

## Effect of physicochemical properties and feed mix ratios on the carbothermic reductions of iron ore with coke

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**Abstract.** This study aimed to investigate the effect of physicochemical properties and mix ratios of iron ore (oxide feed): coke (reductant) on the carbothermic reductions of iron ore. Coke size was fixed at  $\leq 63$   $\mu\text{m}$  while iron ore size varied between 150–63  $\mu\text{m}$  and  $\leq 63$   $\mu\text{m}$  respectively. Mix ratios were changed from 100:0 (reference) to 80:20 and 60:40 while the temperature, heating rate and soaking duration in muffle furnace were fixed at 1100 °C, 10 °C/min and 1 hour. Particle size analyzer, XRF, CHNS and XRD analyses were used for determination of raw feed characteristics. The occurrence of phase transformations from various forms of iron oxides to iron during the carbothermal reductions were identified through XRD profiles and supported with weight loss (%). XRF analysis proved that iron ore is of high grade with 93.4% of  $\text{Fe}_2\text{O}_3$  content. Other oxides present in minor amounts are 2%  $\text{Al}_2\text{O}_3$  and 1.8%  $\text{SiO}_2$  with negligible amounts of other compounds such as  $\text{MnO}$ ,  $\text{K}_2\text{O}$  and  $\text{CuO}$ . Composite pellet with finer size iron particles ( $\leq 63$   $\mu\text{m}$ ) and higher carbon content of 60:40 exhibited 45.13% weight lost compared to 32.30% and 3.88% respectively for 80:20 and 100:0 ratios. It is evident that reduction reactions can only occur with the presence of coke, the carbon supply. The small weight loss of 3.88% at 100:0 ratio occurs due to the removal of moisture and volatiles and oxidations of iron ore. Higher carbon supply at 60:40 leads into better heat and mass transfer and diffusivity during carbothermic reductions. Overall, finer particle size and higher carbon supply improves reactivity and gas-solid interactions resulting in increased reductions and phase transformations.

**Keywords:** carbothermic reductions; diffusivity; physicochemical properties; reactivity; reductant

### 1. Introduction

Mineral processing involves liberating and concentrating economic metals and minerals from a mined resource using comminution (particle size reduction), classification (particle size screening), and separation technologies (e.g., flotation, gravity separation, magnetic separation, electrical

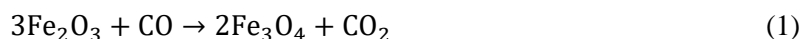
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conduction, etc.) (Balasubramaniam 2015, Dworzanowski 2013, Wills and Munn 2006).

Following to this, mineral concentrates undergo metal extraction which involves extracting valuable metals and metal-bearing compounds from the concentrate and removing gangue minerals and metals using chemical reactions. The metals and compounds will further undergo metal recovery involving impurity removal and concentrating the extracted metals and compounds into a purer form (Whitworth *et al.* 2022). Pyrometallurgy is an extractive metallurgy process that involves high temperature reactions and processes for extraction and metal refining. Reduction is the main changes at a high temperature and refers to acquisition of metal from its oxides by using furnace.

The metallurgical process reaction involved in iron ore reduction for iron and steelmaking occurs by direct reduction and indirect reductions with numerous energy sources such as coal, coke, biomass, hydrogen, waste plastics etc. (Babich and Senk 2013). Indirect reduction includes the reduction by carbon monoxide and hydrogen (Habashi 1997, Gupta 2003, Gosh and Chatterjee 2010). The reduction of iron oxide by carbon monoxide and hydrogen are given in Eqs. (1)-(6).



