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Investigation on the pre-reduction of ilmenite pellet using H_2/CO atmospheres

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ABSTRACT
In this study, ilmenite and bentonite were uniformly mixed, pelletised, and baked at temperatures up to 1200°C. The reduction of ilmenite pellet by H_2/CO mixtures and H_2 pure gas was studied in the temperature range of 760–890°C. Reduced pellet microstructures and phases analysed by, Optical Microscopy (OM), Scanning Electron Microscopy (SEM), and Energy Dispersive X-Ray Analysis (EDX) Mapping. Experimental works shown that between 760°C, 880°C, and 890°C, the H_2 amount and reduction temperature have a considerable influence on the degree of metallisation. The obtain results demonstrate the degree of metallisation increased with increase of reduction temperature and reaction time. The degree of metallisation could reach 93.64% when the sample was reduced to 890°C for 360 min in pure H_2 atmosphere. In microstructural analysis, metallic iron particles became smaller and more uniformly distributed as hydrogen content of the reducing gas increased.

L'ilménite est l'une des plus importantes sources de titane dans la nature. Une utilisation majeure de l'ilménite est la production de laitier à haute teneur en oxyde de titane et de fonte brute. Afin de produire du laitier d'oxyde de titane d'une grande pureté à partir de l'ilménite, on a besoin de procédés appropriés ayant moins d'impacts environnementaux. Le gaz synthétique, réformé par le gaz naturel, peut diminuer l'utilisation du charbon et le dégagement de CO_2 lors du traitement de l'ilménite, qui implique la réduction et la métallisation de l'oxyde de fer. Dans cette étude, l'ilménite et la bentonite ont été mélangées uniformément, bouletées et cuites à des températures allant jusqu'à 1200°C. On a étudié la réduction des boulettes d'ilménite par des mélanges H_2/CO et du gaz H_2 pur dans la gamme de température de 760 à 890°C. On a analysé les microstructures et les phases des boulettes réduites par microscopie optique (MO), microscopie électronique à balayage (MEB) et cartographie d'analyse par rayons X à dispersion d'énergie (EDX). Les travaux expérimentaux ont montré qu'entre 760, 880 et 890°C, la quantité de H_2 et la température de réduction avaient une influence notable sur le degré de métallisation. Les résultats obtenus démontrent que le degré de métallisation augmente avec l'augmentation de la température de réduction et du temps de réaction. Le degré de métallisation pouvait atteindre 93.64% lorsque l'échantillon était réduit à 890°C pendant 360 min dans une atmosphère de H_2 pur. Dans l'analyse microstructurale, les particules de fer métallique devenaient plus petites et distribuées plus uniformément à mesure que la teneur en hydrogène du gaz réducteur augmentait.

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Titanium; Ilmenite; reduction; iron metallisation; hydrogen; carbon monoxide; temperature; pellet

1. Introduction

Titanium is a relatively abundant element in the earth's crust and is commonly found in ilmenite ($FeO{\cdot}TiO_2$), rutile (TiO_2), and titanomagnetite (Fe_2TiO_4–Fe_2O_4) in igneous and metamorphic rocks. Rutile is easily processed into titanium metal or TiO_2 pigment using the chlorination method, while ilmenite is more difficult to process owing to its high iron content. Research shows that almost 95% of titanium is used for producing white TiO_2 pigment, which has a wide range of applications in industries such as paint, plastics, rubber, as well as being used as a flux in glass and paper production [1]. 90% of the world's titanium reserves are found in the form of ilmenite, with only 10% in the form of rutile and other ores. As a result, the treatment of ilmenite is a critical concern in the field of metallurgy [2]. Ilmenite ($FeTiO_3$) is the most significant mineral source of titanium, with TiO_2 content ranging from 45% to 60%. The global TiO_2 pigment industry has a value of US $15 billion, with an annual production of 7.7 million tons [3]. In post-treatment processes used for ilmenite concentrate, slag production is a commonly employed method for reducing and separating metallic iron from ilmenite. This results in the production of high titania slag and pig iron. The high titania slag can be used in subsequent processes, while the pig iron is sold as a byproduct [4].

