# Calcination Behaviour of Nsuta Rhodochrosite Ore in the Presence and Absence of End-of-Life High Density Polyethylene\*

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# Abstract

This research investigated the calcination behaviour of the Nsuta Rhodochrosite (MnCO<sub>3</sub>) in the presence and absence of end-of-life high density polyethylene (HDPE) using a custom-made palm kernel shell fired furnace. Samples of pulverised Nsuta rhodochrosite were heated rapidly for 30, 40, 50 and 60 minutes, coupled with temperature measurements to determine the maximum temperature attained in the fireclay crucible. The procedure at 60 min was repeated using three blends of rhodochrosite samples containing different masses of HDPE (30 g, 40 g and 50 g) and heated for an hour. For gas analyses studies during calcination, cylindrical compacts of rhodochrosite ore in a LECO<sup>TM</sup> crucible were heated rapidly with and without high density polyethylene (HDPE at C/O ratio = 1.0, 1.5, and 2.0) in a horizontal tube furnace for 600 s at 1150 °C under high purity argon gas and the off gas was continuously analysed for CH4, CO and CO2 using an online infrared gas analyser. The content of H<sub>2</sub> in the off gas was detected using a GC3 gas chromatographic analyser equipped with a thermal conductivity detector. The Nsuta rhodochrosite ore was found to consist of a mixture of manganese II carbonate (MnCO<sub>3</sub>), silica (SiO<sub>2</sub>), mixed transition metal carbonate of the form Ca(Mn, Mg)(CO<sub>3</sub>)<sub>2</sub> and mixed metal silicate of the form Ca<sub>0.6</sub>Mg<sub>1.94</sub>Si<sub>2</sub>O<sub>6</sub>. Calcination results indicated visible colour changes (from grey to dark brown), along with significant changes in the mass before and after calcination. In the absence and presence of the polymer, measured temperatures in the crucible ranged from 1001 °C to 1366 °C and 1361 °C to 1369 °C, respectively. Analyses by XRF showed marginal increase in the content of Mn in the calcined ore with HDPE addition. Gas analyses indicate that blending the carbonate with HDPE before heating results in significant decrease in the amount of CO<sub>2</sub> emitted.

Keywords: Land Tenure Security, Registration, Spatial Data, Attribute Data

# **1** Introduction

The Nsuta Manganese Mine is located on latitude  $5^{\circ}16'59''$  North and longitude  $1^{\circ}58'0''$  West in the Western Region of Ghana. Although the mine has produced oxide ore for a number of years, such production is no longer sustainable due to the depletion of the oxide ore (Ali and Amankwah, 2013). As such, production is now fully on the manganese carbonate (averaging 34.16% Mn (Kesse, 1985)) which is close to metallurgical grade of 38 - 55% Mn (Ali and Amankwah, 2013). The conversion of manganese carbonate to oxide is usually by calcination (Amankwah and Pickles 2005; Dankwah 2014) and may follow the following reactions, depending on the furnace environment:

$$MnCO_3 = MnO + CO_2 \tag{1}$$

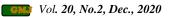
$$3MnCO_3 = Mn_3O_4 + 2CO_2 + CO$$
(2)

From equations (1) and (2) the equilibrium constants ( $k_p$ ) for the calcination process (assuming pure solid species) are related to the partial pressures of gaseous species (CO and CO<sub>2</sub>, i.e. pCO and pCO<sub>2</sub>) as follows:  $k_{p_1} = pCO_2$  (3)

$$k_{p_2} = (pCO_2)^2 . pCO \tag{4}$$

In another development, a study conducted by O'Shaughnessy et al. (2004) concluded that Nsuta manganese carbonate is still acceptable in its nature for manganese ferroallov production. The thermal decomposition of metal carbonates and or hydroxides have been investigated before (Borgwardt, 1985; Chen et al., 2009; Kim and Kwon, 1998; Li et al., 2005; Samtani et al., 2002; Telfer et al., 2000; Wang et al., 2007; Yamaguchi et al., 1980). The effect of  $CO_2$  partial pressure on the thermal decomposition kinetics of metal carbonates such as CdCO<sub>3</sub>, MnCO<sub>3</sub>, PbCO<sub>3</sub> and ZnCO<sub>3</sub> were investigated by Criado et al., 1998; Criado et al., 1987; Criado et al., 1982; Yamada et al., 2009; Yamaguchi et al., 1980). In all these investigations no polymers were added and, accordingly, there was no investigation on the effect of gases from the thermal decomposition of polymers. The addition of a polymer to metal reduction processes is thought to have an attenuating effect on especially the  $CO_2$  that is evolved into the atmosphere (Dankwah et al., 2011; Dankwah et al., 2012; Dankwah et al., 2013).

