpe- Kogi*
S1	40PWS+30PP+20LDPE +10PS	80.86	-
S2	40PWS+30PP+20LDPE +10PET	87.10	89.99
S 3	PWS	77.85	90.52
S 4	PP	79.09	76.92
S5	LDPE	68.70	81.19
S6	PS	74.42	-
S7	PET	91.24	87.32

*Dankwah et al., 2016

As shown in Table 3, the average extent of reduction of the Shieni iron ore ranged from 68.70% to 91.24%, slightly lower than the 76.92% to 90.52% previously reported for the Itakpe Kogi iron ore by Dankwah *et al.*, 2016. The best reductant was observed to be PET, as opposed to the PWS reported for Itakpe Kogi iron ore (Dankwah *et al.*, 2016)

4 Conclusions

The reduction of the Sheini iron ore has been investigated using carbonaceous material prepared from two combinations of mixed plastics waste MP1 and MP2 generated from HDPE, LDPE, PP, PS and PET blends as reductants in a domestic microwave. Each of the carbonaceous materials was characterised by SEM, XRD and FTIR and the extent of reduction was determined for each of the reductants. Major findings of the investigation are:

- i. Mixed plastics waste are a potential source of carbonaceous materials for iron oxide reduction;
- The Sheini iron ore is a low grade hematite ore with an average grade of about 48 wt Fe₂O₃;
- iii. The Sheini iron ore can be successfully reduced using MPW as reductant;
- iv. The measured extent of reduction ranged from about 68.70.95% to about 91.24%, with the highest attained for raw PET.

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