

# Waste Leather Shoes, a Potential Resource for the Ferrochromium Industry

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## Abstract

Globally, billions of worn out leather shoes and other leather materials are indiscriminately discarded by the Security forces, Mining companies, etc., at landfill sites or in make-shift dug dumps, with no proven avenues for recycling. Leather materials contain Cr<sub>2</sub>O<sub>3</sub> through the process of tanning; landfilling of worn out leather materials and the other processes mentioned above can easily result in the oxidation of insoluble Cr<sub>2</sub>O<sub>3</sub> (Cr<sup>3+</sup>) to soluble CrO<sub>4</sub><sup>2-</sup>(Cr<sup>6+</sup><sub>aq</sub>), a potential carcinogen. In this investigation, pulverised samples of green ash were produced from crumbed samples of waste leather shoes through an ashing process that lasted for 60 min. Samples of the green ash were then characterised by XRF and XRD; this was followed by high temperature (~1600 °C) reduction studies of composite pellets of ash containing blends of high density polyethylene (HDPE) and charred palm kernel shell (PKS) as reductant in a laboratory horizontal tube furnace. From the results, it was observed that waste leather shoes generated approximately 14.0 wt% ash when ashed completely in air and that the generated ash is a rich source of Cr<sub>2</sub>O<sub>3</sub>, with the Cr<sub>2</sub>O<sub>3</sub> concentration ranging from ~47.44 wt% to ~55.44 wt%, depending on the type of leather shoe ashed. Further, the reduction runs revealed that the offgas consists predominantly of CO, with extent of reduction in excess of 99% attained within 40 min. The observed composition range of Cr<sub>2</sub>O<sub>3</sub> in the generated ash makes waste leather shoes potential resource for the Ferrochromium industry.

**Keywords:** Waste leather shoe; Cr<sub>2</sub>O<sub>3</sub>; CrO<sub>4</sub><sup>2-</sup>; Green ash; Extent of Reduction

## 1 Introduction

Globally, billions of worn out leather shoes and other leather materials are indiscriminately discarded by the Security forces, Mining companies, etc., at landfill sites or in make-shift dug dumps, with no proven avenues for recycling. Leather materials contain Cr<sub>2</sub>O<sub>3</sub> through the process of tanning; landfilling of worn out leather materials and the other processes mentioned above can easily result in the oxidation of insoluble Cr<sub>2</sub>O<sub>3</sub> (Cr<sup>3+</sup>) to soluble CrO<sub>4</sub><sup>2-</sup>(Cr<sup>6+</sup><sub>aq</sub>), a potential carcinogen.

Chrome ores vary widely in composition and have conventionally been classified as metallurgical grade, chemical grade and refractory grade according to end use (Olsen, 1997). The pure mineral chromite FeO.Cr<sub>2</sub>O<sub>3</sub> has a composition of 67.9 wt % Cr<sub>2</sub>O<sub>3</sub> (46.4 wt % Cr) and 32.1 wt % FeO (26.0 wt % Fe) with a Cr/Fe ratio of 1.78. However, pure chromite mineral does not occur in nature and it is rare to find an ore containing more than 60.0 wt % Cr<sub>2</sub>O<sub>3</sub> (Olsen, 1997), as illustrated

in typical chromite ore analyses (by XRF) from the most important chromite producing Countries (Table 1).

**Table1 Typical Chromite Ore Analyses from Different Sources (Olsen, 1997)**

	South Africa	Zimbabwe	Turkey	Former USSR	India
Cr <sub>2</sub> O <sub>3</sub>	45	51	48	55	49.5
FeO	26	15	14	12.5	14.5
CaO	0.3	0.2	0.6	2.0	0.2
MgO	11.0	13.0	17.5	16.5	15.5
Al <sub>2</sub> O <sub>3</sub>	15.0	11.8	13.0	8.0	11.0
SiO <sub>2</sub>	3.5	6.0	5.5	4.5	6.5
Others*	0.3	0.2	1.3	1.5	2.8
Cr/Fe Ratio	1.5	3.0	3.0	3.8	3.0

\* Include H<sub>2</sub>O

The smelting reduction process of chromite ores aims at reduction of oxides of iron and chromium with the other constituents of the ore reporting to the slag. For the slag to be sufficiently fluid, it should contain some 26 to 32 wt % SiO<sub>2</sub> (Olsen, 1997).