Dankwah (2014) investigated the effect of HDPE and linear low density polyethylene (LLDPE) on  $CO_2$  emissions during the calcination of reagent



grade MnCO<sub>3</sub>. Using a horizontal tube furnace at various C/O ratios, they observed that the presence of a polymer has a significant attenuating effect on  $CO_2$  emissions. It was further reported that the observed attenuation of  $CO_2$  emissions was accompanied by a simultaneous production of syngas (CO + H<sub>2</sub>) that could be recovered as a beneficial by-product

The aim of this investigation is therefore to establish a fundamental understanding of the calcination behaviour of the Nsuta carbonate ore in the absence and presence of waste plastics.

# 2 Resources and Methods Used

### 2.1 Materials

Pulverised samples (-75  $\mu$ m) of naturally occurring rhodochrosite ore from Nsuta (Fig. 1), Ghana was used for this experimental investigation as a source of MnCO<sub>3</sub>, whilst samples of pure water sachets (PWS) collected from the university of Mines and Technology, Tarkwa were used as the source of reductant.



Fig. 1 Pulverised Nsuta Rhodochrosite

Samples of HDPE were subjected to a meltingquenching process (Fig. 2) to render them brittle, followed by milling in a laboratory ball mill to obtain pulverised carbonaceous reductant as shown in Fig. 3.



Fig. 2 Formation of Embrittled HDPE after a Melting-Quenching Process



Fig. 3 Samples of Pulverised Carbonaceous Material obtained from Heat-treated HDPE

# **2.2 Experimental Procedure**

#### 2.2.1 Calcination of Nsuta Rhodochrosite Ore

About 50 g of pulverised Nsuta Rhodochrosite ore was placed in a fireclay crucible and calcined for 40 minutes in a charred palm kernel shell fired furnace (Fig. 4). The calcined sample was quickly withdrawn from the furnace and the temperature was recorded using a K-type thermocouple; the calcined sample was then allowed to cool and its mass measured and recorded using an electronic balance. This procedure was repeated three times after which the calcined samples were crushed, ground and sieved to  $-75 \,\mu$ m.



Fig. 3 Firing of Crucibles containing Samples

2.2.2 Calcination of Nsuta Rhodochrosite Ore in Horizontal Tube Furnace (Gas Measurements)

About 2.0 g of the ore was compacted in a die to produce cylindrical pellets (~1.2 mm thick and 15 mm diameter) (Fig. 5), by applying a load of 7.5 tonnes for 2 minutes in a hydraulic press. Each cylindrical pellet was placed in LECO<sup>TM</sup> crucible and the sample assembly was placed in the cold zone of an electrically heated horizontal tube furnace (Fig 6), which was purged continuously with argon (of 99.995% purity and flow rate 1.0 L/min) to ensure inert conditions.

The content of H<sub>2</sub> in the offgas was detected by a gas chromatographic (GC3) analyser equipped with a thermal conductivity detector (TCD). The TCD detector responds to the difference between the thermal conductivity of the carrier gas and the analyte peak; the greater the difference in thermal conductivity the better the sensitivity. Argon ( $\kappa$  =39) was used as the carrier gas as the target analyte peak was hydrogen ( $\kappa$  = 419). The other gases, CO ( $\kappa$  =53), CO<sub>2</sub> ( $\kappa$  =34) and CH<sub>4</sub> ( $\kappa$  =73) were measured quantitatively using an IR-gas analyser. Peak determination of these gases on the chromatogram was not satisfactory since their thermal conductivities were close to the carrier gas (argon).



Fig. 5 Cylindrical Pellet of Nsuta Rhodochrosite Ore in a LECO<sup>TM</sup> Crucible

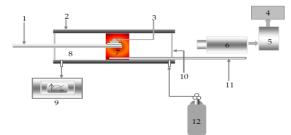


Fig. 6 Schematic of the horizontal tube furnace and IR gas analyser system (1 Sample Rod; 2 Alumina tube; 3 Reaction mixture; 4 PC; 5 DVD; 6 CCD Camera; 7 Hot Zone; 8 Cold Zone; 9 Gas analyser; 10 Quartz window; 11 Thermocouple; 12 Argon gas)

After the furnace had attained the desired hot zone temperature (1150 °C), the sample was pushed in the reaction hot zone and gases (CH<sub>4</sub>, CO and CO<sub>2</sub>) were monitored for 900 s by an infrared gas analyser attached to the system; the results were recorded in a data-logging computer.