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Titanium dioxide pigment is typically produced from titanium ores using either the sulphate process (which accounts for 40% of total TiO_2 production) or the chloride process (which accounts for 60% of total TiO_2 production). The primary differences between these two processes lie in their chemistry and raw material requirements [5]. In the sulphate process, low-grade ilmenites are dissolved in concentrated sulphuric acid. After the iron is separated, the resulting titanium liquor is hydrolyzed to produce hydrated titanium dioxide [6]. In the chloride process, either high-grade rutile or titanium slag is chlorinated in a fluidised bed reactor with the presence of coke to produce $TiCl_4$ vapour. This vapour is then converted to titanium dioxide in the presence of oxygen [6]. In the production of slag, ilmenite can be fed into the process either directly or in a pre-reduced state. Direct feeding is typically used in DC furnaces or AC submerged furnaces, while pre-reduced ilmenite is used in AC open arc furnaces [7].

In industrial practice, pre-reduced ilmenite ore is smelted with carbon in high-temperature furnaces. To achieve pre-reduction, the ore is mixed with a specific amount of carbon that is sufficient to reduce the iron oxide component of the ore. The mixture is then heated in a rotary kiln before being charged in an electric furnace at a temperature of 1650°C. During this process, the remaining iron oxide is reduced to metal, while the titanium is separated as a slag. The purpose of ilmenite smelting is to produce an upgraded titania slag product, which is achieved by partially reducing the FeO from ilmenite ($FeO.TiO_2$) using carbon as a reductant [8]. The resulting upgraded slag typically has an equivalent TiO_2 content (i.e. the total amount of titanium present as TiO_2 and Ti_2O_3) of 85% by mass or greater and is used in the production of pigment using either the chloride or sulphate routes [9].

The resulting pig iron can be used for castings, while the slag can be further processed to extract titanium dioxide. The slag is high in titanium and low in iron, making it preferable to ilmenite for manufacturing TiO_2 pigment or titanium metal [10,11]. To obtain titanium-rich material from ilmenite ores, it is necessary to remove the iron. Various methods have been proposed to refine ilmenite, including smelting, direct acid leaching, selective chlorination, and reduction [12].

Pisila et al. [13] conducted a study to investigate the effect of pre-reduced and raw ilmenite concentrates smelting on energy and carbon consumption in the DC furnace. Their results showed that using pre-reduced ilmenite resulted in lower carbon content in the pig iron, and the energy consumption of the smelting process was 27% lower compared to smelting raw ilmenite. To efficiently carry out the reduction, gaseous reducing agents such as carbon monoxide or hydrogen can be used in a gas–solid reactor, such as a fluidised bed.

Ilmenite is currently processed using large amounts of coal, but it could potentially be substituted with natural gas, which could increase efficiency and lower CO_2 emissions. To reduce CO_2 emissions, a CO/H_2 mixture can be used as a substitute for coal as a reducing agent. Direct reduced iron (DRI) technology, which uses CO/H_2 mixture as a reducing agent, could be adapted for ilmenite reduction. Natural gas, primarily consisting of CH_4, can be reformed to produce synthesis gas, a mixture of carbon monoxide (CO) and hydrogen (H_2) [14].

The use of synthesis gas for reduction is a well-established practice in the iron ore industry for direct-reduced iron (DRI) production. Pre-reduction of ilmenite can be compared to the DRI process since it mainly involves reducing iron oxides. However, a major difference is that a stronger reducing atmosphere is required for ilmenite pre-reduction owing to the presence of titanium and the high stability of iron-titanium intermediate oxides [15]. Merk et al. [11] investigated the reduction of iron oxide in ilmenite by carbon monoxide in the temperature range of 500–1000°C. Their findings indicated that the rate and extent of reduction increased linearly with increasing temperature up to approximately 1000°C. Zhang et al. [16] investigated the reduction of ilmenite concentrates using CH_4-H_2-Ar gas mixtures in a laboratory fixed bed reactor within the temperature range of 850–1350°C. Within this temperature range, iron oxides in ilmenite were rapidly reduced to metallic iron. The rate and extent of ilmenite reduction increased with increasing temperature from 950°C to 1150–1200°C. An increase in methane content resulted in a higher rate of titanium oxycarbide formation, but the reduction extent decreased. On the other hand, an increase in hydrogen content increased both the rate and extent of reduction.

In recent years, there has been a growing interest in the direct reduction of ilmenite ores, and hydrogen has been investigated as the primary reductant for this process [17,18]. Conducted a review of the reduction of ilmenite concentrate using hydrogen gas within the temperature range of 500–1200°C. Their findings showed that the degree of metallisation increased with increasing reduction temperature and reaction time. When the sample was reduced at 1150°C for 80 min, the degree of metallisation reached 87.5%. The final phase constituents mainly consisted of Fe, M_3O_5 solid solution phase (M = Mg, Ti, and Fe), and a small amount of titanium oxide. Hydrogen is a clean energy carrier that enables CO2-free reduction processes. The main difference between hydrogen and carbon as a reducing agent is that the rate of reduction with hydrogen is much faster than that with carbon monoxide or carbon [19].