In the reduction of iron ore with hydrogen, it was generally established that wüstite (FeO) is unstable at temperatures lower than 570 °C under equilibrium condition, but it could be an intermediate product at temperatures below 570 °C under irreversible conditions (Pineau *et al.* 2006, Pourghahramani and Forssberg 2007). Reduction to Fe occurs stepwise from Fe<sub>2</sub>O<sub>3</sub> to Fe<sub>3</sub>O<sub>4</sub> and continues to Fe if the reduction temperature is lower than 570 °C. The intermediate oxide, wüstite is not stable at temperatures lower than 570 °C. However, at temperatures above 570 °C, wüstite must also be considered in the reduction process with a stepwise sequence from Fe<sub>2</sub>O<sub>3</sub> to Fe<sub>3</sub>O<sub>4</sub> to FeO and continues to Fe (Pineau *et al.* 2006, Heidari *et al.* 2021). Generally, a higher temperature can generate more heat hence, they can increase the kinetic parameter of the reduction of hematite, Fe<sub>2</sub>O<sub>3</sub> to magnetite, Fe<sub>3</sub>O<sub>4</sub> (Hou *et al.* 2012, Spreitzer and Schenk 2019). Basically, the carbon gasification reaction is slow at low temperatures. However, the reaction can be increased by increasing the temperature. Previous investigations by Man and Feng (2016) on the effect of iron ore-coal pellets reduction with hydrogen, H<sub>2</sub> and carbon monoxide, CO showed that the reduction reaction that occurs were nearly the same either in CO or H<sub>2</sub> atmosphere at a temperature of 900 °C. However, the carbon gasification reaction was inhibited by the presence of carbon monoxide, and the reaction fraction in H<sub>2</sub> atmosphere was higher than in CO atmosphere at lower temperature. When the temperature was increased up to 1000 °C, the reduction rate was increased too. Obviously, the reduction rate in the H<sub>2</sub> atmosphere is higher compared to in the CO atmosphere. It can be concluded that by increasing the temperature, it will lead to an increase of reduction reaction in both CO and H<sub>2</sub> atmosphere. The reduction of iron ore by hydrogen gas as reducing agent occurs in stages as shown in Eqs. (4)-(6) (Ünal *et al.* 2015).



Thermodynamically, CO has a higher reducing ability at lower temperature whereas H<sub>2</sub> reduction is more stable at higher temperature. From kinetic point of view, due to the atomic size of hydrogen and its diffusivity, H<sub>2</sub> is a faster reductant in comparison to CO at temperature above 850 °C (Heidari *et al.* 2021). Comparative to indirect reduction of CO, direct reduction uses carbon reductant in the iron ore reduction process as given by Eqs. (7)-(9).



Apart from the types of reducing agent, carbon sources, temperature, pressure, gas compositions, iron oxide mineralogy and gangue, the mechanical activation and grain or particle size of ore and reductant also influence the reduction process (Spreitzer and Schenk 2019). As mentioned by Li *et al.* (2018), the rate of reaction can be accelerated by reducing the size of the ore and reductant particles. Particle sizes also could affect the degree of metallization (%) which contributes to the reduction rate. This is caused by the reduction of granularity of iron ore during the reduction reaction. Man *et al.* (2014b) investigated the effect of temperature and time on the reduction behavior of iron ore-coal composite pellets with average particle size of 74 μm for iron ore concentrate and 109 μm for the bituminous coal. The reduction was carried out on a thermogravimetric apparatus which consisted of a vertical furnace, a quartz glass tube reaction chamber, an electronic balance, and a temperature controller. The reaction temperature was varied between 700 °C to 1100 °C while the reaction time fixed at 120 minutes in inert atmosphere with nitrogen flow rate of 0.5 L/min. XRD phase analyses showed that the material mainly composed of hematite (Fe<sub>2</sub>O<sub>3</sub>) and magnetite (Fe<sub>3</sub>O<sub>4</sub>) from the mineral phase composition. At 700 °C, weight of Fe<sub>2</sub>O<sub>3</sub> decreased and the weight of Fe<sub>3</sub>O<sub>4</sub> increased significantly with additional appearance of FeO. At 800 °C, the weight of FeO increased further with coexistence of Fe<sub>2</sub>O<sub>3</sub> and Fe<sub>3</sub>O<sub>4</sub> while at 900 °C, FeO occupied a dominant position in the mineral phase; Fe<sub>3</sub>O<sub>4</sub> and Fe<sub>2</sub>O<sub>3</sub> remained little. Metallic Fe appeared when the temperature reached 1000 °C indicating a high reduction degree. At 1100 °C, metallic Fe dominated the sample with coexistence of iron oxides. The present study aims at investigating the effect of size ranges (150-63 μm) and ≤63 μm of iron ore towards its reduction behavior using metallurgical coke as reductant at a fixed reductant temperature of 1100 °C.

