Utilisation of Polyethylene Terephthalate as a Reductant in Ferrosilicon Production from Pudo Iron Ore and End-of-life Automotive Windshield*

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Abstract

Ferrosilicon is a well-known alloy used in the steel industry to improve the mechanical and physical properties of steel. It is produced through the reduction of iron ore and silica in the presence of a carbonaceous reductant at high temperatures. In this study, we investigated the use of a non-traditional reductant, polyethylene terephthalate (PET), for the production of ferrosilicon, using Pudo iron ore and a waste material, automotive windshield glass (AWS). The experimental approach involved a two-stage process: (1) the production of a reducing agent from PET, and (2) the reduction of Pudo iron ore and silica in the presence of the reducing agent. Samples of the Pudo iron ore, automotive windscreen glass and embrittled samples of waste PET were pulverised to -250 µm. The pulverized PET was mixed in proportions with the pulverised Pudo iron ore, flour and water to form three composite pellets. The pellets upon drying were heated in a domestic microwave oven (DMO) for 40 minutes at a temperature of 1197 ºC. The products were characterized using SEM/EDX and XRF. The ferrosilicon produced had a chemical composition of 1.71% Si, 3.38% C, 1.26% Al, 0.92% Mg, 2.21% Ca, 1.29% Na, 5.21% O and 84.02% Fe. The use of PET as a reductant could provide a more sustainable and cost-effective way of producing ferrosilicon, utilizing waste materials as feedstock. However, the results showed that the silica in the AWS was a poor absorber of microwave energy, making direct alloy formation of ferrosilicon difficult. To improve the efficiency of the process, the authors suggested extending the time of heating in the DMO beyond 40 minutes. Additionally, future work could explore alternative heating methods that are more efficient in reducing the silica in the AWS.

Keywords: Pudo iron ore, automotive windshield, PET, reduction, ferrosilicon, XRF, SEM/EDX.

1 Introduction

Waste management is a challenge facing humanity in modern times and yet technology has not been able to effectively control it in communities worldwide as it appears to have rather worsened the situation (Kwawe, 1995). According to Lyse (2003), Nine (9) out of every 10 African cities are facing serious waste disposal problems. With these wastes generated, one third to one half within most cities in developing nations, (including Ghana) are not collected (UNCHS, 1996). The waste composition in Ghana showed 61% organics, 14% plastics, 6% inert, 5% miscellaneous, 5% paper, 3% metals, 3% glass, 1% leather and rubber, and 1% textiles (Miezah *et al.*, 2015). It is estimated that about 83% of the population in Ghana dump their refuse in either authorised or unauthorised sites in their neighbourhood and due to poor handling of solid waste, unsanitary conditions are created (Benneh *et al.*, 1993).

Glass which forms 3%, plays a very significant role in our society and it is widely used in automobile windscreen production, as a packaging material and in other domestic applications. The most common types of glass are sand (silica) based and the most important, soda-lime silica glass (constituting over 90% of all glass made). Soda-lime silica glass consists of four main ingredients: 61% sand (with an iron content of <0.03%), 18% soda, 13% limestone or lime, 8% other components, mainly alumina, magnesia and refining agents (Abraham, 2015). Plastics account for a whopping 14% of the waste stream hence calls for urgent attention. Consumer plastics consist of various elements such as carbon, hydrogen, oxygen, nitrogen, chlorine and sulphur. Since plastic materials are predominantly hydrocarbons, they can serve as a suitable reductant in the iron making industry (Dankwah *et al.*, 2015).

In Ghana, there are three main iron ore deposits that can potentially be used in iron making and they are; Shieni sedimentary iron ore, Pudo titaniferous-

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magnetiferous iron ore, and Opon-Mansi laterite iron ore. Depending on the location, Pudo iron deposit is known to be a titaniferrous magnetiferrous ore, containing both magnetite and titanium (Kesse and Banson, 1975). Recent works by Aakyiir and Dankwah (2017) shows a non-magnetic ore of relatively low iron oxide. The work done by Kesse and Banson (1975) again showed the non-existence of phosphorus and an extremely low sulphur content when samples of Pudo iron ore were chemically analysed. In reducing Pudo iron ore, work by Dankwah *et al.* (2011) showed that the thermal decomposition of plastic waste materials releases the gaseous reduction species carbon monoxide (CO), methane (CH_4) and hydrogen (H_2) in addition to nanoscale carbon, which are possible reducing agents for metal oxides.