Many studies have focused on the pre-reduction of ilmenite using CO, H_2, and CO/H_2 mixture gases. The goal of the present study is to investigate the factors of different time and temperature on the metallisation degree in the presence of various reducing agent. As mentioned previously, the presence of H2 gas and higher temperatures increases the reaction rates, but the degree of influence from each variable has not been investigated. Also, the pelletising process is employed in this study to enable ilmenite to be fed in a direct reduction shaft furnace instead of fluidised bed reactors which are more complicated in comparison with shaft furnaces.

2. Experimental procedure

2.1. Materials

The raw ilmenite concentrate used in this investigation was obtained from the Kahnooj mine in Iran. Table 1 presents the chemical composition of the ilmenite concentrate.

2.2. Pelletising process

Around 100 kg of ilmenite concentrate, along with 5% by weight of bentonite as a binder and 0.03%wt of NaOH, were thoroughly mixed in a blender. Sodium hydroxide is utilised in the pelletising process to increase the strength of the pellets. This is achieved by altering the chemical composition of calcium-bentonite to sodium-bentonite, which facilitates easier dispersion within the ilmenite matrix. The pelletising process was carried out using a pilot plant balling disc. Spherical green pellets with a size range of 10–16 millimetres were produced. These green pellets were then baked up to 1200°C using the Lurgi method in a pilot plant. After the preparation of pellets, they were subjected to various tests such as Drop Number (DN), Green Compressive Strength (GCS), Dry Compressive Strength (DCS), and cold compression strength (CCS). DCS, GCS, and CCS are different methods for measuring the compressive strength of pellets, with the unit of measurement being kilograms per pellet (kg/pellet). The Dry Compression Strength (DCS) test is conducted on the green pellet obtained from the disc. The Green Compression Strength (GCS) test is performed after the surface moisture of the pellet has been removed. The Cold Compression Strength (CCS) test measures the compressive strength of the pellet after it has been fired and cooled. The pelletising pilot plant and test flow sheet are shown in Figure 1(a) and (b), respectively.

2.3. Reduction process

The reduction of ilmenite pellets in CO/H_2 and H_2 gases with different ratios was investigated using a Linder test (Dynamic Reducibility 7000), which operates according to the ISO 11257 standard for determining low-temperature reduction RDI-DR. According to the ISO 11257 standard, the reducing gas must contain the following percentages: H_2 (55%±1%), CO (36%±1%), CO_2 (5%±1%), and CH_4 (4%±1%). The parameter value for the H_2/CO ratio was selected in accordance with the standard, with a value of 1.57. The ratio of 3 was chosen to observe the reducing effect of H_2, as it is nearly twice the value of 1.57. Flow rate was selected according to standard ISO 11257, the flow rate of the reducing gas, during the entire reducing period, shall be maintained at 13 L/min. Nitrogen (N_2) shall be used as the heating and cooling gas. The test portion was isothermally reduced in a rotating tube bed, at various temperatures, using a reducing gas consisting of H_2/CO mixture gases at different times. The selection of 300 min was based on the ISO 11257 standard, and the 360-min duration was chosen to investigate the effect of increasing the duration on reduction. After 300 or 360 min of reduction, the flow of reducing gas was stopped and the rotation of the reduction tube was stopped. The reduced test portion was then cooled down to room temperature under a flow of N_2 gas.

The reduced product was then sieved using a 3.15 mm square opening sieve and analysed for total iron and metallic iron. The reduction data are presented in Table 2, and the degree of metallisation was calculated from the chemical analysis results. Figure 2(a) shows Linder test equipment and Figure 2(b) a schematic drawing of the experimental apparatus used for pellet reduction using an H_2/CO mixture with different molar ratio of H_2 to CO. The experimental procedure for conducting the reduction test is presented elsewhere [20]. For optical microscopy analysis, selected pellets were mounted in epoxy and polished. The specimens were ground on silicon carbide papers to a 2000 grade

Table 1. Chemical compositions of the ilmenite and bentonite.