## 2. Materials and methods

### 2.1 Sample preparations

Comminution and sizing methods were used to reduce and screen the particle size of raw iron ore and coke particles into desired working size of 150-63 μm and ≤63 μm respectively. Jaw crusher and planetary ball mill (Planetary Mono Mill Pulverisette 6 Classic Line) was used for the crushing and grinding test works. All the milling and grinding process were conducted in dry mode. Grinding bowl and media used are of stainless-steel type (density: 7.8 g/cm<sup>3</sup>) with uniform size grinding balls. Sampling and sample preparation techniques were used to obtain representative and homogenous samples for numerous characterization and reduction test works.

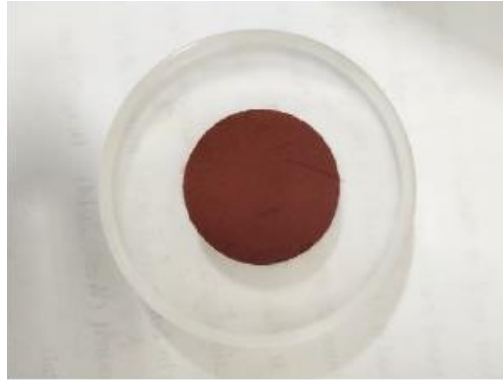


Fig. 1 Iron ore pellet

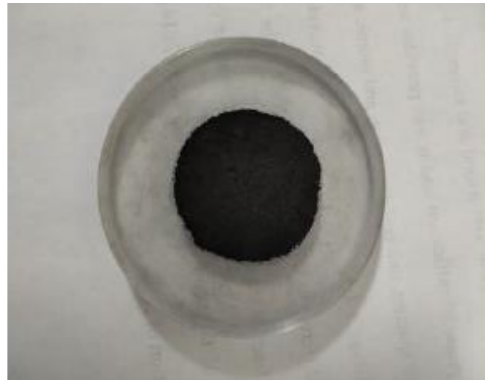


Fig. 2 Coke pellet

### *2.2 Compaction and reduction process*

Iron ore (oxide source) of 150-63  $\mu\text{m}$  and  $\leq 63 \mu\text{m}$  and coke of  $\leq 63 \mu\text{m}$  size ranges were prepared for mixing, blending, compaction and reduction processes. Green pellets consisting of iron ore particles (oxide source) and coke as carbon reductant were prepared using three different ratios of 100:0, 80:20 and 60:40. The fine grounded raw materials were mixed and compacted using a hydraulic press machine. All the green pellets were heat treated in a muffle furnace at 1100  $^{\circ}\text{C}$  for 1 hour of soaking time and a heating rate of 10  $^{\circ}\text{C}/\text{min}$ . Figs. 1-2 show the compacted green pellets of iron ore and coke of approximately 12 mm diameter size.