## 1.1 Carbothermal Reduction of Cr<sub>2</sub>O<sub>3</sub>

The reduction of Cr<sub>2</sub>O<sub>3</sub> by carbon in the absence of iron proceeds according to the reaction equation (1):



The CO pressure is 1 atm at 1240 °C, but the chromium metal produced would react spontaneously with carbon to form one of the carbides Cr<sub>3</sub>C<sub>2</sub>, Cr<sub>7</sub>C<sub>3</sub> or Cr<sub>23</sub>C<sub>6</sub>, with the thermodynamically favoured product being Cr<sub>3</sub>C<sub>2</sub>.



The stability range for the reduction products are shown in Table 2 along with the carbon content of each product.

**Table 2 Stability Range of Carbothermic Reduction Products of Cr<sub>2</sub>O<sub>3</sub>\* (Robiette, 1973)**

Product	Stability Range	wt % C
Cr <sub>3</sub> C <sub>2</sub>	1094-1279	13.3
Cr <sub>7</sub> C <sub>3</sub>	1279-1680	9.0
Cr <sub>23</sub> C <sub>6</sub>	1680-1732	5.7
Cr	≥1732	Solubility of C

\* a<sub>C</sub> = 1; a<sub>Cr2O3</sub> = 1; P<sub>CO</sub> = 1

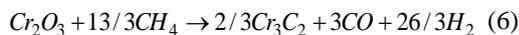
The carbothermal reduction of pure chromium oxide under standard conditions proceeds spontaneously at temperatures above 1113 °C (calculated using data from Knacke *et al.* (1991)) with a standard Gibbs free change given by equation (5):

$$\Delta G^o = 1,078,190 - 777.94T \text{ (J)} \quad (5)$$

The temperature of 1113 °C calculated by data from Knacke *et al.* (1991) agrees and falls within the stability region predicted by Robiette, 1973.

## 1.2 Reduction of Cr<sub>2</sub>O<sub>3</sub> with Methane (CH<sub>4</sub>) Gas

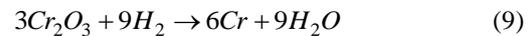
The reduction of chromium oxide with methane occurs through the following reaction:



The standard Gibbs free energy change of this reaction derived using data from Knacke *et al.* (1991) is

$$\Delta G^o = 1,097,519.4 - 989.797T \text{ (J)} \quad (7)$$

Reduction of chromium oxides by methane-hydrogen gas was studied by Read *et al.* (1974) and Qayyum and Reave (1976). Read *et al.* (1974) concluded that the role of methane was to supply carbon, which diffuses into the oxide. In accordance Read *et al.* (1974), the overall reduction reaction (6) is a sum of reactions (8) through (11).



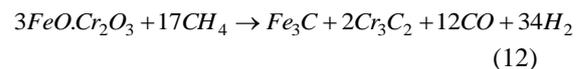
Anacleto and Ostrovski (2004) observed that the optimal conditions for Cr<sub>2</sub>O<sub>3</sub> reduction to the chromium carbide by methane-containing gas include temperature in the range 1100 °C to 1200 °C, methane concentration 15 to 20 vol %, and hydrogen concentration above 20 vol %.

Under non-isothermal conditions, Anacleto and Ostrovski (2004) concluded that chromium oxide reduction by methane-hydrogen mixture started at 770 °C and completed at 910 °C, whereas the carbothermal reduction of chromium oxide under hydrogen started at 860 °C and completed at around 1040 °C. Further, they observed that the rate of reduction by CH<sub>4</sub>-H<sub>2</sub>-Ar gas was much faster than that by graphite, which they attributed to high carbon activity in the reducing gas.

The presence of CH<sub>4</sub> and H<sub>2</sub> is therefore beneficial to the reduction of Cr<sub>2</sub>O<sub>3</sub> to the metal or the carbide.

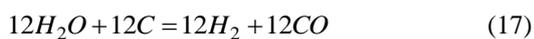
## 1.3 Reduction of Chromite FeO.Cr<sub>2</sub>O<sub>3</sub> with Methane (CH<sub>4</sub>) Gas

Qayyum and Reave (1976) proposed a reduction mechanism, suggesting that under conditions when methane is stable (low CH<sub>4</sub>/H<sub>2</sub> ratio) chromite is reduced by reaction (12), which is similar to reaction (6):



The following reactions were suggested by Qayyum and Reave (1976) at high temperatures and CH<sub>4</sub>/H<sub>2</sub> ratios, when methane is unstable:





## 2 Materials and Methods Used

### 2.1 Ashing of Waste Leather Shoes

Samples of waste leather shoes were collected from a mining company in Tarkwa and a discarded pool from the Minerals Engineering Department of the University of Mines and Technology, Tarkwa, Ghana.