Chemical composition	%TiO_2	%Fe_2O_3	%SiO_2	%Al_2O_3	%CaO	%MgO	%MnO	%Na_2O	%P_2O_5	%K_2O	%LOI
Ilmenite concentrate	43/1	49	4	1	1/1	1/18	0/48	0/06	0/06	0/02	0
Bentonite	0/21	2/54	63/14	11/59	3/03	2/93	0/05	2/14	0/03	0/34	12/98

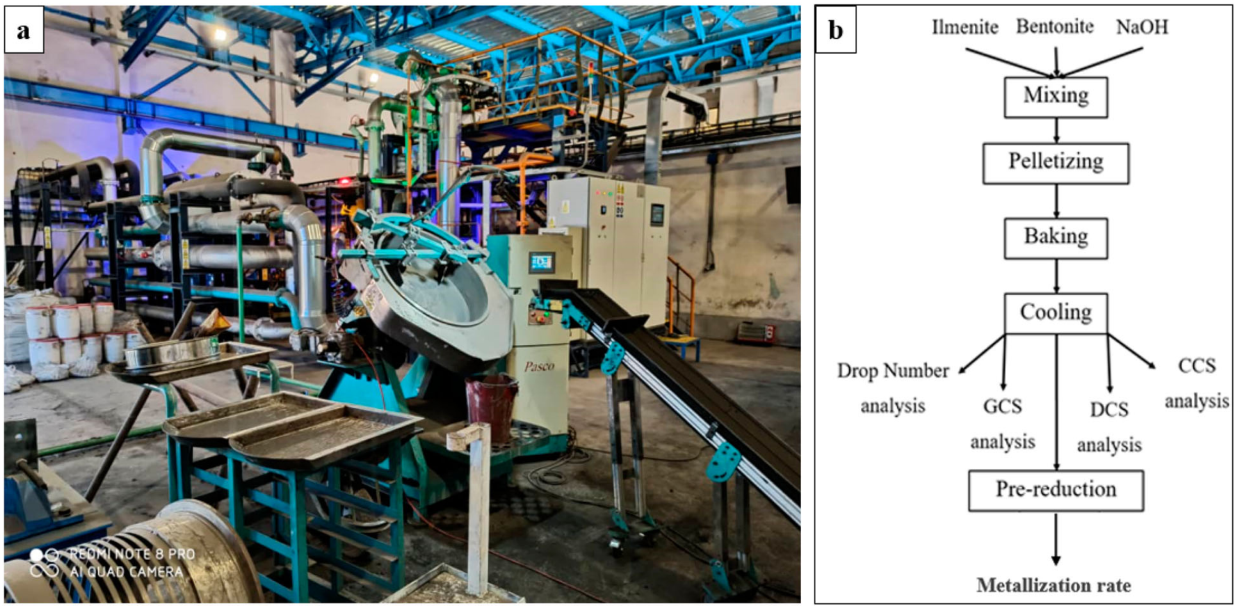


Figure 1. (a) pelletising pilot plant, (b) test flow sheet.

and then polished successively with 6 μm Struers diamond lapping compound. Optical micrographs were captured using an Olympus-CX21 light microscope equipped with an Olympus-SC180 Colour camera and Dino Capture 2.0 software.

2.4. Determination of metallisation degree

To determine the metallisation degree, the experiment was divided into five main steps, which included mixing, pelletising, baking and cooling, pre-reduction, and metallisation calculation. The degree of metallisation, represented as MD and expressed as a percentage by mass, was calculated using the following equation(1):

$$\mathrm{MD} = \frac{M_{\mathrm{Fe\ Metal}}}{M_{\mathrm{Fe\ Total}}} * 100 \tag{1}$$

where M Fe Metal is the weight of metallic iron in the reduction products, and M Fe Total is the total weight of iron in the ilmenite concentrate before reduction, which was measured by chemical titration analysis.

Table 2. Experimental tests.

Number	sample	Reaction Temp(°C)	Reaction Time (hour)	Reduction Gas
1	Experiment 1	760	300	ratio H2/CO: 1.57
2	Experiment 2	880	300	ratio H2/CO: 1.57
3	Experiment 3	890	300	ratio H2/CO: 1.57
4	Experiment 4	890	360	ratio H2/CO: 1.57
5	Experiment 5	760	300	ratio H2/CO: 3
6	Experiment 6	880	300	ratio H2/CO: 3
7	Experiment 7	890	300	ratio H2/CO: 3
8	Experiment 8	890	360	ratio H2/CO: 3
9	Experiment 9	760	300	H2 pure
10	Experiment 10	880	300	H2 pure
11	Experiment 11	890	300	H2 pure
12	Experiment 12	890	360	H2 pure

3. Results and discussion

The results of the ilmenite pellet analysis are presented in Table 3, which includes the determination of moisture based on the ISO 3087 standard and the determination of CCS (cold crushing strength) according to the ISO 4700 standard.