The following equations (1, 2 and 3) can demonstrate the progress of ferrosilicon production:

$Fe_2O_3 + 3C \rightarrow 2Fe + 3CO$ ………. (1)

$SiO_2 + 2C \rightarrow Si + 2CO$ …………… (2)

$xFe + ySi \rightarrow FexSiy$ …………….. (3)

So far, the ferrosilicon industry relies on metallurgical coke as its primary source of reductant and quartz as a source of silica resulting in increasing environmental risk relating to CO_2 emission along with the exorbitant cost of high-grade metallurgical coke. Schei *et al.* (1998) has stated that, the resources for reduction materials are more limited, hence cheaper and more environmentally friendly sources of reductant and silica must be explored.

The process of producing ferrosilicon involves loading metallic iron, silica, and a carbonaceous reducing agent into a submerged furnace to ensure that the heat generated is efficiently transferred to enhance the reduction process, which results in the production of iron-silicon alloys (Dankwah, 2018). Fig. 1 displays the Fe-Si phase diagram for the temperature range 500 to 1700 °C. In the solid state, FeSi, $FeSi_2$, and Si are the stable phases at low temperature on the Si-rich side. However, Fe_3Si is the stable phase in the iron-rich region, with metastable Fe_5Si_3 forming within 825 °C to 1030 °C. It is apparent from Fig. 1 that within 50 wt. % Si, ferrosilicon alloy consists of mainly FeSi, $FeSi_2$ and some Fe_3Si/Fe_5Si_3 phases.

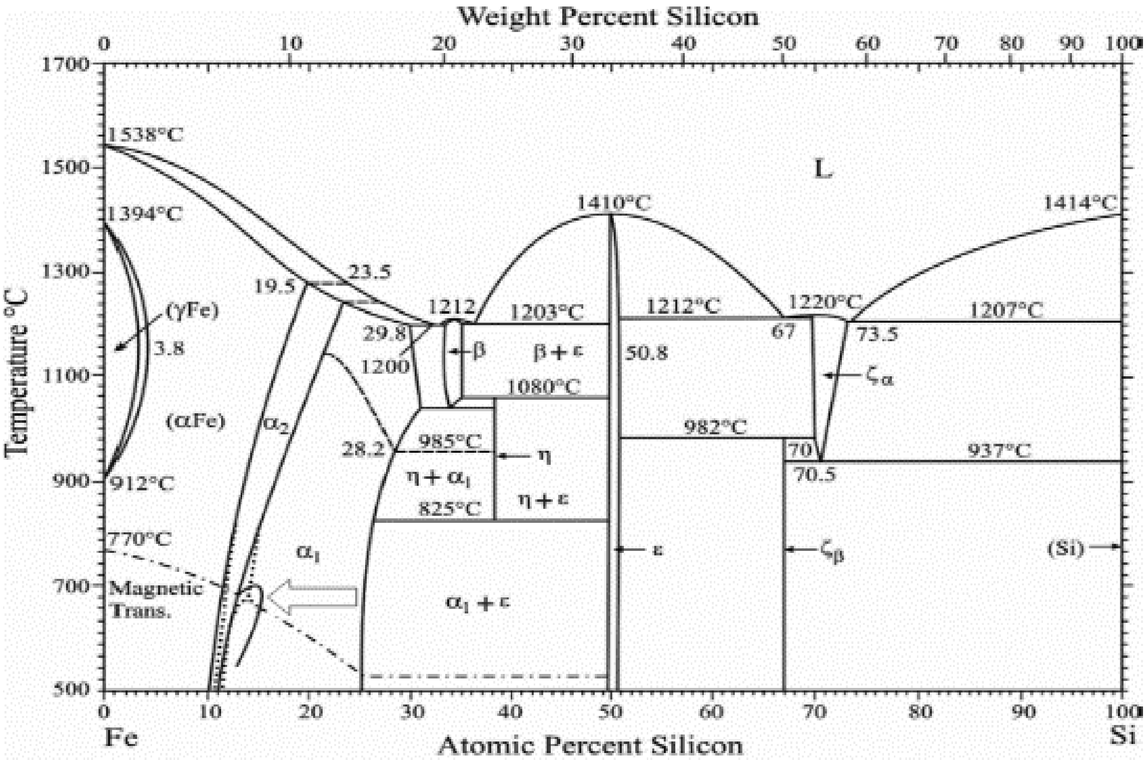

Fig. 1 Iron-Silicon Phase Diagram (Kubaschewski, 1982)

Ferrosilicon is an alloy of not less than 82% iron and 15-16% silicon. The alloy tends to corrode if the composition of silicon is less than 15% and the magnetic susceptibility of the alloy reduces if the silicon content is greater than 16%. The alloy is primarily used as a deoxidant in the manufacture of steel and cast iron. Ferrosilicon (FeSi) increases the strength, stiffness, temperateness, and corrosion resistance of steel (Buah, 2020).

