Recycling Mixed Plastics Waste as Reductant in Ironmaking*

J. R. Dankwah, T. Amoah, J. Dankwah, and A. Y. Fosu

Abstract

One of the major obstacles to the implementation of an appropriate plastics recycling scheme is the inhomogeneity of many plastics waste. Accordingly, most of the existing recycling schemes require a feedstock that is reasonably pure and contains only items made from a single polymer type. However, in reality, waste plastics contain a mixture of plastic types, and are often contaminated with non-plastic items. This demands sorting out, which is expensive and highly labour intensive. In this work, the reduction of reagent grade iron oxide by mixed plastic waste (MPW) has been investigated through experiments conducted in a laboratory scale horizontal tube furnace. Composite pellets of reagent grade iron oxide (97 % Fe_2O_3) with MPW [consisting of 50 wt % high density polyethylene (HDPE), 30% polypropylene (PP), 10% low density polyethylene (LDPE) and 10% polyethylene terephthalate (PET)] were rapidly heated at 1520°C under high purity argon gas and the off gas was continuously analysed for CO, CO_2 and CH_4 using an online infrared gas analyser (IR). The extent of reduction after ten minutes was determined for each carbonaceous reductant and the results were compared with the extent of reduction by conventional metallurgical coke under the same experimental conditions. The results show that iron oxide can be effectively reduced to produce metallic iron using MPW as reductant. An improvement in extent of reduction was observed over metallurgical coke and the individual polymers when MPW was used as reductant. This eliminates the need to sort out individual plastics from municipal solid waste for their effective utilisation as reductants in ironmaking.

Keywords: Reduction, Metallurgical coke, Mixed plastics waste, Extent of reduction

1 Introduction

Globally, the bulk of plastics waste generation is attributed to the fastest growing plastics like High Density Polyethylene (HDPE), Low or Linear Low Density Polyethylene (L/LLDPE), Polypropylene (PP) and Polyethylene Terephthalate (PET). Apart from PET that had a relatively high recycling rate of about 53.2%, only 23%, 31 % and 18.1 % of HDPE, L/LLDPE and PP, respectively, were recycled in Australia in the period July 2011- June 2012 (Anon., 2012). The rest was land filled or dumped illegally. In Ghana, recycling figures are not well documented for waste plastics. As at 2005, an estimated 270 tons of plastic waste was generated every day in Ghana, with 85% coming from non-biodegradable plastic bags containing drinking water and ice cream at an estimated plastics recycling rate of only 2% (Stoler *et al.*, 2012). Locally, attempts have been made at raising public awareness about the plastic waste issue through recycling used sachet wrappers into consumer goods such as shopping bags, fashion accessories, and even clothing (Stoler *et al.*, 2012). However, such measures only temporarily reduce the volume of plastics waste; once these consumer goods reach the end of their useful life, they oftentimes must be discarded as there is a limit to which plastics can be recycled mechanically.

One of the major obstacles to the implementation of an appropriate plastics recycling scheme is the inhomogeneity of many plastics waste.

Accordingly, most of the existing recycling schemes require a feedstock that is reasonably pure and contains only items made from a single polymer type. In the metallurgical industry, particularly in iron and steelmaking technologies, the use of postconsumer plastics as reductants and or source of energy 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; Murakami *et al.*, 2009; Murakami and Kasai, 2011; Dankwah *et al.*, 2012; Dankwah *et al.*, 2013; Dankwah and Koshy, 2014; Dankwah *et al.*, 2015). However, most of the existing research in this area involves the use of a single plastic and/or its blends with metallurgical coke or biomass as reductants. In reality, most postconsumer wastes contain a mixture of plastic types, and are often contaminated with non-plastic items. Little or no information exists in the literature on the use of mixed plastics waste (MPW) as reductants in the metallurgical industry as reductant. The potential feasibility of utilising MPW as reductant in iron oxide reduction is thus the motivation for this research project.