### *2.3 Characterization of raw samples and reduced products*

The chemical compositions of raw iron ore was identified using PANalytical PW4030 X-ray Fluorescence (XRF) technique. Carbon, Hydrogen, Nitrogen & Sulphur (CHNS) Elemental Analyzer was used to determine the chemical composition of coke. BS EN ISO 16948: 2015 test method was used for determination of Carbon, Hydrogen and Nitrogen while Elementar Vario Micro Cube, Germany method used for Sulphur determination. Particle size distribution (PSD) and span values for iron ores and coke were determined by using laser diffraction particle size analyzer of

Table 1 XRF analysis of iron ore

Compound	Fe <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	CuO	SO <sub>3</sub>	ZnO	Br	V <sub>2</sub> O <sub>5</sub>
Concentration Percentage (wt.%)	93.40	0.58	1.8	2.0	0.54	0.054	0.17	0.050	0.10	0.043

Malvern Mastersizer-v3.30 in wet mode using water as dispersant. Eq. (10) represents the formula for width of the particle size distribution/ span determination.

$$\text{Span} = \frac{d_{90}-d_{10}}{d_{50}} \quad (10)$$

where,

$d_{90}$ =90% passing size

$d_{50}$ =50% passing size

$d_{10}$ =10% passing size

The weight loss of reduced pellets was determined through analytical technique and calculated using Eq. (11).

$$\text{Weight loss} = \frac{[(m_i-m_f)]}{(m_i)} \times 100\% \quad (11)$$

where,

$m_i$ =initial mass of pellets (g)

$m_f$ =the mass of pellets (g) after reduction

The phases present in the raw iron ore, coke and reduced samples were analyzed by using the XRD-6000 Shimadzu X-Ray Diffraction (XRD) machine at 1°/min in the 2θ range of 10° to 90° at 40kV and 40 mA in 0.04° steps.

### 3. Result and discussions

#### 3.1 Characterizations of raw samples

##### 3.1.1 Chemical composition analysis

Table 1 shows the XRF analysis results of iron ore. Based on the chemical composition results, the raw iron ore can be classified as high-grade iron ore because it contains a high amount (93.4 wt.%) of hematite (Fe<sub>2</sub>O<sub>3</sub>). Other minor constituents are alumina (Al<sub>2</sub>O<sub>3</sub>) and silica (SiO<sub>2</sub>) with 2.0% and 1.8 wt. % respectively. Negligible amounts of MnO, SO<sub>3</sub>, K<sub>2</sub>O, CuO and ZnO also present in the sample.

##### 3.1.2 CHNS analysis of coke

The CHNS analysis of metallurgical coke sample used is given in Table 2. CHNS analysis proved that coke sample contains high amount of carbon with composition percentage of 90.7%. Besides that, the sample also contains 2.6% Sulfur, 1.5% Hydrogen and 0.6% Nitrogen which all influence the reduction process.

##### 3.1.3 Particle size distributions

Table 2 CHNS analysis of coke

Element	Percentage (%)
Nitrogen	0.6
Carbon	90.7
Hydrogen	1.5
Sulfur	2.6

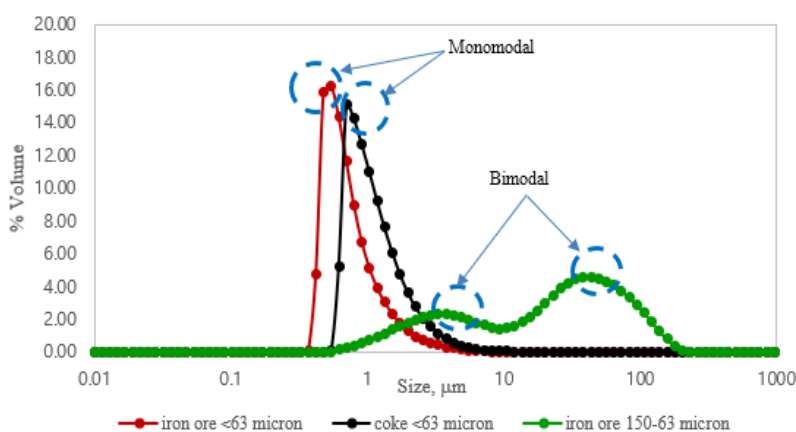


Fig. 3 Particle size distribution for iron ores and coke

The size distribution of iron ore used varies at two size gradations ranging from 150-63  $\mu\text{m}$  and  $\leq 63\ \mu\text{m}$  while the reductant coke size of  $\leq 63\ \mu\text{m}$  used throughout the experimental procedure. Fig. 3 and Table 3 show the particle size distributions pattern and quantitative  $d_{10}$ ,  $d_{50}$ ,  $d_{90}$  values of the two groups of iron ore and coke samples used for reductions.