3.1. Effect of gases mix on metallisation degree

In the experimental range, iron oxides in the ilmenite were reduced to metallic iron depending on temperatures, gas mixtures, and reduction times. Figure 3 shows the effect of temperature on metallisation degree in different reduction atmospheres after 5 hr. It can be seen that by increasing temperature, the metallisation degree is increased in all reduction atmospheres. The degrees of reduction of the sample reduced at 760°C in the 1.57, 3 and pure H_2 are respectively 14.78%, 53.03%, and 64.19% after 5 hr. The reduction rate increased with temperature from 760°C to 890°C; further, an increase in temperature to 890°C had a significant effect. As shown in Figure 3, when hydrogen content is used as a reductant, there is a sharp increase in the reduction rate. The reduction rate increased with increasing temperature from 760 to 890; 64.19 to 91.07, respectively.

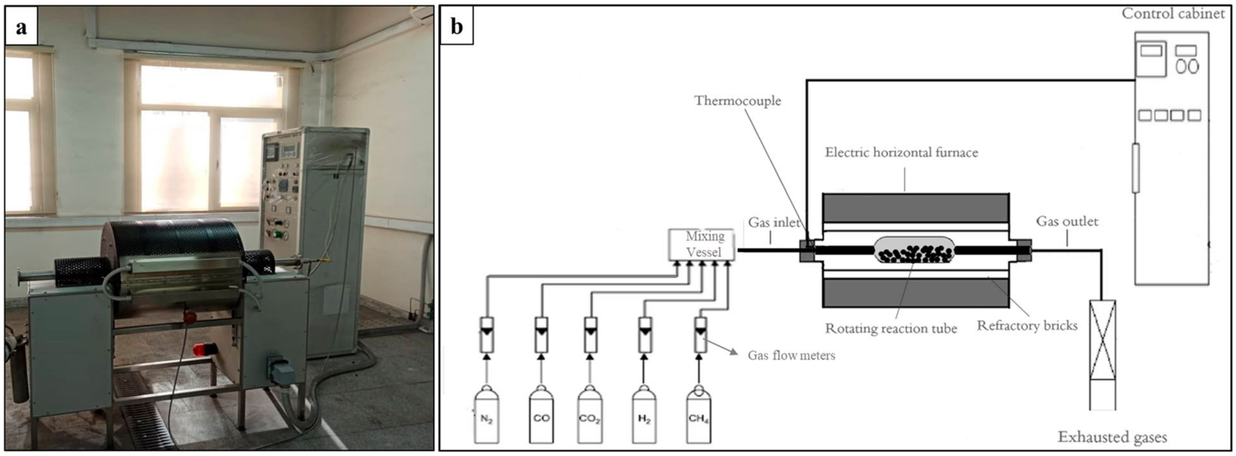


Figure 2. (a) Linder test equipment, (b) Schematic drawing of experimental apparatus for pellet reduction.

Table 3. Ilmenite pelletising results (expressed as average).

Material	Moisture	Drop Number	GCS (g/p)[a]	DCS (g/p)	CCS(kg/p)
Pellet	10/7	10/0	1120	8500	118

[a]- gram per pellet.

The relationship between the metallisation rate calculated by Equation (2) and reduction times at different atmospheres in 890°C is shown in Figure 4. The metallisation rate reaches its maximum value (93.64%) within 360 min at 890°C. Previous investigations on the reduction of ilmenite using hydrogen have suggested that the reduction reactions occur more quickly compared to using carbon monoxide. This could be attributed to the high reactivity of hydrogen, which enables it to effectively reduce iron compounds to metallic iron at a faster rate [21]. The reduction of ilmenite can be performed at lower temperatures using hydrogen as a reductant. However, the reduction reaction rate may be slower at lower temperatures owing to kinetic factors, including the activation energy required for the reaction to take place. Despite this, hydrogen is a highly reactive reducing agent, and its ability to effectively reduce iron compounds to metallic iron at a faster rate compared to other reducing agents such as carbon monoxide has been well established. The small size of hydrogen molecules is a key factor to its effectiveness as a reducing agent in ilmenite reduction. Because hydrogen molecules are small, they can easily penetrate the pores of the ilmenite ore, allowing them to react with the oxygen to produce metallic iron. Hence hydrogen's small size and high reactivity make it an ideal candidate for reduction ilmenite process. This makes hydrogen an attractive option for reducing ilmenite, particularly in cases where faster reduction rates are desired or when other reducing agents are not suitable owing to their chemical or physical properties.