2 Materials and Methods Used

2.1 Materials

The carbonaceous materials used for this investigation consisted of pure water sachets (PWS, CSIR cool brand), waste polypropylene bucket

(PP), waste shopping bags (LDPE) and waste polyethylene terephthalate water bottle (PET, VOLTIC brand) collected from the Timber market, Koforidua, Ghana. MPW was formed using 50 wt % PWS, 30 wt % PP, 10 wt % LDPE and 10 wt % PET. The chemical composition (wt %) of the carbonaceous samples and the ash analyses are given in Tables 1 and 2, respectively. Ash analysis was performed for the PWS only (Dankwah *et al.*, 2015), as the other polymers produced negligible amounts of ash. Table 1 shows that LDPE has the highest carbon content with no oxygen, whilst PET has the highest oxygen content. The chemical composition (wt %) of the reagent grade iron oxide used for the investigation was attained by XRF analysis (Oxford Instruments Lab-X3500 model) and is shown in Table 3.The oxide consists of essentially Fe_2O_3 with minor amounts of other oxides and an LOI content less than 3 wt %. It is expected that when an Fe_2O_3-polymer composite pellet is heated at temperatures well above a 1000 °C, the predominant reactions will be oxygen removal from Fe_2O_3 by C, H, CO, CH_4 or other hydrocarbons from the thermal decomposition of the polymer.

Table 1 Chemical Composition of PWS, PP, LDPE and PET

Component wt. %	C	H	S	N	O	Moisture	Ash
PWS	72.32	12.8	0.28	0.23	4.76	2.87	6.3
PP	83.8	13.9	0.092	0.14	0	0.01	0.45
LDPE	84.9	14.2	0.81	0.08	0	0	0
PET	62.5	4.2	0	0	33.3	0.18	0.04

Sources: (Dankwah *et al.*, 2015; Dankwah and Koshy, 2014; Dankwah *et al.*, 2013)

Table 2 Ash Analysis of PWS as Revealed by XRF

Component	SiO_2	Fe_2O_3	Al_2O_3	TiO_2	P_2O_5	CaO	MgO
wt.%	41.45	10.79	6.77	2.82	0.82	26.60	2.14
Component	Na_2O	K_2O	ZnO	CuO	SO_3	Mn_3O_4	SrO
wt.%	1.95	2.56	0.43	0.37	1.45	0.37	0.12

Table 3 Chemical Composition (XRF) of Reagent Grade Iron Oxide

Component	Composition (wt.%)
Fe_2O_3	96.89
SiO_2	0.445
CaO	0.0225
MnO	0.020
ZnO	0.0115
TiO_2	0.134
SO_3	0.257
LOI	2.217
TOTAL	100

2.2 Methods

Samples of PWS, PP, LDPE and PET were washed, dried and cut into smaller sizes using a pair of scissors. They were then heat treated at temperatures below 300 °C, followed by quenching to render them brittle. The embrittled polymer was then pulverised in RETSCH pulveriser model 5657 to produce fine powders (-250 µm) of carbonaceous materials as shown in Figs (1a, 2a, 3a and 4). The morphology of the pulverised carbonaceous samples was observed by Scanning Electron Microscopy (SEM), as illustrated in Figs (1b and 3b). The pulverised polymeric samples were also characterised by XRD (Figs 3, 5 and 8) using an EMPYREAN Diffractometer. The operating conditions of the EMPYREAN Diffractometer are summarised in Table 4.

Table 4 Operating Conditions ofthe XRD Machine

Operating Parameters	Values/Condition
X-Ray Diffraction Unit	EMPYREAN
Scan Axis	Gonio
Monochromator Used	NO
Generator Voltage	45 kV
Current	40 mA
Scanning Range	10.0156-80.00001
Scan Step Size	0.026261
Scan Type	Continuous
Time Per Step	27.54