The particle size distributions in Fig. 3 showed mono-modal distributions for iron ore ( $\leq 63\ \mu\text{m}$ ) and coke ( $\leq 63\ \mu\text{m}$ ) whilst bi-modal distributions for iron ore of 150-63  $\mu\text{m}$ . Dominantly one major population of particles and two major particle populations can be seen to exist in iron of  $\leq 63\ \mu\text{m}$  and 150-63  $\mu\text{m}$  respectively. Span value of 3.325 compared to an ideal value of 1.00 proved that iron ore of 150-63  $\mu\text{m}$  range have a broad particle size distribution. Comparatively, span values of 1.444 and 1.391 for coke and iron ore of  $\leq 63\ \mu\text{m}$  significantly shows that the particles are of controlled and more homogeneous size distributions. The  $d(90)$  sizes of 2.28  $\mu\text{m}$ , 1.47  $\mu\text{m}$  and 93.8  $\mu\text{m}$  for coke and iron ores of  $\leq 63\ \mu\text{m}$  and 150-63  $\mu\text{m}$  sizes showed that 90% of passing size are within the specified range. As mentioned by Palaniandy *et al.* (2009), the volume mean diameter  $d(4,3)$  was more preferred when the particle size distribution had poly modal distribution, and  $d(50)$  did not effectively represent the particle size distribution of the samples. High specific surface areas (SSA) of 641.2  $\text{m}^2/\text{kg}$  and 777.2  $\text{m}^2/\text{kg}$  for coke and  $\leq 63\ \mu\text{m}$  size iron ore, computed using laser diffraction technique proved greater SSA for finer sizes compared to its coarser counterpart (150-63  $\mu\text{m}$ ).

### 3.1.4 Structural phases of raw samples

The observation of the phases and elements that present in samples was conducted by using X-Ray Diffraction (XRD) analysis and Expert High Score plus software. Fig. 4 shows the XRD diffraction analysis of raw iron ore.