**Fig. 1 Samples of Waste Leather Shoes used for the Ashing Process**

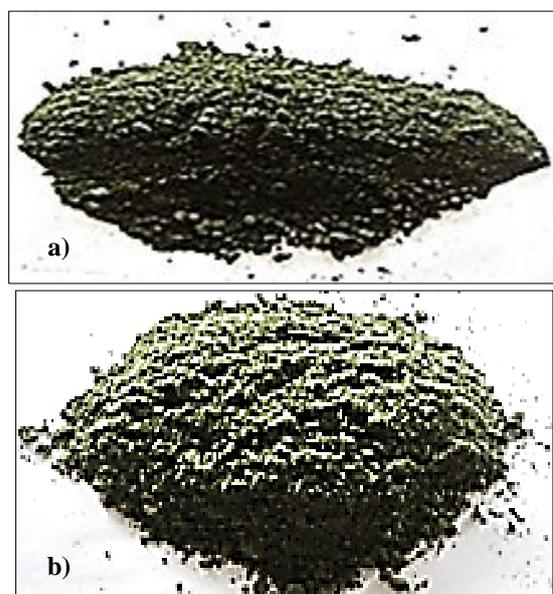
The shoes were washed to remove any associated debris and dried in air for four hours. The dry shoes were dismantled by removing the soles and any accompanying metal lockets for lacing. The leather parts were then cut into crumbs and weighed using a laboratory scale (Metler Toledo). About 500 g of the crumbs were loaded into an airtight stainless steel container and were charred at  $\sim 500$  °C for 30 min. Charred samples were then placed in fireclay crucibles that were carefully placed on charred palm kernel shell fired heating source, as illustrated in Fig. 2. Complete ashing of the chars was done in excess supply of air for 30 min.

The ashed leather shoes were allowed to cool after which they were pulverised and sieved to 75% passing 125  $\mu\text{m}$ .



**Fig. 2 Firing of Crucibles containing Samples**

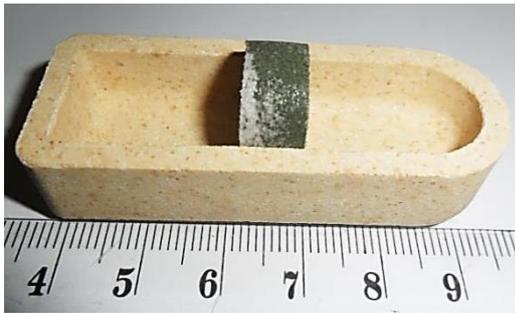
The amount of ash (wt % ash) obtained in each experimental run was calculated. Samples of ash obtained from two experimental runs are shown in Fig 3. As indicated in Fig 3 the ash samples are light to dark green in appearance, suggesting that  $\text{Cr}_2\text{O}_3$  may be present. Representative samples were submitted for analyses by XRF and XRD analyses.



**Fig. 3 Samples of Green Ash obtained from heating waste leather shoes**

### 2.2 Preparation of Ash-Carbonaceous Material Composite Pellet

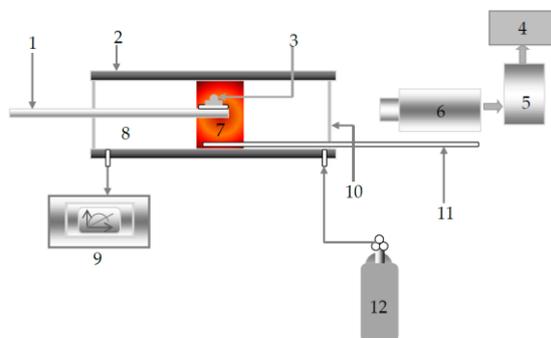
About 3.30 g of leather waste ash ( $\sim 52.85$  wt%  $\text{Cr}_2\text{O}_3$ ) was subsequently mixed with PKS-HDPE (at PKS:HDPE ratio of 3:7) and compacted in a die to produce cylindrical pellets ( $\sim 1.2$  mm thick and 15 mm diameter) by applying a load of 7.5 tonnes for 2 minutes in a hydraulic press. One composite pellet at a time was placed in a LECO crucible as shown in Fig. 4, ready for reduction in a horizontal tube furnace.