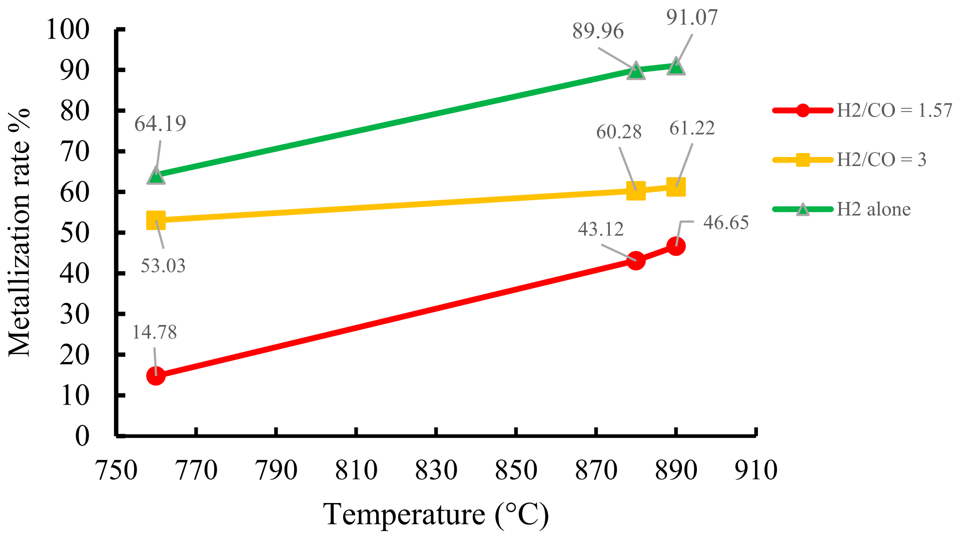


Figure 3. Effect of temperature on metallisation degree in different reduction atmosphere after 5 h.

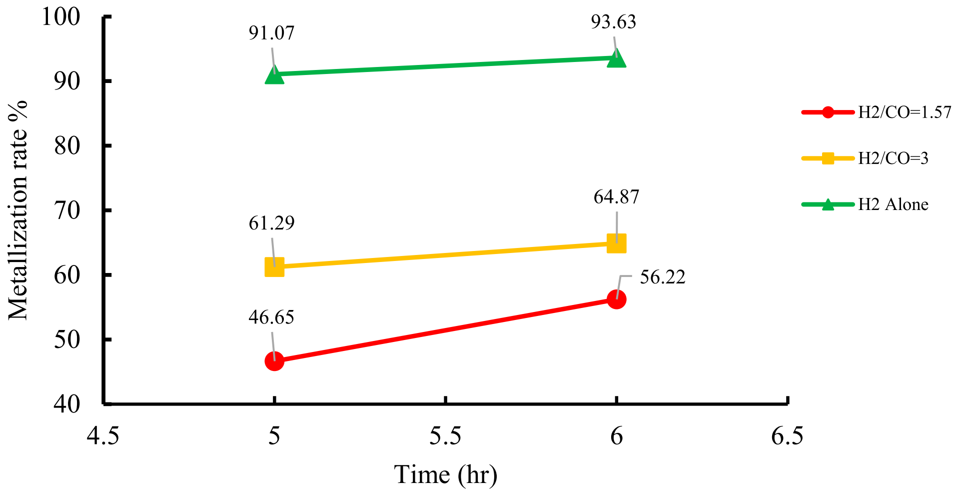


Figure 4. Effect of time on metallisation degree in different reduction atmosphere at 890°C.

3.2. Microscopic analysis

Optical Microscopy, SEM and EDX Mapping analysis was employed to study the reduction behaviour of H_2 pure and H_2/CO mixture gases, identify the phases and morphology of iron produced. The microstructures of the samples were analysed by OM and are shown in Figure 5. Samples from Experiments 3, 6, and 9 were selected as they underwent reduction at the highest temperature together with the highest gas content. The bright particles in the images are iron, the grey areas oxides phases and the black areas are pores and resins. Optical microscope images showed that increasing amounts of hydrogen content resulted in smaller more uniformly distributed metallic iron particles as the bright area developed.