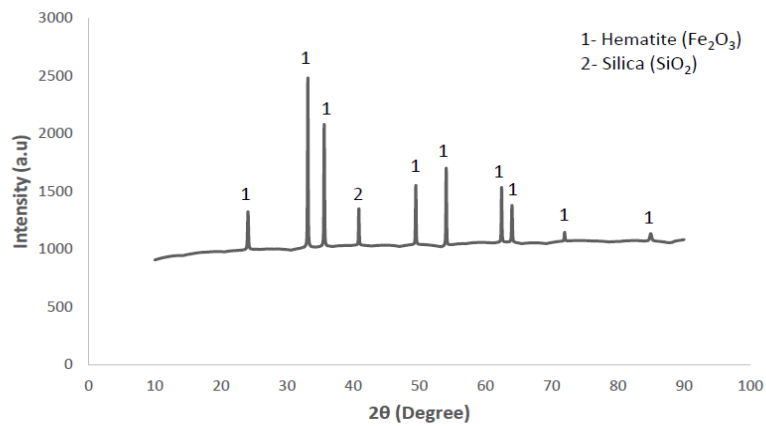


Fig. 4 XRD pattern of raw iron ore

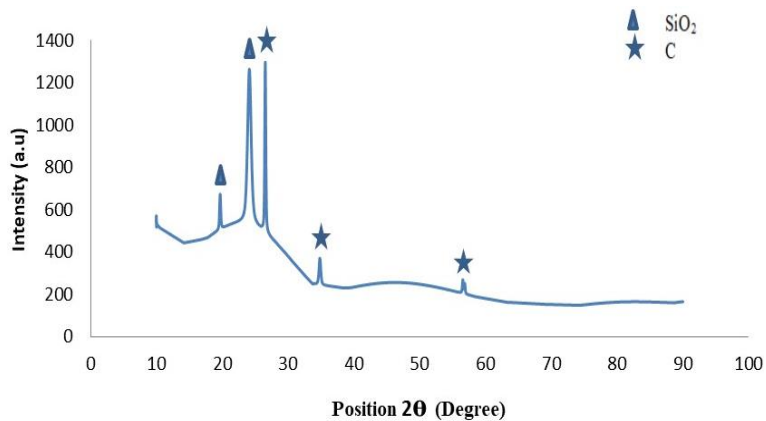


Fig. 5 XRD pattern of coke

XRD analysis showed the presence of dominant hematite ( $\text{Fe}_2\text{O}_3$ ) and minor  $\text{SiO}_2$  phases in the raw iron ore. The highest peak of hematite was detected at  $2\theta$  angle of  $33.11^\circ$  corresponding to the iron oxide (104) plane. The peaks of iron ore also were detected at  $2\theta$  angles of  $24.12^\circ$ ,  $35.11^\circ$  and  $49.41^\circ$  which lies on (012), (110) and (024) planes respectively. Fig. 5 shows the XRD pattern of coke. Based on the graph, the highest peak of carbon was obtained at  $2\theta$  angle of  $26.6^\circ$  corresponding to the coke (002) plane. The peaks of carbon were also found at  $34.8^\circ$  and  $56.8^\circ$  which lies on the (010) and (004) planes respectively.

### 3.2 Iron ore reductions

#### 3.2.1 Phase changes prior to reduction

Coke is primarily used as a reducing agent and energy carrier. Figs. 6-7 show the phases present and difference in peak intensities with different amount of carbon for particle size between 150 to  $63\ \mu\text{m}$  and also  $\leq 63\ \mu\text{m}$  respectively.

Based on Figs. 6-7, the phases that present in the reduced pellets of 150- $63\ \mu\text{m}$  size were hematite, magnetite, and silica. However, the reduced finer fractions of  $\leq 63\ \mu\text{m}$  size shows the

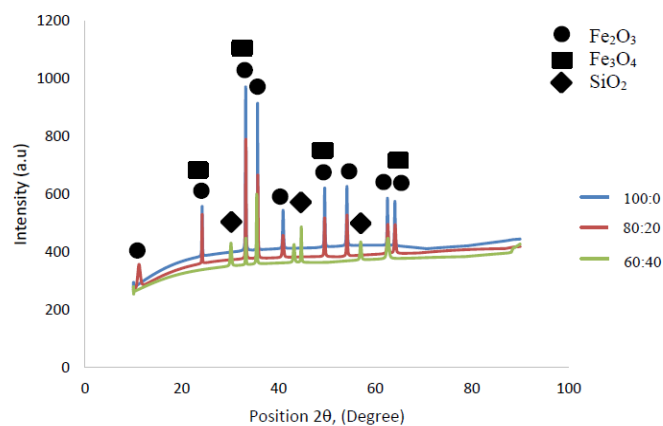
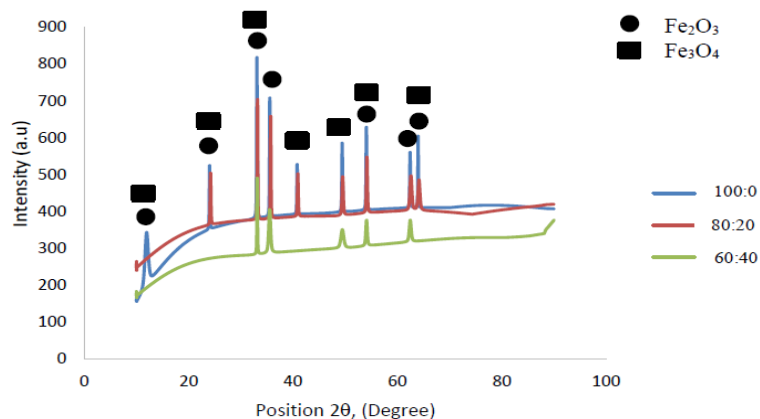
Fig. 6 Phases and intensity variations for mixture of iron ore (150-63  $\mu\text{m}$ ) and  $\leq 63 \mu\text{m}$  cokeFig. 7 Phases and intensity variations for mixture of  $\leq 63 \mu\text{m}$  iron ore and  $\leq 63 \mu\text{m}$  coke