**Fig. 4 Ash-Carbonaceous Material Composite Pellet**

### 2.3 Reduction of Ash-Carbonaceous Material Composite Pellet in a Horizontal Tube Furnace

A schematic arrangement of the horizontal tube furnace, equipped with an infrared gas analyser is shown in Fig. 5



**Fig. 5 Schematic of the horizontal tube furnace and IR gas analyser system (1 Sample Rod; 2 Alumina tube; 3 Reaction mixture; 4 PC; 5 DVD; 6 CCD Camera; 7 Hot Zone; 8**

Cold Zone; 9 Gas analyser; 10 Quartz window; 11 Thermocouple; 12 Argon gas)

The LECO crucible-cylindrical pellet sample assembly was carefully pushed into the ‘cold’ zone (Temperature ~ 300 °C) of the furnace, which was purged continuously with argon (Air Liquid Alphagaz HP 99.995% purity; <5 ppm O<sub>2</sub>) at a flow rate 1.0 L min<sup>-1</sup> to ensure inert conditions.

After the furnace had attained the desired hot zone temperature (1600 °C) the sample assembly was pushed further into the reaction hot zone and offgas (CO and CO<sub>2</sub>) measurement by a continuous infrared gas analyser commenced and lasted for 40 min.

## 3 Results and Discussion

### 3.1 Amount of Ash Produced from Waste Leather Shoes

The weight of ash produced along with the wt% ash for each of the five experimental runs conducted in this investigation is shown in Table 3. It is apparent from Table 3 that waste leather shoes could generate approximately 14 wt% ash.

### 3.2 Characterisation of Ash by XRD

Result of XRD analysis of an ashed sample of the waste leather shoes is shown in Fig. 6. The diffractogram consists predominantly of sharp peaks of Cr<sub>2</sub>O<sub>3</sub> along with some CaCO<sub>3</sub> and SiO<sub>2</sub>. The ash sample is therefore a rich source of Cr<sub>2</sub>O<sub>3</sub>, evidenced by the large number of sharp distinct peaks of the oxide.

**Table 3 Experimental Runs showing the Content of Green Ash Produced from the Ashing Process**

Experiment	Weight of leather (g)	Ash Weight (g)	Percent Ash (%)
1	501.00	70.74	14.12
2	500.38	69.48	13.89
3	503.22	69.99	13.91
4	500.35	69.08	13.81
5	503.39	70.46	14.00
<b>Average</b>	501.67	69.95	13.95

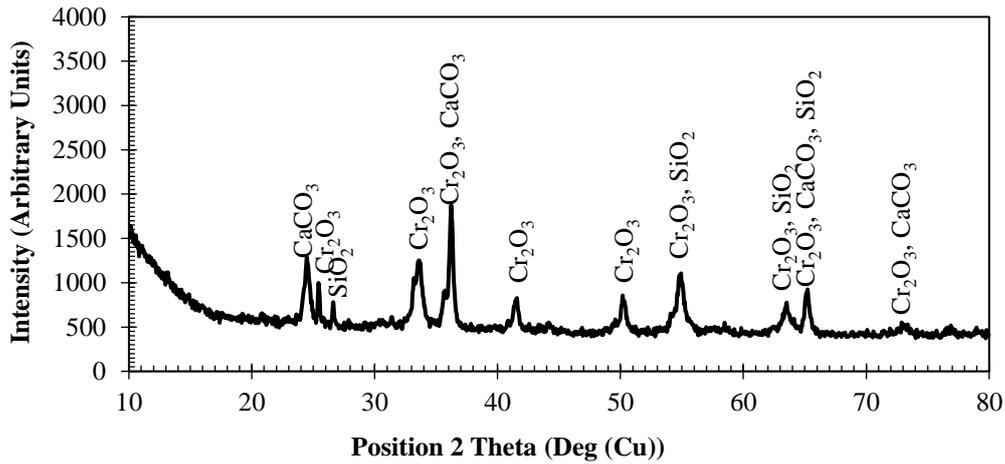


Fig. 6 X-Ray Diffractogram of Ashed Sample of Waste Leather Shoes

### 3.3 Composition of Ash by XRF

The  $\text{Cr}_2\text{O}_3$  content of ores commercially used for ferrochrome production generally varies from 42 to 55 wt % (Olsen, 1997). The compositions by XRF of the maximum and minimum values of  $\text{Cr}_2\text{O}_3$  in the leather waste ash obtained from the heating process are shown in Table 4, which indicates that the compositions of  $\text{Cr}_2\text{O}_3$  in waste leather ash (Black leather: 55.54 wt %; Brown leather: 47.44 wt %) are within the range of  $\text{Cr}_2\text{O}_3$  commercially utilised for the ferrochromium process. Waste leather ash is therefore a potential source of  $\text{Cr}_2\text{O}_3$  for the ferrochromium industry.