To further investigate the formation and distribution of iron metal, SEM–EDX analysis was conducted. The SEM–EDX analysis of the H_2 pure reduction sample at 890°C and 360 min is shown in Figure 6 that Figure 6(a) and (b) display the distribution of iron (Fe) and titanium (Ti) in the morphology SEM image, respectively. Additionally, the X-ray *Kα* mappings of the primary elements are depicted, revealing their distribution. These six maps confirm the optical microscopic analyses. It is seen from the three maps that the products of Fe and Ti have good segregation. The micrographs reveal two distinct regions which appear as bright and dark grey phases. To identify the present phases, EDX was performed at regions marked in the SEM image of Figure 6. The results of EDX analysis show that region a consists of approximately 95.52% Fe and 0.56% Ti, so the bright phase is primarily the iron phase. Region b is made up of approximately 27.02% Fe and 38.08% Ti with little amounts of Oxygen, Aluminium, Silicon, and Calcium. Therefore, the dark grey phase is mainly titanium oxides.

3.3. Reduction mechanism

3.3.1. Chemical reaction of metallic iron

The possible chemical reactions for the reduction of iron oxides in ilmenite concentrate can be divided into several stages. In first step, ferric iron is reduced to ferrous iron and in the next step conversion of the

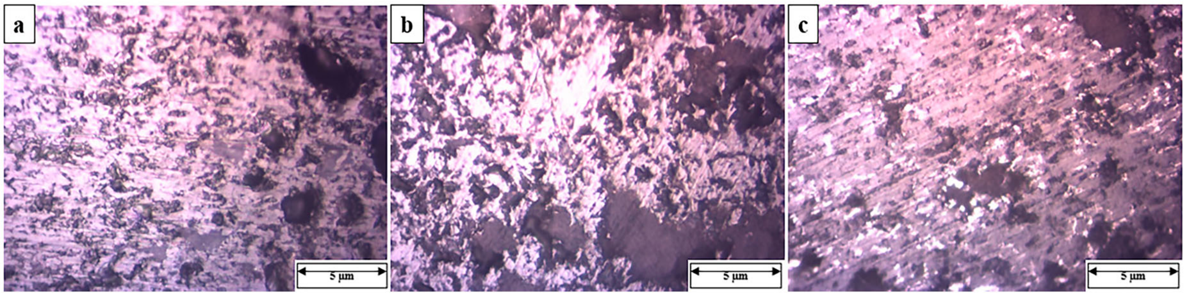
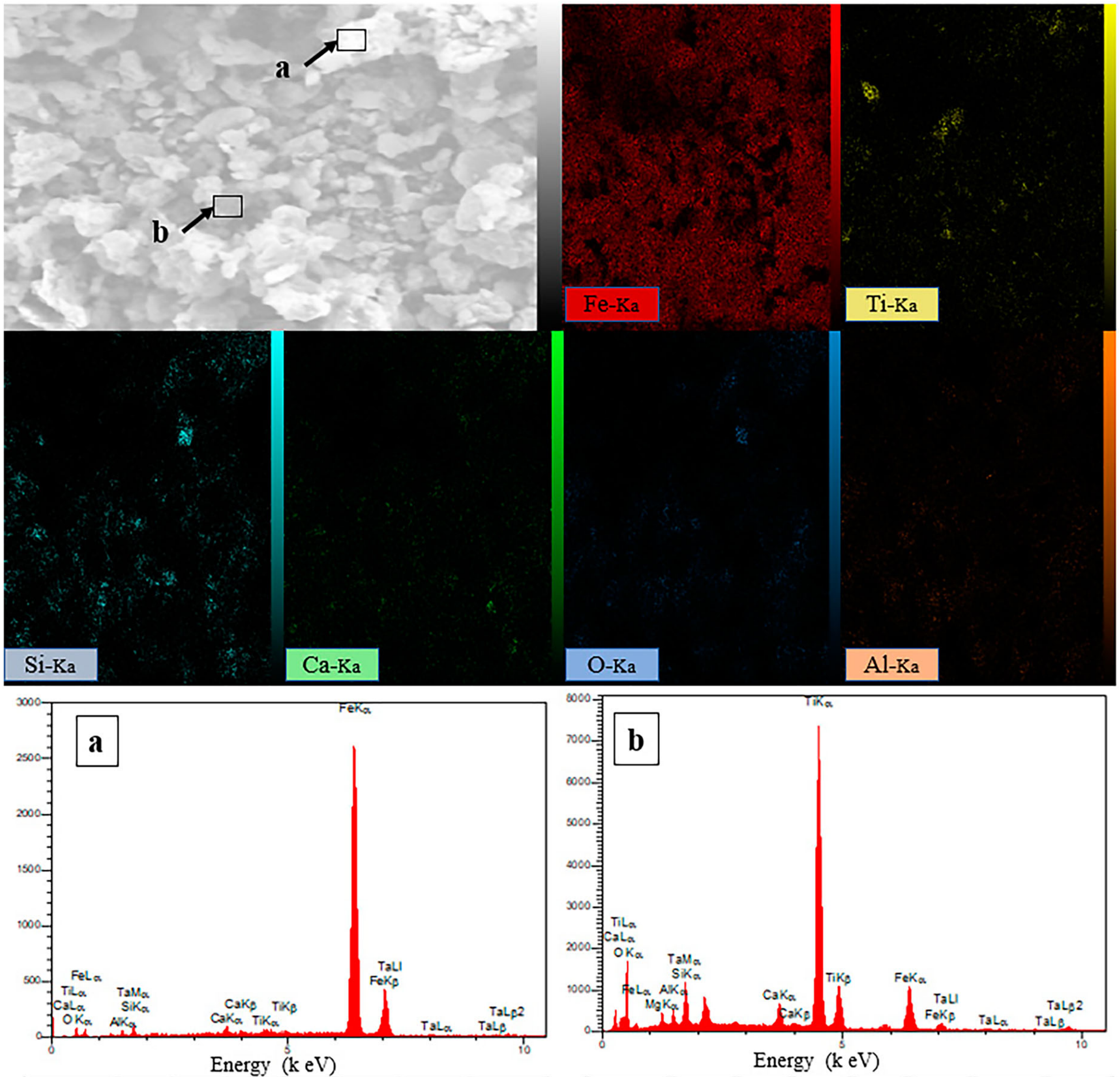