The XRD patterns of PWS and PP consist of crystalline peaks of C_2H_4 and C_3H_6, respectively while that of PET is amorphous, with no well-defined peaks. The iron oxide (Fig 10) was subsequently mixed with the carbonaceous blends and compacted in a die to produce cylindrical pellets (11.6-12.0 mm thick and 14 mm diameter) (Fig11), by applying a load of 7.5 tonnes for 1 minute in a hydraulic press.
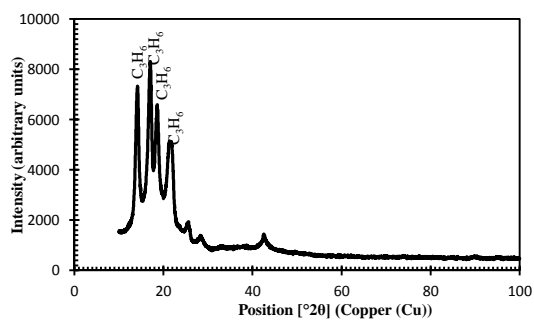

a)

b)

c)
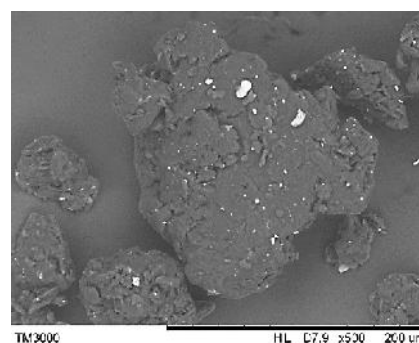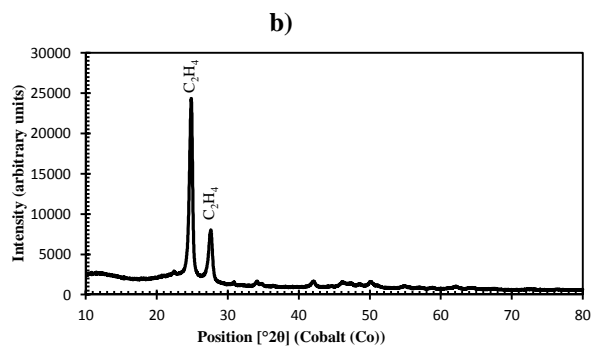
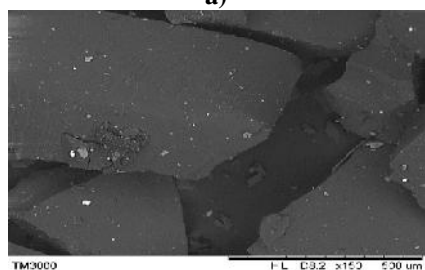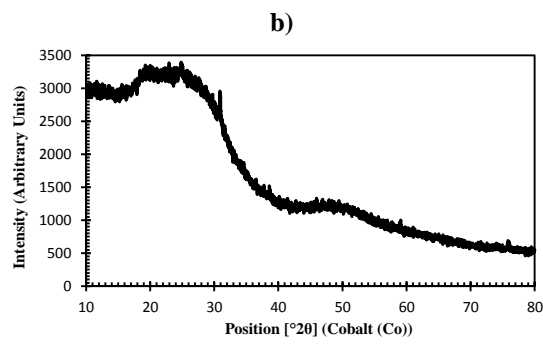**Fig. 1 a) Photograph b)SEM and c) XRD Patterns of Pulverised Carbonaceous Materials Generated from PWS**

a)

b)

Fig 2. a) Photographand b) XRD Patterns of Pulverised Carbonaceous Materials Generated from PP
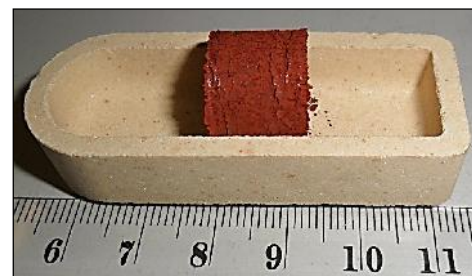

a)

b)

Fig. 3 a) Photograph b)SEM and c) XRD of Pulverised Carbonaceous Materials Generated from PET

c)

Fig. 4 Pulverised Carbonaceous Materials Generated from LDPE

Fig. 5 Reagent Grade Iron Oxide (Fe$_2$O$_3$)

Fig. 6 Fe$_2$O$_3$-MPW Composite Pellet in a LECO Crucible

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2.2.1 The Reduction Process

The reduction process involved three steps: the reactions in a custom-made horizontal resistance heated furnace (Fig7), visual observation using a CCD camera and off-gas analysis using a continuous infrared gas analyser, to monitor off-gases produced by the reduction reaction. The results (gas analyses and visual imaging) were recorded in a data-logging computer.