Table 4 XRF Analysis of Ashed Samples of Waste Leather Shoe

COMPONENT	BLACK	BROWN
%MgO	3.871	1.752
%Al <sub>2</sub> O <sub>3</sub>	11.724	3.302
%SiO <sub>2</sub>	17.355	3.274
%P <sub>2</sub> O <sub>5</sub>	2.926	20.606
%SO <sub>3</sub>	8.497	8.061
%K <sub>2</sub> O	0.456	0
%CaO	2.049	0.432
%TiO <sub>2</sub>	0.081	0.169
%MnO	0.473	0.454
%Fe <sub>2</sub> O <sub>3</sub>	0.926	0.374
%V <sub>2</sub> O <sub>5</sub>	0	0.045
%Cr <sub>2</sub> O <sub>3</sub>	51.539	47.442
%Rb <sub>2</sub> O	0.003	0
%SrO	0.012	0
%Y <sub>2</sub> O <sub>3</sub>	0	0

%ZrO <sub>2</sub>	0.089	14.079
%WO <sub>3</sub>	0	0.012
SUM	100	100

### 3.4 Reducibility of Cr<sub>2</sub>O<sub>3</sub> from Waste Ash Leather

The production of ferrochromium requires the simultaneous reduction of both  $\text{Fe}_2\text{O}_3$  and  $\text{Cr}_2\text{O}_3$  by C, H<sub>2</sub> or a reductant containing both C and H. The reductant used in this investigation (blend of PKS and HDPE) acted as source of C, H<sub>2</sub>, CO and CH<sub>4</sub> (Dankwah *et al.* 2011).

#### 3.4.1 Off Gas Analysis

The concentration (vol %) of CO and CO<sub>2</sub> in the offgas during the reduction process is shown in Fig. 7.

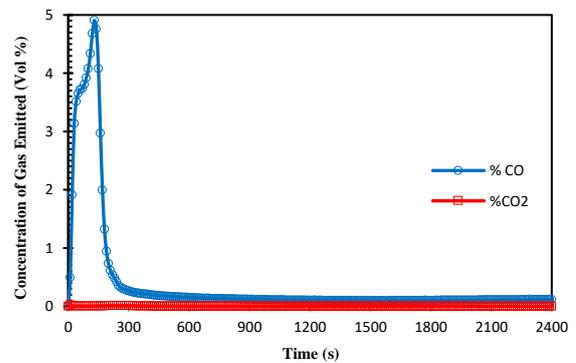
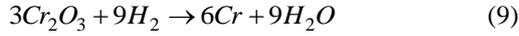
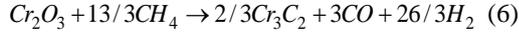
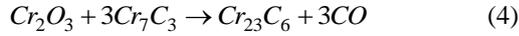
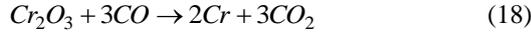


Fig. 7 Concentration of Offgas Evolved during Reduction of  $\text{Cr}_2\text{O}_3$  from Ashed Leather Waste at 1600 °C

The off gas consists predominantly of CO, with virtually no CO<sub>2</sub> present. This is an indication of a direct reduction of Cr<sub>2</sub>O<sub>3</sub> by C and/or CH<sub>4</sub>, which is consistent with the reactions 1-4, 6, and 8-11:



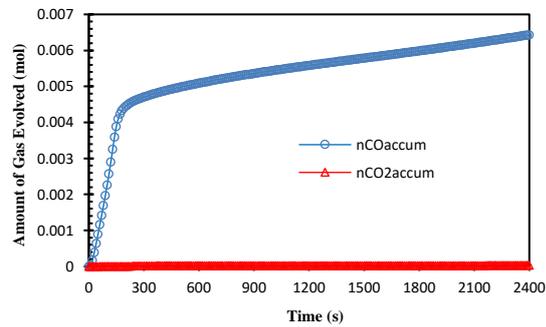
Of course, the absence of CO<sub>2</sub> in the offgas could also be understood from the fact that reaction (1) is actually the sum of the two reactions:



and the Boudouard reaction,



The accumulated amounts of CO and CO<sub>2</sub> evolved over the period of 40 min are shown in Fig. 8, and as expected the CO<sub>2</sub> plot lays perpetually on the horizontal axes, suggesting no CO<sub>2</sub> activity, as elucidated above. The rate and extent of reduction are therefore expected to rely heavily on the concentration of CO in the offgas.