#### **3** Results and Discussion

#### 3.1 Nature of Nsuta Rhodochrosite Ore

3.1.1 Results of XRD, XRF, SEM/EDS Analyses of Nsuta Rhodochrosite

Fig. 7 shows the X-Ray diffraction patterns of pulverised samples of as-received Nsuta

Rhodochrosite ore. Well defined peaks of MnCO<sub>3</sub> can be seen along with peaks of SiO<sub>2</sub>, Ca(Mn, Mg)(CO<sub>3</sub>)<sub>2</sub> and Ca<sub>0.6</sub>Mg<sub>1.94</sub>Si<sub>2</sub>O<sub>6</sub>. The XRF results are shown in Table 1. It is clear from Table 1 that MnO is the major component and as would be expected SiO<sub>2</sub>, CaO and MgO are also present. The silica content of this ore is above 10 wt%, making it suitable for the production of silicomanganese, unless the ore can be upgraded to decrease the content of SiO<sub>2</sub>. A desirable future of this ore is the phosphorous content which is below 0.1 wt%. The content of phosphorous in a manganese ore is critical and must typically be kept below 0.1 wt% as it cannot be removed during processing and accordingly end up in the final product to cause problems.

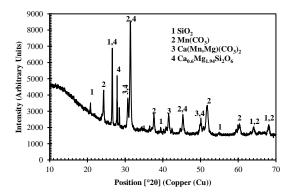
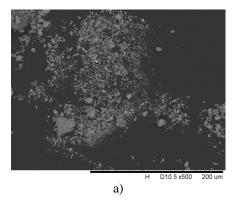


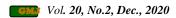
Fig. 7 X-Ray Diffractogram (XRD) of Nsuta Rhodochrosite Ore utilised for the Investigation

Table 1 XRF of Pulverised Rhodochrosite Ore.	
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Component	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	MgO	MnO	TiO <sub>2</sub>
wt %	15.02	2.20	4.98	39.30	0.03
Component	Na <sub>2</sub> O	$P_2O_5$	$V_2O_5$	CaO	NiO
wt %	0.66	0.08	0.03	5.91	0.03
Component	Fe <sub>2</sub> O <sub>3</sub>	$K_2O$	$SO_3$	ZnO	LOI
wt %	1.24	0.28	0.44	0.01	29.80

Results of the SEM of the ore at two different magnifications ( $\times$ 500 and  $\times$ 2.0k) are shown in Fig. 8a and 8b, respectively.





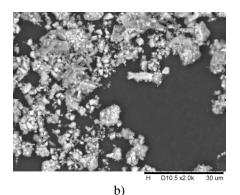
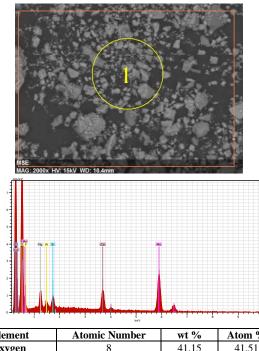


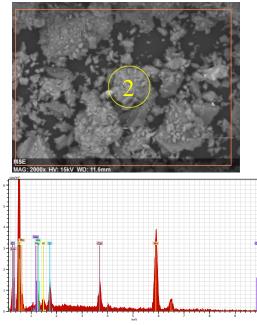
Fig. 8 Scanning Electron Micrograph of Nsuta Rhodochrosite Ore utilised for the Investigation: a) ×500 b) ×2.0k

The results of SEM/EDS analyses of the Nsuta Rhodochrosite are shown in Figs 9 and 10, for Regions 1 and 2, respectively. The major elements are oxygen, manganese and carbon with minor amounts of calcium, silicon, magnesium, aluminium, fluorine and arsenic. This cluster of elements suggest the presence of silica, and carbonates/silicates of manganese, calcium and manganese that were observed in the XRD in Fig. 7.



