

Production of Iron Nuggets from the Akpafu-Todzi Iron Ore and Artisanal Ferrous Slag using Post Consumer Thermosets (Waste Electrical Sockets) as Reductants*

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Abstract

Post-consumer thermosets are difficult to recycle because, unlike thermoplastics, they cannot be remoulded to create other items as a result of the extensive cross-linkages in their structure. The increased production of thermoset blends and composites in recent years has greatly increased the amount of waste materials. However, higher levels of carbon and hydrogen present in thermosets make them a potential reductant in the iron extractive industries. In this research work, postconsumer thermoset was transformed into carbon resource through a charring process. The resulting carbonaceous material from the thermoset was used as reductant in the production of metallic iron from the Akpafu-Todzi iron ore and artisanal slag using the microwave technology through the composite pellet approach at varying firing times. Analyses by XRF, XRD and SEM/EDS showed that the Akpafu Todzi iron ore is comprised of the iron oxides hematite (Fe_2O_3) and wustite ($\text{Fe}_{0.942}\text{O}$), while the artisanal slag was predominantly fayalite (Fe_2SiO_4). Complete reduction of the ore was attained after 120 min reduction but the maximum extent of reduction was 78.84% for the slag, demonstrating the potential of postconsumer thermosets to function effectively as a reductant in the iron extractive industry.

Keywords: Reduction; Akpafu-Todzi Iron Ore; Post Consumer Thermosets; Waste Electrical Sockets

1 Introduction

Waste electrical sockets (WES) are difficult to recycle because various forms are produced from thermosetting polymers which cannot be remoulded after setting. Currently, incineration at landfills remains the major mode for recycling WES. However, uncontrolled incineration of WES in the open could be a harmful process because electrical sockets are produced from melamine or urea formaldehyde, making products of such incineration processes potential hazardous materials. Work done by Dankwah *et al.*, (2016a) has shown that controlled charring can result in the production of charred carbonaceous matter that can serve as potential reductants for iron oxide reduction.

Some research investigations have been conducted on utilisation of postconsumer plastics as reductants or as a source of energy in the metallurgical field (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.*, 2013; Dankwah and Koshy, 2014; Dankwah *et al.*, 2015a; Dankwah *et al.*, 2015b). A review of these investigations shows that

thermoplastic polymers and or their blends with metallurgical coke, graphite, or biomass are the dominant reductants often used for the production of metallic iron from reagent grade iron oxides or electric arc furnace (EAF) slags containing iron oxide (Dankwah *et al.*, 2016). In addition to the above polymers and their blends, other researchers have utilised elastomers and thermosetting plastics as reductants (Dankwah *et al.*, 2012; Mansuri *et al.*, 2013; Rajarao *et al.*, 2014a, Rajarao *et al.*, 2014b, Nath *et al.*, 2012, Dhunna *et al.*, 2014 and Dankwah and Baawuah, 2015). This category of polymers includes end-of-life rubber tyres, compact discs, melamine and bakelite as reductants or for carbon dissolution studies. Rajarao *et al.* (2014b) studied the structural changes in the chars produced during the rapid pyrolysis of waste compact discs (CDs) in the temperature range 550-1550 °C. They observed an increase in the porosity and C/O ratios of the chars with an increase in the pyrolysis temperature up to 850 °C. They achieved a maximum porosity at 850 °C with char surface area of 334 m²/g. Using the chars produced at 1550 °C as reductants they were able to achieve about 90% iron oxide reduction (Dankwah *et al.*, 2016).

Recovery of iron from waste bearing ferrous slags is more complicated than from the naturally occurring

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oxide because the slag typically exists as a spinel fayalite, which is difficult to reduce carbothermally. Cheng *et al.* (2013) investigated the direct reduction experiment on iron-bearing waste slag. They observed that addition of Al_2O_3 to the slag can improve the metallisation rate from between 74–77% to over 88% at temperatures around 1250 °C. Heo *et al.* (2016) investigated the recovery of iron and removal of hazardous elements from waste copper slag *via* a novel aluminothermic smelting reduction (ASR) process. They observed that iron recovery exhibited a maximum value at Al/FeO ratio of 0.53. Sarfo *et al.* (2017) investigated the carbothermal reduction of copper smelter slag for recycling into pig iron and glass. They observed that most of the copper and iron can be recovered as pig iron from the initial slag using minor amounts of lime and alumina and treating at 1350 °C or higher for 30–90 min.