The sample assembly was inserted in the furnace, which was purged continuously with argon (of 99.95 % purity and flowing at 1L/min) to ensure inert conditions in the furnace. After the furnace had attained the desired hot zone temperature (1520 °C), the sample was pushed into the reaction hot zone and the reactions were monitored for 10 minutes. This time was selected since initial trials showed that the reactions ceased and thus there were no further changes in gas composition or degree of reduction beyond this time.

Fig 7 Inlet Photograph of the Horizontal Tube Furnace-IR Gas Analyser System

3 Results and Discussion

### 3.1 Nature of Metal Produced

Particles of reduced iron metal, which were clearly visible to the naked eye (Fig 8), were removed magnetically and analysed for C and S using a LECO C/S analyser. The reduced metal was highly carburised with low sulphur content. The metal particles are nearly spherical, suggesting that they crystallised out from the liquid state at an experimental temperature (1520 °C) below the melting point of metallic iron (~1537 °C). This gives an indication that the reduced metal picked enough carbon after reduction to stay in the liquid state and solidified after the tray had been withdrawn from the reaction zone to the cold zone.


Fig. 8 Nature of Metal Produced from Reduction of Iron Oxide by MPW at 1520 ºC

3.2 Gas Generation Rates (IR Analyser) During Reduction of Fe_2O_3

The contents (vol %) of CO, CO_2 and CH_4 in the off-gas were measured continuously by an infrared (IR) gas analyser. The results are shown in Fig9. Gas emissions commenced after 40s, 80 s, 60 s, 60 s and 40 s of pushing the tray into the reaction zone for PWS, LDPE, PP, PET and MPW, respectively. CO appears to be the dominant gas for all the individual polymers and MPW. The highest CO peak was observed for MPW whilst the lowest was observed for PWS. Two CO peaks (one major and one minor) are observed for the low oxygen polymers PWS, LDPE and PP, whilst only one is observed for PET (33.3 wt % O) and MPW (with 10 wt % PET). Fig. 9 also suggests that not all the CH_4 evolved is utilised for reduction in a single step. To ensure effective utilisation of CH_4, for example in a rotary hearth furnace, pellets could be arranged in layers, so that CH_4 from the bottom layer of pellets could act as reductant for the layers above.
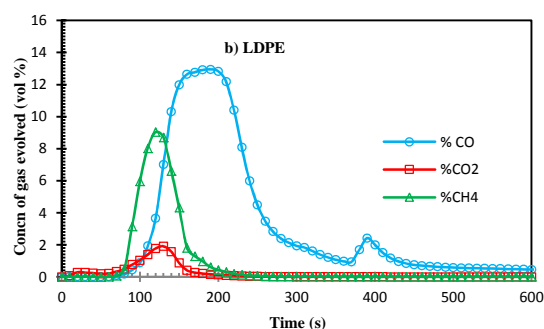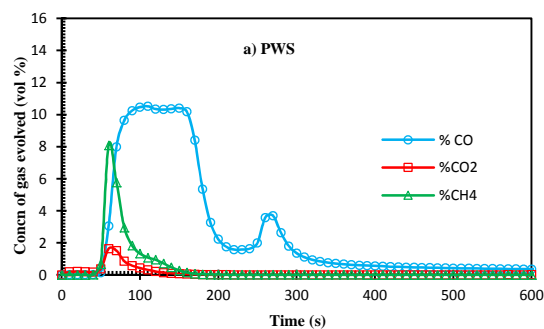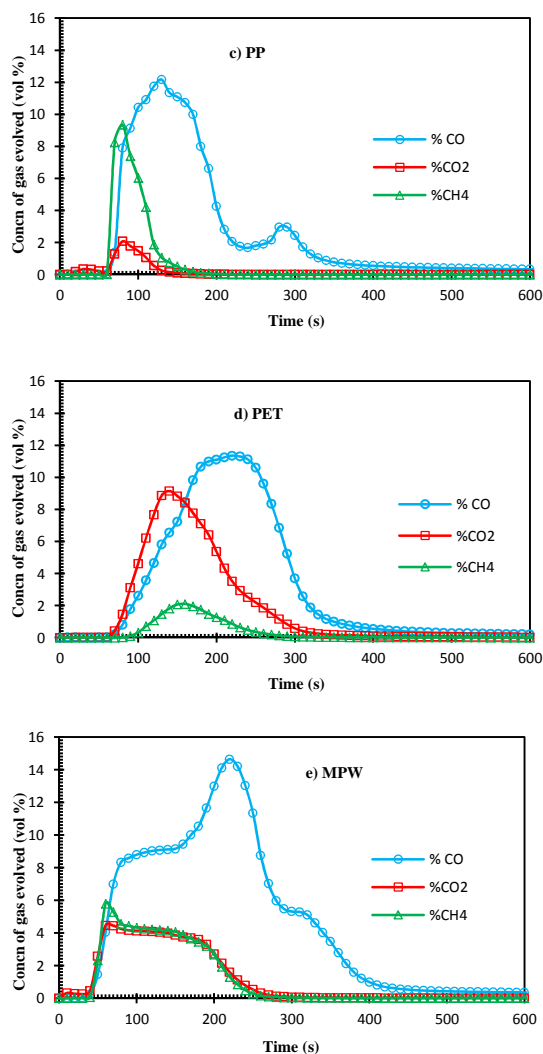
Fig. 9 Concentration of Gas Generated During the Reduction of Fe_2O_3 by each Carbonaceous Reductant at 1520 °C