Glass, in reflecting its abundant use today, is one of the main components of the waste stream. Automotive windscreen glass and consumer plastics have indeed become nuisance, health risky and a cost burden in the society. Inorganics forming 39% of the waste stream makes it worrying as they are not easily biodegraded. Recycling of recyclable part of these waste as raw materials for production will serve a purpose of protecting the environment, conserving resource, and promoting the socioeconomic development of Ghana. In 2010 alone, appreciable percentages of recycling were recorded across different countries worldwide; Austria achieved 70%, Germany achieved 62%, while Belgium achieved 62% (Abolo *et al.*, 2018).

The objective of this study is to explore the possibility of utilizing discarded automotive windshield glass (AWS) as a feasible source of silicon for the production of ferrosilicon from blends of Pudo iron ore, AWS and post-consumer plastics (PET) in a domestic microwave oven.

2 Resources and Method

2.1 Materials

The materials utilized in this research project comprised of a magnetic iron ore (depicted in Fig. 2) sourced from Pudo, located in the Upper West Region of the Republic of Ghana. Additionally, end-of-life automotive windshield (illustrated in Fig. 3) was acquired from Bogoso Junction, a suburb of Tarkwa. Similarly, end-of-life Polyethylene Terephthalate (PET) (shown in Fig. 4) was collected from the vicinity of the University of Mines and Technology. Wheat flour was procured from the Tarkwa Market.

Fig. 2 Pulverised Iron Ore Sample used for the Research

(a) (b)

Fig. 3 (a) Raw and (b) Pulverised Sample of Automotive Windshield (AWS)

(a) (b)

Fig. 4 (a) Spent PET Bottles and (b) PET Bottles Pulverised

2.2 Methods

The work was carried out in a series of stages, which involved the preparation of pulverized iron ore, carbonaceous materials from PET, and automotive windshield. The quantities of each component needed for the pellet formation were then calculated. The Pudo iron ore was blended with the automotive windshield and PET to form pellets. The pellets were then dried at room temperature and heated using microwaves. Temperature readings were recorded using a BENETECH GM1600 Infrared Thermometer (Fig. 5). Finally, the materials and products were characterized. These stages are described below.

Fig. 5 BENETECH GM1600 Infrared Thermometer used for Temperature Measurements in this Investigation

2.2.1 Preparation of Pulverised Iron Ore Sample

5 kg of the ore was sampled for this project. The sampled iron ore, with particle sizes which ranged from 120 mm to 150 mm was sun dried and control-crushed to sizes from 30 mm to 50 mm using a mallet. The reduced sample was subsequently passed through a jaw crusher to obtain particle sizes which ranged from 5 mm to 15 mm. The crushing was continued with a cone crusher which brought the particle size of the ore to -5 mm. The downsizing was finalized using a roll crusher which rendered the sample to a particle size range of 2.4 mm to 1.6 mm. The crushed sample was then milled using a ball mill for thirty minutes and sieved to 80% passing -250 µm particle size.

2.2.2 Making of Pulverised Carbonaceous Materials from PET

Samples of PET were cut with a pair of scissors, followed by heating to temperatures of around 180 °C to ensure melting of the polymer. The completely molten polymer was then quenched to render the whole mass brittle. The embrittled mass was milled and sieved to 80% passing -250 µm.

2.2.3 Preparation of Pulverised Automotive Windscreen

The end-of-life automotive windscreen glass weighing approximately 7 kg was initially crushed using a mallet and then downsized to -5 mm size range via a cone crusher. The resulting sample was further processed using a roll crusher, resulting in particle sizes ranging from 2.4 mm to 1.6 mm. Subsequently, the crushed glass was milled in a ball mill for 30 minutes and sieved to obtain a -250 µm particle size. Finally, a 50 g sample of wheat flour was screened with a 250 µm sieve and utilized as a binding agent in the subsequent stage of the project.

2.2.4 Pellets Formation and Drying

The pulverised automotive windscreen (AWS) and Pudo iron ore were blended in different proportions to form pellets. The proportion of Pudo iron ore was 80% and that of AWS was 20%, both held at a constant with 100% PET powder as reducing agent.

Pellets weighing 50 g were formed comprising 14 g of the reducing agent (PET), 1 g of flour as a binder, and 35 g of a mixture of the Pudo iron ore and the AWS as the source of silica inclusive.