Dankwah *et al.* (2016a) were able to produce premium grade metallic iron nuggets from the Agbaja iron ore (Nigeria) using charred carbonaceous materials obtained from WES. More information is needed in the literature on the use of end-of-life thermosets as reductants for the production of metallic iron from other naturally occurring iron ores.

The Akpafu-Todzi iron deposit is located in the Volta Region of Ghana and is believed to be a low to medium grade ore. Processing of the ore has been on a limited scale by the local blacksmiths for simple implements like hoes and cutlasses owing to the location and grade of the ore as well as a lack of appropriate source of carbonaceous materials for effective reduction. For reduction studies, Dankwah *et al.* (2016b) produced metallic iron from the Akpafu-Todzi ore using carbonaceous materials generated from palm kernel shells and their blends with post-consumer high density polyethylene. However, the nature of the artisanal slag generated, as waste material as well as its processing to create valuable materials has not been widely researched into.

This work therefore investigates the potential for utilising WES as reductant for the production of metallic iron from the Akpafu-Todzi iron ore and samples of slag materials through the microwave approach. These slag samples were part of the waste obtained from the reduction of the ore by the local artisans.

2 Resources and Methods Used

2.1 Carbonaceous Materials Preparation

Samples of WES (Fig. 1) were collected from the surroundings of the University of Mines and

Technology (UMaT), Tarkwa. These were broken down into pieces and washed thoroughly to remove dust. The cleaned thermosets were thereafter air dried for two days. This was followed by charring under controlled temperature conditions for 45 mins using a gas fired furnace.



Fig. 1 Waste Electrical Socket

The charred thermoset (Fig. 2) was ground using a ball mill for 10 minutes. The ground thermoset (Fig. 3) was screened for 15 minutes at amplitude of 20 using standard sieve sizes of 106 μ m, 90 μ m, 75 μ m and 53 μ m (Dankwah *et al.*, 2016a).



Fig. 2 Charred Electrical Socket



Fig. 3 Samples of Pulverised Charred Electrical Socket

2.2 Preparation of Ore Samples

Medium grade Akpafu-Todzi iron ore (65.947 wt % Fe_2O_3) and artisanal slag samples (70.377) were used as the source of iron oxide (Table 1). The morphologies of the Akpafu-Todzi iron ore (Fig. 4) and the slag (Fig. 5) were observed by Scanning Electron Microscopy (SEM). The pulverised samples were characterised by XRD using an EMPYREAN Diffractometer.

Table 1 Chemical Composition (XRF) of Akpafu-Todzi Iron Ore and Artisanal Slag

Component	Composition (wt %)	
	Ore	Slag
Fe ₂ O ₃	65.947	70.377
SiO ₂	20.055	28.412
Al ₂ O ₃	2.340	2.251
CaO	0.043	1.795
Mn ₃ O ₄	0.043	1.433
TiO ₂	0.087	0.112
Cr ₂ O ₃	1.218	0.344
MgO	0.154	0.505
P ₂ O ₅	0.296	0.168
SO ₃	0.159	0.046
LOI	11.18	5.02



Fig. 4 Spherical Pellets formed from Milled Ore Sample and WES



Fig. 5 Spherical Pellets formed from Milled Slag Sample and WES

2.2.1 Reduction Studies

Each dry pellet was placed in a fireclay crucible and positioned at the central location of the microwave as shown in (Fig. 6) to ensure maximum concentration of the radiation emitted. The crucible was taken out after the desired time of firing and quenched in air to stop any further reactions. The weight of reduced iron pellet was recorded. This procedure was repeated for the rest of the pellets at different firing times.