Element	Atomic Number	wt %	Atom %
Oxygen	8	41.15	41.51
Carbon	6	37.63	50.56
Manganese	25	15.09	4.43
Calcium	20	2.72	1.10
Fluorine	9	1.22	1.04
Magnesium	12	1.08	0.72
Silicon	14	0.75	0.43
Aluminium	13	0.35	0.21
,	TOTAL	100	100





Element	Atomic Number	wt %	Atom %		
Oxygen	8	40.71	56.48		
Manganese	25	36.89	14.90		
Carbon	6	11.56	21.35		
Calcium	20	4.14	2.29		
Silicon	14	2.50	1.98		
Magnesium	12	1.92	1.75		
Arsenic	33	1.10	0.33		
Aluminium	13	1.07	0.88		
Germanium	32	0.12	0.04		
T	TOTAL	100	100		
E' 10 CEM/EDS & Number Divide the start of the					

Fig. 10 SEM/EDS of Nsuta Rhodochrosite Ore utilised for the Investigation (Region 2)

#### 3.2 Results of Calcination of Rhodochrosite Ore

The results of temperature and weight loss measurements are shown in Tables 2 and 3 for calcination in the absence and presence of HDPE, respectively.

## Table 2 Calcination of Raw Nsuta

Rhod	ochrosi	te

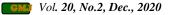
ID	Time (min)	$W_{1}\left(g ight)$	$W_{2}\left(g ight)$	ΔW (%)	T <sub>max</sub> (°C)
А	30	200	167.6	16.2	1001.2
В	40	200	166.7	16.7	1339.4
С	50	200	160.3	19.9	1360.1
D	60	200	146.9	26.6	1366.3

**Table 3 Calcination of Blended Nsuta** 

**Rhodochrosite Ore** 

WORE (g)	W <sub>HDPE</sub> (g)	WTOTAL (g)	WAFTER (g)	Time (min)	ΔW (%)	T <sub>max</sub> (°C)
200	30	230	145.4	60	36.78	1361
200	40	240	144.9	60	37.00	1367
200	50	250	151.2	60	34.26	1369

In the absence of the polymer, the % weight loss of the ore increased progressively from 16.2% to 26.6% after calcination for 30 min and 60 min, respectively. Within this period the temperature in



the fireclay crucible showed a corresponding increase from about 1001  $^\circ C$  to over 1366  $^\circ C.$ 

The calcined ore changes colour from grey to dark brown as shown in Fig. 11.



a) Original Ore



b) Calcined Sample after 30 min



c) Calcined Sample after 40 min



d) Calcined Sample after 50 min



#### e) Calcined Sample after 60 min

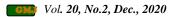
# Fig. 11 Appearance of the Ore at various stages of Calcination

In the presence of the polymer, the %weight loss of the blend increased from 36.78% to 37.00% for 30.0 g and 40.0 g HDPE addition. A possible explanation to this observation could be the extra heat supplied to the system by the thermal decomposition of the polymer, evidenced by the increase in temperature recorded in the crucible. Going beyond 40.0 g HDPE addition resulted in a decline in the weight loss. Examination of the calcined mass showed that the entire polymer escaped from the blend after calcination. In Table 4, a new column is introduced showing % weight loss values based on the raw mass of the ore (200.0 g).

Table 4 % Weight Loss against Amount of HDPE added

W <sub>HDPE</sub> (g)	ΔW* (%)	T <sub>max</sub> (°C)
30	27.3	1361
40	27.55	1367
50	24.4	1369

The temperature and weight loss profiles are illustrated in Fig. 12.



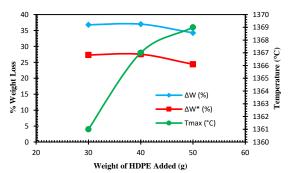


Fig. 12 Weight Loss and Temperature Profiles for Calcination of Compacts containing HDPE

Compared to the raw ore for the same calcination period of 60 min, HDPE addition resulted in a marginal increase in the % weight loss from 26.6% for the raw ore to 27.3% and 27.55% for 30.0 g and 40.0 g addition, respectively. Beyond this amount, it shows a decline. For a Carbonate ore of 200.0 g the amount of HDPE added must not exceed 40.0 g for effective calcination. Within this period the recorded maximum temperature increased marginally from 1361 °C to 1369 °C.

The appearance of the calcined ore in the presence of various amounts of the polymer is illustrated in Fig. 13. Obviously, each of them appears brown and there is no clear distinction between the observed colours.



a) Calcined Blend (30 g PWS addition)



b) Calcined Blend (40 g PWS)



c) Calcined Blend (50 g PWS)

#### Fig. 13 Appearance of the Ore + HDPE Blend for various HDPE Additions for Calcination at 60 min

Results of XRF analyses of the calcined ores are shown in Tables 5 and 6 for calcination in the absence and presence of the polymer after 60 min of heating. Calcination of the ore increases the Mn content of the ore from 39.30 wt% MnO to 45.96 wt% MnO and 47.65 wt% MnO in the absence and presence of the polymer, respectively. Although calcination of the Nsuta rhodochrosite results in an improvement in the content of the manganese content, addition of the polymer results in only a marginal improvement, consistent with the observed marginal increase in the temperature recorded in the crucible.