The various components were mixed thoroughly in a bowl, with water added in drops. The wet material was then moulded into spherical balls with the aid of the binder in each case. A total of three pellets were formed from the Pudo iron ore and the automotive windshield glass with PET for the study and the average outcome determined. Fig. 6 shows a sample of the pellets.

The formed pellets were dried at room temperature for 72 hours to ensure the preservation of the chemical composition of the iron ore-polymer composites. Drying was done to remove moisture and to enhance the mechanical strength of the pellets for subsequent processes.

Fig. 6 Pellet formed from blend of Pudo iron ore, AWS and PET

2.2.5 Microwave Heating of Pellets

The dried pellets were heated in fireclay crucibles in a domestic microwave oven for 40 minutes (Binatone, 800 W, 2.45 GHz) (Fig. 7). The temperature of the hot pellet (1197 ºC) was recorded immediately after the crucible was withdrawn from the DMO. It was then allowed to cool.

Fig. 7 Domestic Microwave Oven (Binatone, 800 W, 2.45 GHz) used for the Investigation

2.2.6 Characterisation of Materials and Products

Analytical tools such as XRF and SEM/EDX were employed to determine the chemical compositions of materials and products involved in the experiments. The Pudo Iron Ore and end-of-life automotive windshield glass were analysed using XRF and the reduced pellets analysed by SEM/EDX.

3 Results and Discussion

3.1 Nature of the Pudo Iron Ore

The XRF analysis of the Pudo iron ore used for the research is illustrated in Table 1. Major compounds identified and shown in weight percentages are; Fe_2O_3, TiO_2, SiO_2, Al_2O_3, MgO, V_2O_5, and minor amounts of Na_2O, K_2O, Mn_3O_4, and CaO. The concentration of SiO_2 in the ore was ascertained to be 9.1%, a substantial finding. This contributed to the SiO_2 composition of the pellet formed, with end-of-life windshield glass representing the primary resource for SiO_2 content.

Table 1 Composition by XRF of Pudo Iron Ore

Component	Composition (wt. %)
Na_2O	0.13
MgO	2.71
Al_2O_3	7.56
SiO_2	9.1
P_2O_5	0.07
SO_3	0.02
K_2O	0.11
CaO	0.13
TiO_2	10.94
V_2O_5	1.075
Cr_2O_3	0.098
Mn_3O_4	0.24
Fe_2O_3	82.37
NiO	0.079
CuO	0.057
ZnO	0.022
HfO_2	0.034
L.O.I.	-14.66
TOTAL	100.09

3.2 Nature of Automotive Windscreen Glass

The XRF analysis of the automotive windscreen glass utilised in the research is illustrated in Table 2. Major compounds identified and shown in weight percentages are; SiO_2, Na_2O, CaO, MgO, V_2O_5 and minor amounts of Fe_2O_3, Al_2O_3, K_2O and SO_3.

Table 2 Composition by XRF of Automotive Windscreen Glass

Component	Composition (wt. %)
MgO	3.872
Al_2O_3	0.601
Na_2O	14.789
SiO_2	71.169
SO_3	0.123
K_2O	0.501
CaO	7.533
Fe_2O_3	0.764
Rb_2O	0.002
SrO	0.006
Y_2O_3	0.001
ZrO_2	0.003
ThO_2	0.001
LOI	0.635
SUM	100

3.3 Nature of Reduced Mass

A sample of the reduced hot product obtained after 40 minutes of microwave irradiation of pellets is shown in Fig. 8. Fig. 9 (a) shows a mixture of iron filings, spherical iron nuggets, and irregularly shaped forms of suspected particles of ferrosilicon alloy. Fig. 9 (b) shows isolated suspected particles of ferrosilicon after reduction. The spherically shaped metal nuggets (Fig. 9 b) indicate that the metal solidified from the liquid state. This is as a result of molten metal attempting to minimize surface energy. Iron filings were also obtained from the reduction of all pellets and were separated by a handheld low intensity magnet as shown in Fig. 10.

Fig. 8 Hot Reduced Pellet in a Crucible

(a) **(b)**

Fig. 9 (a) Microwave Irradiated Hot Mass Poured onto a Metallic Tray and (b) Cooled Reduced Mass

Fig. 10 Attraction of Reduced Mass (Metallic Filings) to a Magnet

The reduced pellet was analysed by SEM/EDX to determine the chemical composition of the mass. Fig. 11 shows the SEM/EDX photomicrograph of the reduced pellet revealing an uneven intrinsic particle size distribution. Particle shapes also differ across the reduced mass. There is a significant area in the pellet that appears light, indicating a reduction in metal oxides, while the small, dark regions dispersed within the lighter region signify unreduced portions of the pellet.