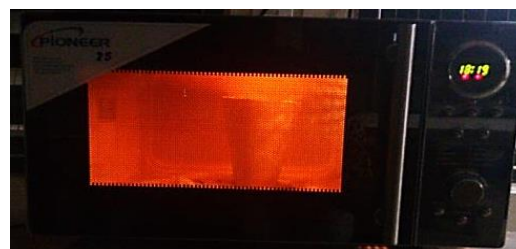
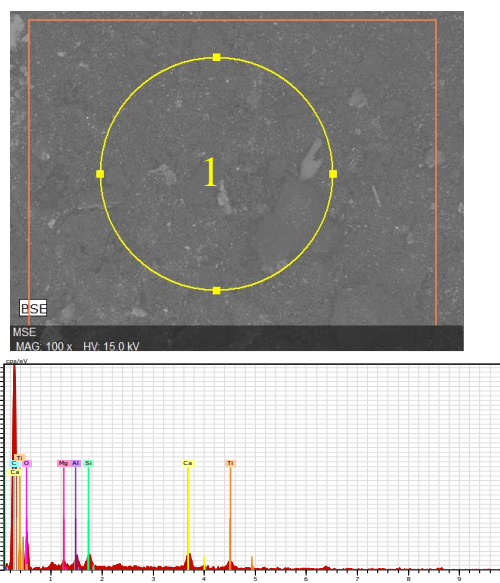


Fig. 6 Reduction Process in a Domestic Microwave Oven

3 Results and Discussion

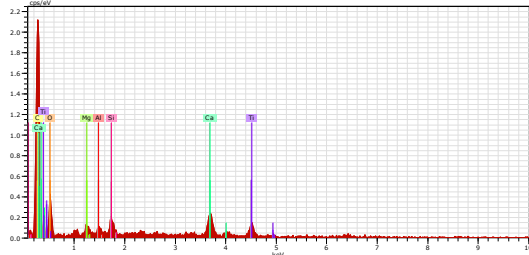
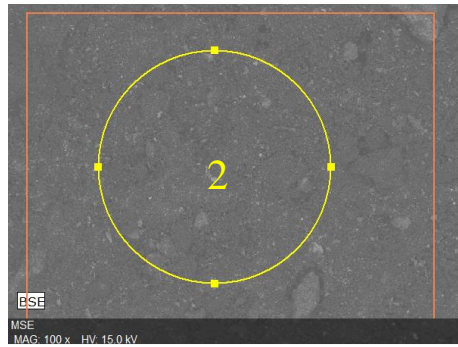
3.1 Nature of Charred Carbonaceous Material Derived from WES

Figs. 7 and 8 show SEM/EDS analyses of the solid product obtained after charring WES. Elemental analyses of regions 1 and 2 show a carbon content (of 67.35 wt % and 64.13) reasonably above those of various forms of coals (~60 wt %) and polyethylene terephthalate (PET) (~62.5 wt %). It should therefore be possible to reduce iron oxide with charred WES. However, the efficiency of reduction will depend on the nature of ash obtained after the charring process.



Element (K-series)	Weight %	Atomic %
C	67.35	76.11
O	24.56	20.84
Ca	3.26	1.11
Ti	2.17	0.62
Si	1.31	0.63
Al	0.90	0.45
Mg	0.44	0.24
SUM	100	100

Fig 7 SEM/EDX Analysis of Region 1 from Charred WES



Element (K-series)	Weight %	Atomic %
C	64.13	74.06
O	25.01	21.69
Ca	4.18	1.45
Ti	2.95	0.86
Si	1.80	0.89
Mg	1.19	0.68
Al	0.74	0.38
SUM	100	100

Fig. 8 SEM/EDX Analysis of Region 2 from Charred WES

The morphology of the ash obtained after the charring process is shown in the SEM of Fig 9 whilst the XRD is shown in Fig 10. The ash particles appear to be irregularly shaped as indicated in Fig 6 and consist of several crystalline peaks of calcite, quartz, corundum, halite, silicon and bornite.