#### Table 5 XRF of Pulverised Rhodochrosite Ore after Calcination (Raw)

		emation	(1140)			
Component	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	MgO	MnO	TiO <sub>2</sub>	
wt %	19.20	8.41	4.52	45.96	0.22	
Component	SO <sub>3</sub>	$P_2O_5$	$V_2O_5$	CaO	SrO	
wt %	2.67	0.08	0.02	13.63	0.06	
Component	Fe <sub>2</sub> O <sub>3</sub>	$K_2O$	Other oxides*			
wt %	4.09	1.06	0.08			

\*Other oxides: Cr<sub>2</sub>O<sub>3</sub>, NiO, BaO, HfO<sub>2</sub>, PbO, ZnO, CuO, WO<sub>3</sub>

 Table 6 XRF of Pulverised Rhodochrosite Ore after Calcination (with HDPE)

Component	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	MgO	MnO	TiO <sub>2</sub>	
wt %	19.02	7.53	3.40	47.65	0.22	
Component	$SO_3$	$P_2O_5$	$V_2O_5$	CaO	SrO	
wt %	3.15	0.09	0.02	13.58	0.06	
Component	Fe <sub>2</sub> O <sub>3</sub>	$K_2O$	Other oxides*			
wt %	4.15	1.04	0.09			

\*Other oxides: Cr<sub>2</sub>O<sub>3</sub>, NiO, BaO, HfO<sub>2</sub>, PbO, ZnO, CuO, WO<sub>3</sub>

#### 3.3 Gas Measurement during Calcination of Nsuta Carbonate Ore

The composition of the offgas (CO, CO<sub>2</sub> and CH<sub>4</sub>) from the horizontal tube furnace as measured by a continuous infrared gas analyser during calcination of each of the compacts is described in this section. Fig. 14 shows the gas emission behaviour of the polymer-free compact. In the absence of HDPE the offgas from the calcination of Nsuta rhodochrosite consists predominantly of CO<sub>2</sub> (maxima at 12.23 vol %) and some CO (maxima at 2.71 vol %), with virtually no traces of CH<sub>4</sub>.

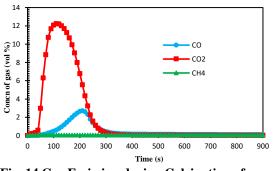


Fig. 14 Gas Emission during Calcination of Nsuta Rhodochrosite Ore in Air.

Fig. 15 represents the case where the ore was mixed with pulverised HDPE at C/O of 1.0. From Fig. 15 it is apparent that  $CO_2$  (maxima at 7.14 vol %) is no longer the predominant gas in the composition of the offgas, as CO (maxima at 11.18 vol %) now takes over and the content of  $CO_2$  declines. Significant amounts of CH<sub>4</sub> (maxima at 2.27 vol %) are also observed, probably from the thermal decomposition of the polymer.

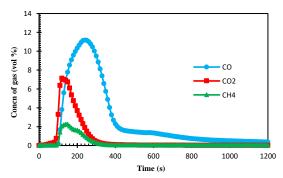


Fig. 15 Gas Generation Behaviour during the Calcination of MnCO<sub>3</sub>-HDPE compact (C/O = 1.0)

From Fig. 16, increasing the C/O ratio from 1.0 to 1.5 does not appear to change the composition of the offgas from the scenario observed for a C/O ratio of 1.0. The observed maxima change from 7.14, 11.18 and 2.27 vol% to 5.71, 10.14 and 2.80 vol% for CO<sub>2</sub>, CO and CH<sub>4</sub>, respectively.

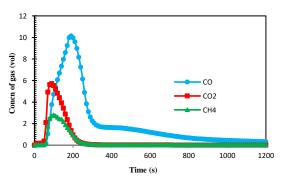


Fig. 16 Gas Generation Behaviour during the Calcination of MnCO<sub>3</sub>-HDPE compact (C/O = 1.5)

At C/O of 2.0, the corresponding maxima are 9.75, 5.72 and 4.75 for CO, CO<sub>2</sub> and CH<sub>4</sub>, respectively, as illustrated in Fig. 17.