**Fig. 8 Amount of Offgas Evolved during Reduction of Cr<sub>2</sub>O<sub>3</sub> from Ashed Leather Waste at 1600 °C**

### 3.4.2 Rate and Extent of Reduction from Off Gas Data

The off-gas data was generated continuously. Accordingly, the overall rate of gas generation could be described by a continuous function  $R_o$

(mol s<sup>-1</sup>), from which the extent of reduction can be estimated by integration (Dankwah *et al.*, 2011). Consequently, the extent of reduction ( $f$ ) after time  $t$  can be calculated from equation (20) (Dankwah *et al.*, 2011):

$$f = \frac{1}{N_o} \int_0^t R_o \cdot dt \quad (20)$$

where  $N_o$  is the total amount of reducible oxygen in converting the chromium oxide from (Cr<sub>2</sub>O<sub>3</sub>) to Cr.

$R_o$  can be estimated from the individual molar fluxes,  $J_i$ , (mol cm<sup>-2</sup> s<sup>-1</sup>) as shown in equation (21), assuming that the gases behave ideally (room temperature = 20 °C) (Dankwah *et al.*, 2011):

$$J_i = \frac{273}{293} \times F_{Ar} \times \frac{\%i}{22.4 \times \%Ar} \cdot \frac{1}{A} \quad (21)$$

From a mass balance for oxygen (based on complete transformation of (Cr<sub>2</sub>O<sub>3</sub>) to Cr), the flux of O is calculated from equation 22 (Dankwah *et al.*, 2011).

$$-J_o = J_{CO} + 2 \cdot J_{CO_2} + J_{H_2O} \quad (22)$$

The rate of removal of O from Cr<sub>2</sub>O<sub>3</sub> is thus given by equations 23 and 24 (Dankwah *et al.*, 2011):

$$R_o = -\frac{d(n_o)_t}{dt} = -AJ_o \quad (23)$$

$$R_o = 0.0416 \times F_{Ar} \times \left( \frac{2 \times \%CO_2}{\%Ar} + \frac{\%CO}{\%Ar} + \frac{\%H_2O}{\%Ar} \right) \quad (24)$$

Where:  $J_i$ ,  $n_o$ ,  $F_{Ar}$  and  $A$  are the molar flux (mol of species  $i$ /cm<sup>2</sup>.sec), amount of O (mol) at time  $t$ , Ar flow rate ( $F_{Ar} = 1.0$  L/min) and reaction area (cm<sup>2</sup>) respectively.

Equation 24 can usually be approximated by equation 25 at temperatures above 700 °C and C/O > 1.0, where %H<sub>2</sub>O ~ 0 (Dankwah *et al.*, (2011); Rezan *et al.*, 2012); Dankwah *et al.*, (2017).

$$R_o \approx 0.0416 \times F_{Ar} \times \left( \frac{2 \times \%CO_2}{\%Ar} + \frac{\%CO}{\%Ar} \right) \quad (25)$$

Based on the amount of removable oxygen from the ore, the extent of reduction,  $f$  can be expressed by equations 26 and 27 (Dankwah *et al.*, 2017).

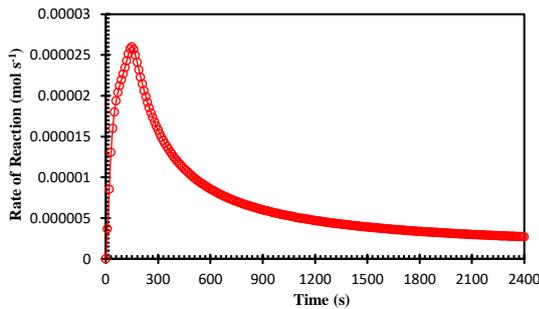
$$f = \frac{\text{amount of oxygen removed}}{\text{total amount of removable oxygen}} \quad (26)$$

$$f = \frac{n_{CO} + 2n_{CO_2} + 2n_{H_2O}}{N_O} \quad (27)$$

Again, under the conditions of the experiment,  $n_{H_2O} \sim 0$ . Consequently, the extent of reduction can be approximated by equation 28 (Dankwah *et al.*, 2017).