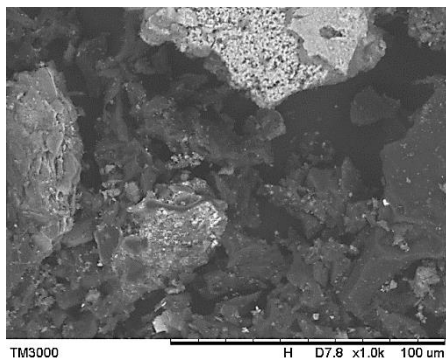


Fig. 9 SEM Photomicrograph of Ash obtained after Charring of WES (Dankwah *et al.*, 2016a)

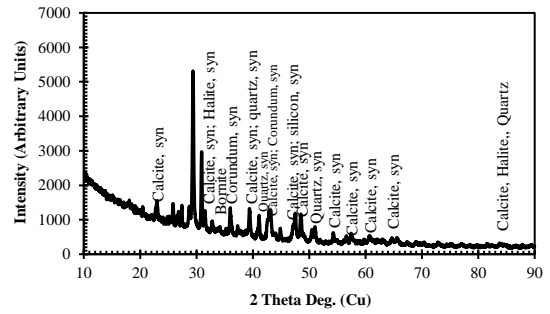
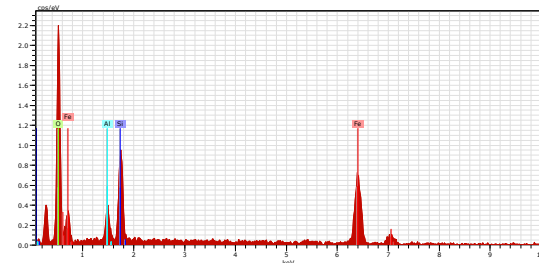
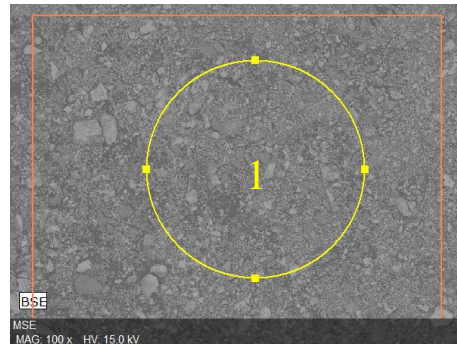


Fig. 10 XRD Patterns of Ash obtained after the Charring Process (Dankwah *et al.*, 2016a)

3.2 The Nature of the Akpafu-Todzi Iron Ore

The SEM/EDS analyses of the Akpafu-Todzi iron ore before reduction are illustrated in Figs 11 and 12 for two regions 1 and 2. Region 1 shows an iron content of 44.07 wt %, with Region 2 slightly different at 42.88 wt %.



Element (K-series)	Weight %	Atomic %
Fe	44.07	20.45
O	39.83	64.52
Si	11.93	11.01
Al	4.18	4.01
SUM	100	100

Fig. 11 SEM/EDS Analysis of Akpafu-Todzi Iron Ore (Region 1) (Dankwah *et al.*, 2016b)

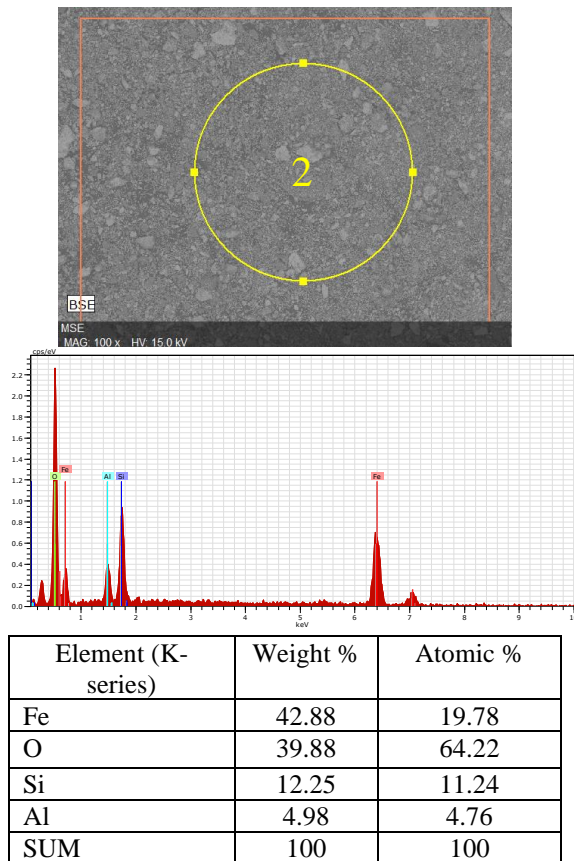


Fig. 12 SEM/EDS Analysis of Akpafu-Todzi Iron Ore (Region 2) (Dankwah *et al.*, 2016b)