For all the polymers utilised in this investigation, CO emissions are higher than the corresponding emissions for CO_2. The relatively lower values recorded for CO_2 compared to CO may be an indication of direct reduction of Fe_2O_3 by C (equation 1) or a dominant Boudouard reaction (equation 2) or carbon gasification reaction (equation 3), Dankwah et al., 2011.

$$Fe_2O_3 + 3C = 2Fe + 3CO \qquad (1)$$
$$CO_2 + C = 2CO \qquad (2)$$
$$H_2O + C = CO + H_2 \qquad (3)$$

Another possible reaction is the direct reduction of Fe_2O_3 by CH_4, which was the predominant hydrocarbon detected in the off-gas above 1500 °C.

$$3CH_4 + Fe_2O_3 = 2Fe + 6H_2 + 3CO \qquad (4)$$
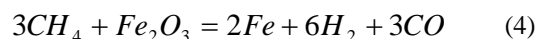
The free energy change for equation (4) is expressed as a function of temperature by equation (5), (Barin, 1995) with an equilibrium temperature of approximately 650 °C and well below the experimental temperature of 1520 °C.

$$\Delta G = 241,925.6 - 261.73T \quad [J\ mol^{-1}] \qquad (5)$$

Aside of solid carbon and gaseous hydrogen, Fig. 9 reveals that the gaseous reductants CO and CH_4 are also released into the furnace environment when waste polymers are used as reductants for metal oxide reduction at high temperatures.