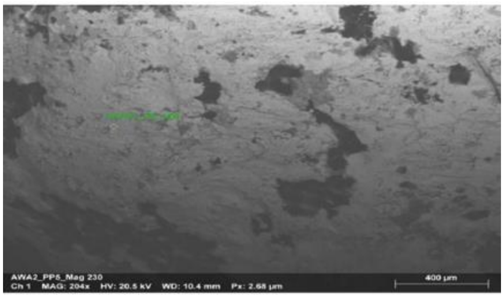

Fig. 11 SEM Photomicrograph of Reduced Pellet

Table 3 shows the chemical composition at 727.19 µm point on the 100% PET pellet by SEM/EDX analysis. The weight percentage of iron was found to be 84.02 with silicon being 1.71.

Table 3 Chemical Composition (by SEM/EDX) of the Reduced Pellet

Element	Weight [%]	Atom [%]
Carbon	3.38	11.91
Oxygen	5.21	13.77
Sodium	1.29	2.37
Aluminium	1.26	1.97
Silicon	1.71	2.58
Magnesium	0.92	1.62
Calcium	2.21	2.34
Iron	84.02	63.44
TOTAL	100	100

Fig. 12 and Fig. 13 show the SEM/EDX spectra of the sample metallic products. Fig. 12 reveals significant iron peaks with amounts of silica, chromium, sodium, carbon and aluminum.

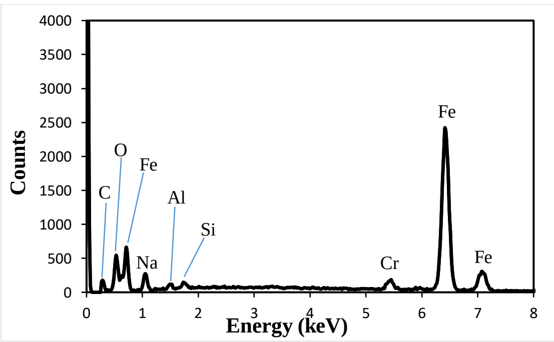

Fig. 12 SEM/EDX spectrum of Reduced Pellet

Fig. 13 and Fig. 14 are respectively the SEM/EDX line photomicrograph and line spectrum showing chemical composition across pellet surface over a distance of 1400 µm.

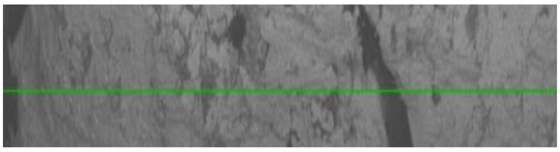

Fig. 13 SEM/EDX Line Photomicrograph

Observed in Fig. 14, the concentration percentage peaks of iron well dominated over the other elements across the surface followed by silicon. The highest concentration percentage peak of iron reached 84.02% at point 727.19 µm and that of silicon peak, 31.58% concentration at point 619.46 µm. This was expected as the reduction temperature for silicon dioxide (around 1700 °C) is higher than that of the iron oxide (750 °C) according to the Ellingham diagram and also due to the higher amounts of iron oxide (28 g) than silica (7 g) in the pellets formed.

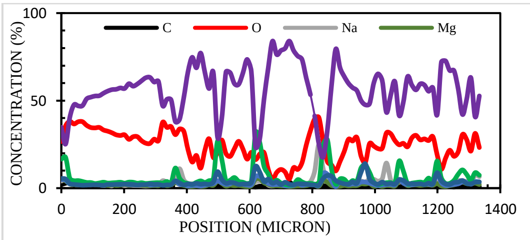

Fig. 14 SEM/EDX Line Spectrum of Reduced Pellet

4 Conclusions and Recommendations

4.1 Conclusions

Ferrosilicon alloy could be produced from the Pudo iron ore blended with end-of-life automotive windshield glass and PET as a reductant in a domestic microwave oven.

End-of-life automotive windshield can be recycled by employing it in ferrosilicon production.

4.2 Recommendations

It is recommended from this research that future studies focus on:

i. Extending the heating duration in the domestic microwave oven beyond 40 minutes.

ii. The cost analysis of using end-of-life plastics as reducing agent at the industrial level.

iii. Further studies could provide valuable insights into the feasibility of utilizing waste materials as feedstock for the steel industry, potentially reducing environmental impact and addressing raw material availability concerns.

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