The ore was also characterised by XRD. The diffraction patterns are shown in Fig 13. The diffraction patterns correspond to those of SiO₂, Fe_{0.942}O and Fe₂O₃. Contrary to earlier observations that the Akpafu Todzi iron consists of predominantly of hematite (Fe₂O₃) and quartz (SiO₂), peaks of wustite (Fe_{0.942}O) are seen in the diffractogram of the ore shown in Fig. 13, suggesting that iron exists as the mixed ferrous oxides wustite and hematite. Production of metallic iron from the ore therefore requires simultaneous reduction of both hematite and wustite according to Equations (1) and (2):

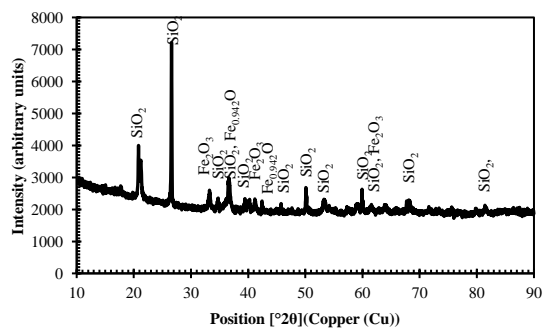
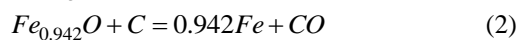


Fig 13. XRD of Akpafu-Todzi Iron Ore

3.3 The Nature of the Akpafu-Todzi Slag

The SEM/EDS analyses of the Akpafu-Todzi slag before reduction are illustrated in Figs 14 and 15 for two regions 1 and 2. Region 1 shows an iron content of 50.93 wt %, with Region 2 not very different at 51.73 wt %. These values are higher than those observed for the ore and are just below the minimum 55 wt % Fe required for economic extraction of iron ores.

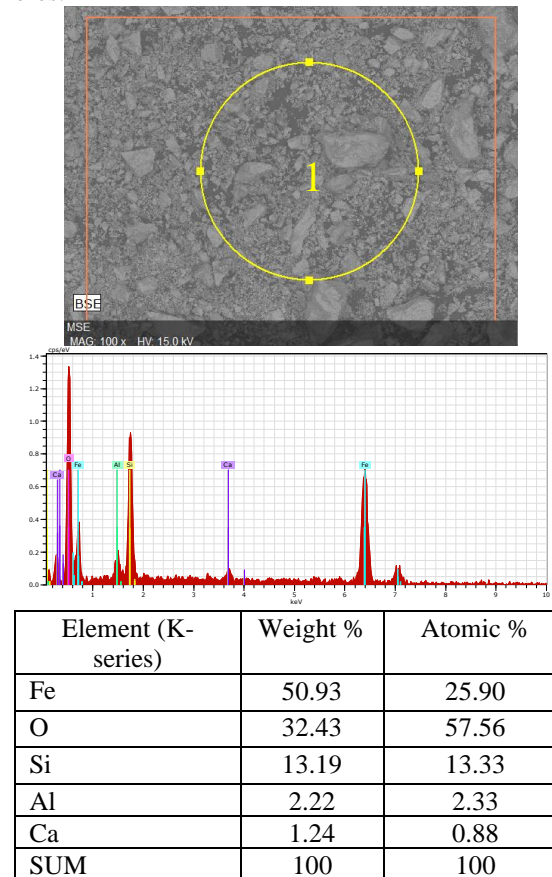
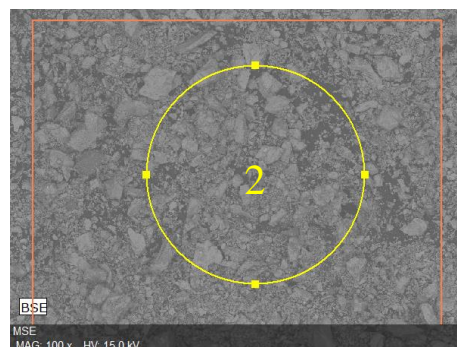
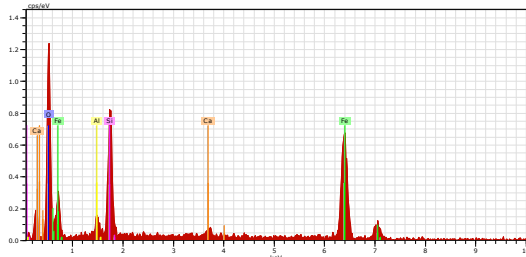