3.3 Extent of Reduction

The oxygen contents of the reduced pellets were measured using a LECO Nitrogen/Oxygen analyser (model TC-436 DR 602-500-600, LECO Corporation, Michigan, USA). The extent of reduction was determined from the oxygen values based on the initial concentration of removable oxygen from the reagent grade iron oxide (Fe_2O_3), which is about 30.06 wt. %. The results are compared with reduction by metallurgical coke under the same experimental conditions and are illustrated in Fig 10 for all the reductants and Fig. 11 for PWS and Coke. Reduction was very rapid for all the polymers and it was essentially complete in less than 300 s compared to metallurgical coke that required about 600 s to reach completion. The observed superior reduction of iron oxide by the polymers over reduction by metallurgical coke is attributed to the extra reducing gases H_2 and CH_4 made available in the reaction system through the thermal decomposition of the polymers. The beneficial effect of H_2 on the reduction of iron oxide by carbon was elucidated in earlier investigation (Bonalde et al., 2005; Dankwah et al., 2011; Dankwah et al., 2013; El-Geassy and Rajakumar, 1985; Ono-Nakazato et al., 2003; Donskoi et al., 2003; Shi et al., 2008). Blending of the polymers resulted in an improvement in reduction with the times for the observed complete reduction of the pellet being 270 s, 220 s, 210 s, 190 s and 140 s for PWS, LDPE, PP, PET and MPW, respectively.
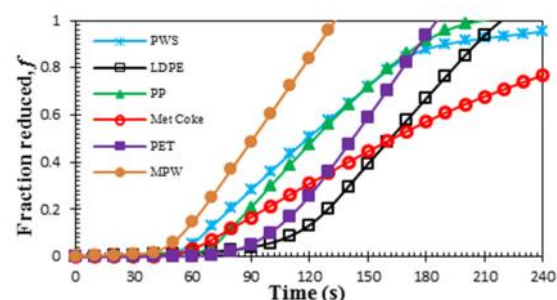
Fig 10 Observed Extent of Reduction for each Carbonaceous Reductant during the Reaction of Fe_2O_3 by Various Reductants
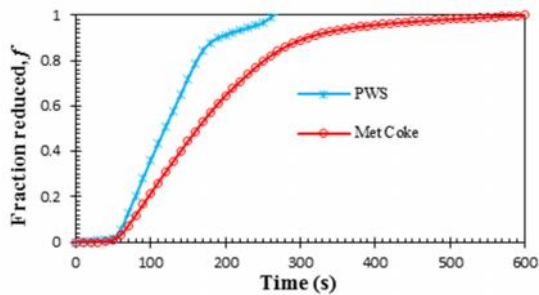
Fig. 11 Observed Extent of Reduction for PWS and Coke

The observed improvement in extent of reduction in the presence of polymer is consistent with previous observations by Rahman *et al.,*2006 (sessile drop approach), Kongkarat *et al.*, 2011 (sessile drop approach), Dankwah *et al.,* 2011 and Dankwah *et al.,* 2012 (crucible reduction approach).

## 3.4 Environmental Considerations (CO_2 evolution)

The concentration (vol %) and accumulated amount (mol) of CO_2 evolved from the reduction of iron oxide by each carbonaceous reductant are compared with those of conventional metallurgical coke under the same experimental conditions. The results are shown in Fig 12. As would be expected, the highest concentration (9.15 vol %) was recorded in the reduction by PET, followed by metallurgical coke (7.43 vol %) and MPW (4.46 vol %). The minimum concentration was observed for the reduction by PWS (1.63 vol %). However, the total (accumulated) amount of CO_2 emissions by each carbonaceous material is a function of the area under each graph.
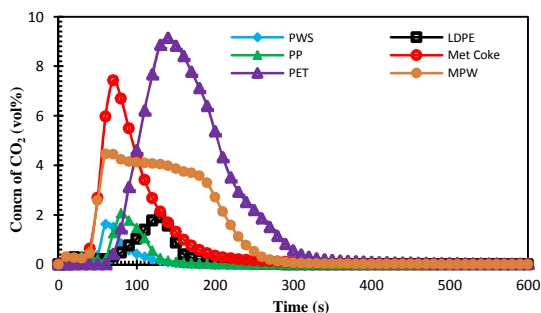
Fig.12 Concentration of CO_2 Generated from the Reactions of Fe_2O_3 with each Carbonaceous Reductant at 1520 °C