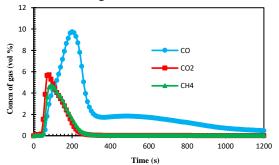
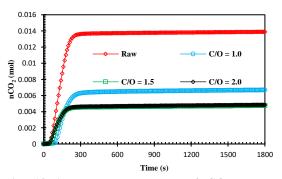
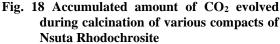


Fig. 17 Gas Generation Behaviour during the Calcination of MnCO<sub>3</sub>-HDPE compact (C/O = 2.0)

# **3.4** CO<sub>2</sub> Emissions during Calcination of Nsuta Carbonate Ore

One major concern associated with the calcination of metal carbonates is the evolution of large amounts of CO<sub>2</sub>, as is typically the case observed in cement making. The accumulated amounts (mol) of CO<sub>2</sub> emitted in the calcination of Nsuta Rhodochrosite are plotted together and illustrated in Fig. 18. A sharp decline in the content of CO<sub>2</sub> in the offgas (from ~0.0138 mol to 0.0067 mol) is observed when HDPE is incorporated in the compact at C/O of 1.0. Increasing the content of HDPE further to C/O ratio of 1.5 decreases the content of CO<sub>2</sub> to 0.00472 mol after 15 min of calcination. Beyond this value of HDPE, no difference in the content of CO<sub>2</sub> is observed, even at C/O of 2.0 as shown in Fig. 18.





As elucidated earlier, in the presence of HDPE,  $CH_4$  is produced along with CO and CO<sub>2</sub>. CO takes over as the dominant gas as  $CO_2$  declines, suggesting that the  $CH_4$  produced from the thermal decomposition of HDPE is reformed by  $CO_2$  from the calcination of the ore, in accordance with equation (5).

$$CO_2 + CH_4 = 2CO + 2H_2$$
 (5)

From equation (5), the CO<sub>2</sub> generated during calcination of Nsuta rhodochrosite ore can be transformed to syngas, if the ore is mixed with pulverised HDPE. A similar observation was made by Dankwah (2014) and Koshy *et al.* (2017) in the thermal decomposition of compacts of HDPE with reagent grade MnCO<sub>3</sub> and Guangzhou Siderite ore (FeCO<sub>3</sub>) from China, respectively. Evidence of the transformation of CO<sub>2</sub> to syngas (CO and H<sub>2</sub>) is provided by the significantly higher values of CO observed in the compacts containing the polymer (Fig. 19) and the significant peak of H<sub>2</sub> observed in the gas chromatogram of Fig. 20.

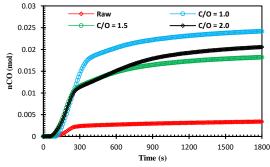


Fig. 19 Accumulated amount of CO evolved during calcination of various compacts of Nsuta Rhodochrosite

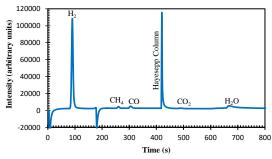


Fig. 20 Gas chromatogram obtained after calcination of MnCO<sub>3</sub>-HDPE compact (C/O = 2.0) for 15 min at 1200 °C

### 4 Conclusions

The calcination behaviour of the Nsuta rhodochrosite ore has been investigated in the presence and absence of HDPE. The as-received ore and the calcined products were characterised by XRF, SEM/EDS and XRD analyses. The major conclusions in this investigation are:

 (i) The Nsuta rhodochrosite ore consists of a mixture of manganese II carbonate (MnCO<sub>3</sub>), silica (SiO<sub>2</sub>), mixed transition metal carbonate of the form Ca(Mn, Mg)(CO<sub>3</sub>)<sub>2</sub> and mixed metal silicate of the form  $Ca_{0.6}Mg_{1.94}Si_2O_6$ 

- (ii) Calcination of the ore resulted in visible colour changes (from grey to dark brown), along with significant changes in the mass before and after the heating process.
- (iii) In the absence and presence of the polymer, measured temperatures in the crucible ranged from 1001 °C to 1366 °C and 1361 °C to 1369 °C, respectively.
- (iv) Analyses by XRF showed marginal increase in the content of Mn (measured as wt% MnO) in the calcined ore with HDPE addition
- (v) Blending the carbonate with the HDPE before heating results in significant decrease in the amount of CO<sub>2</sub> emitted into the atmosphere. The observed decrease in CO<sub>2</sub> emissions is accompanied by a simultaneous production of syngas (CO + H<sub>2</sub>) that can be recovered as a beneficial by-product

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