Fig. 14 SEM/EDS Analysis of Akpafu-Todzi Slag (Region 1)





Element (K-series)	Weight %	Atomic %
Fe	51.73	26.63
O	31.37	56.37
Si	13.82	14.14
Al	1.88	2.00
Ca	1.20	0.86
SUM	100	100

Fig. 15. SEM/EDS Analysis of Akpafu-Todzi Slag (Region 2)

The slag was also characterised by XRD. The diffraction patterns are shown in Fig. 16. The diffraction patterns correspond predominantly to those of fayalite (Fe_2SiO_4) and some SiO_2 .

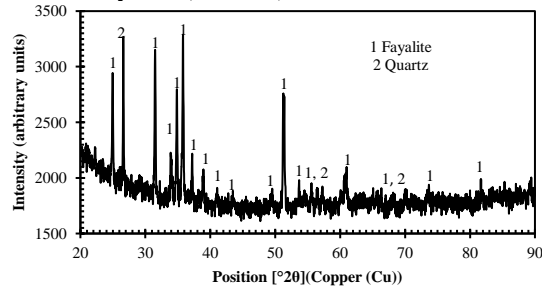


Fig. 16 XRD of Akpafu-Todzi Artisanal Slag

3.4 Reduction of Akpafu-Todzi Iron Ore and Slag by WES

Fig. 17 shows the nature of metal produced from the reduction of the Akpafu-Todzi slag by carbonaceous materials generated from WES.



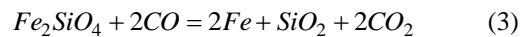
Fig. 17 Green Ore Pellet before and after Firing Slag-WES Composite Pellet

The reduced metals were separated from the unreduced portion of the slag using a hand-held magnetic. Part of the separated metals is shown in Fig. 18.



Fig. 18 Metallic Iron Nuggets formed from Slag Sample after Firing

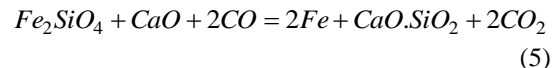
The XRD diffractograms in Fig. 16 showed the slag as consisting predominantly of fayalite (Fe_2SiO_4) and quartz (SiO_2). The possible reactions in the microwave oven are:



The Gibbs free energy change for reaction (3) is given by the expression in eqn. (4), with an equilibrium temperature of approximately 910 °C:

$$\Delta G = 12\,477 - 10.55T \quad (4)$$

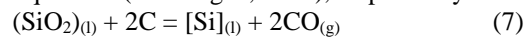
Thermodynamic calculation results of fayalite reduction by CO indicates that addition of an alkaline oxide like CaO can improve the fayalite reduction by chemically combining with SiO_2 to form wollastonite ($CaO \cdot SiO_2$) as shown in eqn. (5) (Xian-Lin *et al.*, 2015; Cheng *et al.*, 2013)



$$\Delta G = -79\,956 + 13.06T \quad (6)$$

Increasing the content of CaO will enhance the formation of dicalcium silicate ($2CaO \cdot SiO_2$) by solid-phase reaction (Xian-Lin *et al.*, 2015; Cheng *et al.*, 2013; Heo *et al.*, 2013). This contributes to the separation of slag and iron by natural pulverisation of dicalcium silicate during cooling (Xian-Lin *et al.*, 2015; Cheng *et al.*, 2013; Heo *et al.*, 2013).