The accumulated amount of CO_2 evolved by each carbonaceous material is shown in Fig 13. Again, as would be expected, the highest accumulated amount of CO_2 is observed for the reduction by PET. Incorporation of PET in the mixed plastics waste (MPW) tends to negatively affect the emission behaviour of MPW and it places it above Met Coke. The large CO_2 emissions by PET was reported by

Dankwah *et al.*, 2014 and was ascribed to the high oxygen content of PET (33.3 wt %). Currently, PET is the most widely recycled plastics globally with about 53.2% recycling rate in Australia (Anon., 2012). Accordingly, just as the chlorine content in polyvinyl chloride (PVC) prohibits its usage as reductant (to prevent dioxin formation and high temperature corrosion of tanks), the large oxygen content in PET may restrict its incorporation into MPW to values less than 10 wt% to minimise CO_2 emissions.
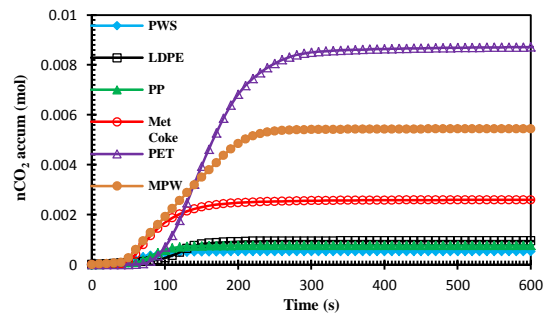
Fig. 13 Accumulated Amount of CO_2 Generated from the Reactions of Iron Oxide with each Carbonaceous Reductant at 1520 °C

In its place, oxygen-free polymers like polystyrene (PS) and expanded polystyrene (EPS) could be used. Work is underway to ascertain the effect of PET replacement by PS and or EPS on direct CO_2 emissions, when MPW is used as reductant in ironmaking.

The very low CO_2 emissions by PWS, PP and LDPE agree with the observations by Matsuda *et al.*, 2008, that metallic iron can be produced from iron oxide using waste plastics as reductants, with virtually no CO_2 emissions.

The CO_2 emissions discussed thus far are direct emissions from the reduction of Fe_2O_3 by the various reductants utilised in this investigation. These emissions do not take into account CO_2 emissions arising out of the electrical energy that was used to power the furnaces for the reaction. The calorific values of three of the polymers (PWS, LDPE, PP) utilised in this investigation are higher than that of metallurgical coke or solid carbon (Rose and Cooper, 1977; Scott, 1999; EPIC, 2004). Accordingly, electrical energy consumption is expected to be lower for MPW than for metallurgical coke. This will lead to further reduction in CO_2 emissions when polymers are used as reductants. Singh *et al.,* (2010) observed a 6.5% reduction in CO_2 emissions when coal was fired with 25% HDPE (measured in terms of thermal energy). They attributed this to the higher heating value of HDPE (46.2 MJ/kg) as compared with that of coal.

The use of waste polymers as reductants for iron oxide reduction in the manner described in this

investigation makes the concept ideal for application in rotary hearth furnaces, where the strength of pellets is not an issue. For application into Blast furnace ironmaking, studies into the compressive strength of the green iron oxide-polymer composite pellets are essential to ensure that pellets do not rupture into fine powders in the cohesive zone when charged from the top. Work is currently underway on room temperature strength development of green iron oxide-polymer composite pellets.

4 Conclusions

The reduction of iron oxide has been investigated using carbonaceous material prepared from mixed plastics waste. Major findings of the investigation are:

(i) Iron oxide can be successfully reduced using carbonaceous materials generated from MPW.

(ii) Gas analysis studies revealed that the exhaust gas consists of CO_2, CO and CH_4.

(iii) Blending the individual polymers to form MPW resulted in an improvement in the extent of reduction.

(iv) Direct CO_2 emissions by PWS, PP and LDPE were lower than those by metallurgical coke under the same experimental conditions.

However, direct CO_2 emissions by PET and MPW were higher than those by metallurgical coke. This was attributed to the high oxygen content of PET.

Acknowledgements

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