Table 3 Quantitative particle size values

Particle Properties	Coke	Iron ore ( $\leq 63 \mu\text{m}$ )	Iron ore (150-63 $\mu\text{m}$ )
$d[3,2] \mu\text{m}$	19.5	13.8	7.23
$d[4,3] \mu\text{m}$	71.5	99.7	38.4
$d(10) \mu\text{m}$	0.733	0.5	2.42
$d(50) \mu\text{m}$	1.07	0.695	27.5
$d(90) \mu\text{m}$	2.28	1.47	93.8
SSA ( $\text{m}^2/\text{kg}$ )	641.2	777.2	106.4
Span, $\psi$	1.444	1.391	3.325

coexistence of hematite and magnetite phases only. The presence of  $\text{Fe}_3\text{O}_4$  indicates the occurrence of reduction reactions and phase transformations. Accordingly, the disappearing  $\text{SiO}_2$  peaks in the reduced product of finer sizes gives an insight of impurity and gangue removal during the reduction.

### 3.2.2 Weight loss percentage



Table 4 Weight loss of samples prior to reduction reaction

Ratio of iron ore:coke (%)	Particle Size					
	150-63 $\mu\text{m}$			$\leq 63 \mu\text{m}$		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
100:0	3.78%	3.5%	3.39%	3.93%	3.77%	3.94%
80:20	27.01%	25.14%	26.29%	32.21%	32%	32.7%
60:40	36.94%	34.67%	36.39%	43.4%	46.45%	45.53%

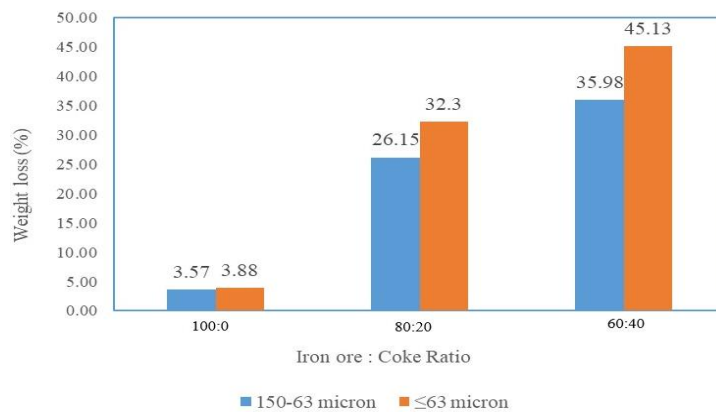


Fig. 8 The average weight loss for all six samples

In the reduction reaction of iron ore, coke was used as a reducing agent. As stated by (Pourghahramani and Forssberg 2007, Heidari *et al.* 2021), reduction reactions of iron ore is a complex process because it is a heterogeneous reaction in which several simultaneous reactions usually occur involving  $\text{Fe}_2\text{O}_3$ ,  $\text{Fe}_3\text{O}_4$ ,  $\text{FeO}$  and  $\text{Fe}$  phases. The process could be described through a chain of single reactions, a combination of single or double reactions or a triple reaction. It is known that particle size and coke content can influence the reduction rate of iron. Table 4 shows the weight loss details of each sample after undergoing the reduction process while Fig. 8 displays the graph of average weight loss for all six samples.