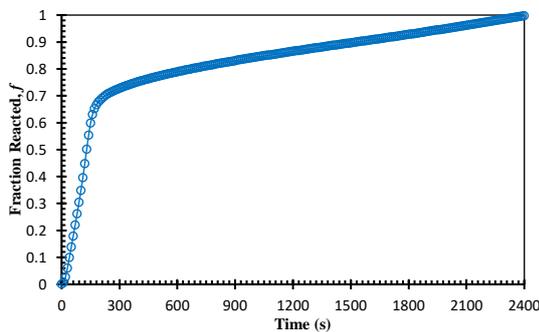
$$f \approx \frac{n_{CO} + 2n_{CO_2}}{N_O} \quad (28)$$

These two plots are shown in Figs 9 and 10.



**Fig. 9 Rate of Reduction of  $Cr_2O_3$  from Ashed Leather Waste at 1600 °C**

As shown in Fig. 9, the reaction proceeds rapidly and attains a maximum rate of reduction after only 150 s and slows down afterwards. The extent of reduction, as expressed in equation (28), is plotted in Fig. 10. The observed extent of reduction after 40 min is 99.68%, and it suggests that  $Cr_2O_3$  from waste leather ash can be successfully reduced by blends of PKS and HDPE.

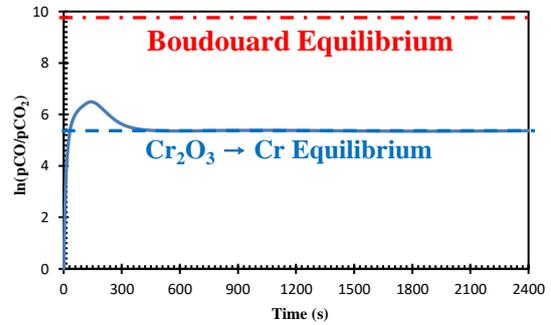


**Fig. 10 Plot of Fraction of  $Cr_2O_3$  Reacted from Ashed Leather Waste at 1600 °C**

### 3.4.3 Reduction kinetics and the influence of the Boudouard reaction

Fig. 11 shows a plot of  $\ln(P_{CO}/P_{CO_2})$  as a function of time for the reduction of  $Cr_2O_3$  in waste leather ash by PKS-HDPE blend at 1600 °C. The Boudouard and  $Cr_2O_3$ -Cr equilibrium lines at 1600 °C are shown in the plot. It is seen from Fig. 11

that in the first 360 s,  $\ln(P_{CO}/P_{CO_2})$  shows a complete shift away from the  $Cr_2O_3$  equilibrium line as well as from the Boudouard equilibrium line, suggesting that both reactions (Boudouard and  $Cr_2O_3$  transformation to Cr) display a very strong influence on the reduction process. After this period,  $\ln(P_{CO}/P_{CO_2})$  approaches the  $Cr_2O_3 \rightarrow Cr$  equilibrium line completely up to the end of the reduction process (360-2400 s). It means that the Boudouard reaction exerts a very strong influence over the entire duration of the reduction process.



**Fig. 11 Plot of  $\ln(P_{CO}/P_{CO_2})$  as a Function of Time**

## Conclusions and Recommendations

This investigation produced green ashes from waste leather shoes and assessed the potential of such ashes as chemical feedstock for the ferrochromium industry. The ash was characterised by XRD and XRF analyses followed by reduction studies of composite pellets of the ash containing blends of PKS-HDPE as reductant.

Important findings from the investigation are:

- i. Waste leather shoes can be heated under controlled environments to produce approximately 14 wt% green ash suitable for use as chemical feedstock for the ferrochromium industry;
- ii. Characterisation of the ash by XRD and XRF revealed distinct peaks of  $Cr_2O_3$  at concentration ranging from 47.44 – 55.44 wt%  $Cr_2O_3$ ;
- iii. Reduction studies on composite pellets of the ash (containing blends of PKS-HDPE) showed an extent of reduction of over 99%, after 40 min of heating in a horizontal tube furnace.

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