The reduction of silica by carbon proceeds according to the steps below. In the slag phase SiO_2 can be carbothermally reduced to produce silicon metalloid or the refractory material silicon carbide (SiC), according to eqn. 7-8 (Safarian *et al.*, 2008) and eqn. 9-10 (Turkdogan, 1980), respectively.

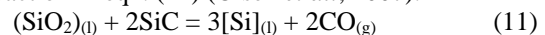


$$\Delta G^\circ = 677\,662 - 347 T \text{ (J)} \quad (8)$$



$$\Delta G^\circ = 604\,679 - 339 T \text{ (J)} \quad (10)$$

Silica can be reduced by SiC in accordance with the reaction in eqn. (11) (Olsen *et al.*, 2007).



$$\Delta G^\circ = 823\,627 - 362 T \text{ (J)} \quad (12)$$

The equilibrium temperatures for the reaction equations (7), (9) and (11) are 1680, 1510 and 2002 °C, respectively. These are extremely high temperatures and processes relying on such reactions would typically consume large amounts of energy.

The maximum temperature recorded in the microwave was about 1197 °C, far below the melting point of metallic iron and excluding the reduction of SiO₂ to Si. The metal formed was spherical, suggesting that it solidified from the liquid state. The metal formed is therefore highly carburised or contained some other alloying elements. However, the metal produced from the ore is irregularly shaped as shown in Fig. 19. It suggests solid state reduction.



Fig. 19 Reduced Iron formed from Ore after Firing

3.5 Extent of Reduction of Akpafu-Todzi Iron Ore and Slag by WES

The extents of reduction of the ore and slag by WES were measured at different times. The results are shown in Fig. 20.

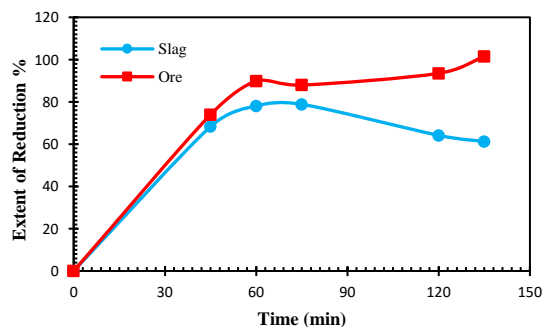


Fig. 20 Graph of Extent of Reduction versus Firing Time

Whereas the ore attained complete reduction, the maximum extent of reduction for the slag is 78.84 %. This disparity in extent of reduction can be attributed to the relative difficulty in reduction of fayalite (the major composition of the slag) as opposed to the reduction of hematite and wustite (from the ore). Accordingly, it is much easier to reduce the ore than the slag, although the content of Fe in the latter is higher. The 78.84% extent of reduction of fayalite is similar to results obtained by

Cheng *et al.* (2013) who attained a metallisation of 77% and 74% at 1250 °C and 1200 °C, respectively. Consequently, even without the usual CaO and/or Al₂O₃ additions, charred WES can perform effectively as reductant for the Akpafu-Todzi ore and the artisanal slag.

4 Conclusions

The reduction of the Akpafu-Todzi iron ore and slag has been investigated using carbonaceous material prepared from waste electrical sockets. Major findings of the investigation are:

- i. Waste electrical sockets (WES) are a potential source of carbonaceous materials for iron oxide reduction:
- ii. The Akpafu-Todzi ore consists of the mixed ferrous ores hematite (Fe₂O₃) and wustite (Fe_{0.942}O) along with quartz (SiO₂):
- iii. The Akpafu-Todzi slag is predominantly a fayalite (Fe₂SiO₄) with significant amounts of quartz.
- iv. The Akpafu-Todzi iron ore and slag can be successfully reduced using carbonaceous materials generated from WES.

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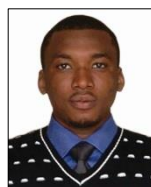
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