Mechanical activation via comminution process (crushing and grinding) plays a significant role in reducing the particle size of raw iron ore and coke. Based on the data of weight loss that were obtained, it was found that both size range react differently towards the reduction process. The samples in range of  $\leq 63 \mu\text{m}$  have greater loss in weight compared to 150-63  $\mu\text{m}$  size range. Finer particle size ( $\leq 63 \mu\text{m}$ ) have greater specific surface area (refer to Table 3) and leads to greater solid-gas contact and diffusivity for removal of large amount of oxygen bind together with hematite ore. The transformation of iron oxides can be observed from the corresponding change of peak. Initially the sample was dominated by hematite ( $\text{Fe}_2\text{O}_3$ ) and at 1100 °C, the magnetite ( $\text{Fe}_3\text{O}_4$ ) increased significantly indicating the reduction process. Previous investigations on the effect of particle size had been reviewed and summarized by Spreitzer and Schenk (2019). These includes investigations by Teplov in 2012 on the reduction kinetics of magnetite concentrates of different grain sizes with hydrogen at low temperatures (300-570 °C) which proved that reduction rate clearly increases with decreasing particle size, especially at the initial and final stages of reduction due to changing the rate-limiting step. Also reported were studies by Chen *et al.* in 2015 on the reduction kinetics of

hematite particles with an average particle size of 21  $\mu\text{m}$  between 1150 and 1350  $^{\circ}\text{C}$  using flash reactor. It was proven that higher reduction degree (more than 90%) could be achieved in a few seconds due to the good kinetic behavior of the ultra-fine material used in combination with hydrogen as a reducing agent. Temperature used also plays a significant role in reduction process as formation of dense iron layers at lower temperature could prevent gas contact with the iron oxide interface, hence slow down the reductions (Man *et al.* 2014a).

The samples with mixing ratio of 60:40 iron ore-coke content also displayed greater weight loss compared to 80:20 mix ratio. This is due to high carbon content and moles of reacting coke assist in greater removal of oxygen and reductions of iron ore. It also indicates a better performance of coke in the reduction of iron due to greater gas diffusivity. Since the carbon content is high, it increases the reduction rate. According to Man *et al.* (2014a, b), the increase of mass loss rate with temperature could be due to the increase of reacting moles which lead to the increase of adsorption rate. Increasing temperature also leads to an increase in mass transfer rate during diffusion and chemical reaction rate. Iron ore-coke weight ratio of 100:0 was used as reference to prove that reduction reaction only occurs with the presence of coke. The weight of all samples of iron ore-coke weight ratio of 100:0 was seen to reduce slightly prior to 1-hour heat treatment in the furnace. The weight loss still occurred due to the removal of moisture and volatile contents. Even though no reduction reactions as no reducing agent (coke) used but oxidation reactions might occur due to the gain of oxygen from furnace atmosphere.

#### 4. Conclusions

The effect of physicochemical properties such as chemical compositions and particle size distributions and feed mix ratios towards the reduction of iron ore have been investigated. Composite pellets were prepared using iron ore of 150-63  $\mu\text{m}$  and  $\leq 63$   $\mu\text{m}$  sizes whereas size of coke particles fixed at  $\leq 63$   $\mu\text{m}$ . Few observations as listed below were concluded from this investigation.

- The finer size iron particles ( $\leq 63$   $\mu\text{m}$ ) consisting of higher carbon content of 60:40 exhibited 45.13% weight lost compared to 32.30% and 3.88% respectively for 80:20 and 100:0 ratios. Higher weight loss % in the mixture of finer particle sizes showed that reduction rate is greater as finer particles contribute to larger specific surface area for better diffusion of gas for reaction mechanisms.
- The pellets consisting of wider range of iron particles (150-63  $\mu\text{m}$ ) displayed lesser average weight loss of 35.98%, 26.15% and 3.57% at 60:40, 80:20 and 100:0 ratios. Although greater moles of carbon react in the 60:40 ratio, the wider range of particle populations can be clearly seen as decreasing the reductions of iron ore. Hence, the importance of particle size is clearly substantial.

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