Figure 5. OM images of ilmenite concentrate reduced at 890°C: (a) H_2/CO: 1.57; (b) H_2/CO: 3 and (c) H_2 pure (bright area: metallic Iron, grey area: high titania phase, dark area: pores & resin).



Element	O	Al	Si	Ca	Ti	Fe	Other
% Wt.	1.3	0.23	0.54	0.59	0.56	95.52	1.2

Element	O	Al	Si	Ca	Ti	Fe	Other
% Wt.	26.79	0.68	0.25	2.39	38.08	27.02	4.79

Figure 6. SEM images of reduced sample at 890°C, Morphology, Fe, Ti, Si, Ca, O and Al *Ka* X-ray mapping and EDS results of regions (a) Fe, and (b) Ti.

ferrous iron to metallic iron. The reduction of iron oxides can be assumed to proceed according to below reactions [22]:

$$3Fe_2O_3(s) + H_2(CO)(g) \rightarrow 2Fe_3O_4(s) + H_2O(CO_2)(s) \quad (2)$$

$$Fe_3O_4(s) + H_2(CO)(g) \rightarrow 3FeO(s) + H_2O(CO_2)(g) \quad (3)$$

$$FeO(s) + H_2(CO)(g) \rightarrow Fe(s) + H_2O(CO_2)(g) \quad (4)$$

The ilmenite then reacts further with CO or H_2 between 670°C and 890°C as shown in reaction (5):

$$FeTiO_3(s) + H_2(CO)(g) \rightarrow TiO_2(s) + Fe(s) + H_2O\ (CO_2)(g) \quad (5)$$

3.3.2. Reduction with H_2/CO mixtures and pure H_2

By increasing the amounts of H_2/CO mixtures, the reduction rate of iron oxides increased and the degree of iron metallisation increased. When pure H_2 gas was used as the reducing agent, the reduction rate was greater than H_2/CO mixtures. Greater increasing reduction of ilmenite by hydrogen, it could have two reasons: At first, the equilibrium constant of the H_2 reducing gas increases with increasing temperature, because this is naturally endothermic reaction, causing to a greater reduction rate at higher temperatures and therefore driving force of the reaction increased. Second, at high temperature contributes to high mass transfer coefficient by increasing the movement of gas molecules and decreasing the resistance to diffusion. Moreover, according to Arrhenius equation, the rate of reaction constant increases and the reaction resistance decreases [20]. Thus, it can be concluded that the effect of temperature so important and have two results on the gas diffusion and resistance to chemical reactions can exceed the reduction potential.

3.4. Effect of pre-reduction on energy consumption and slag electrical conductivity

Main reaction of an ilmenite smelter can be written as follows:

$$FeO + C \rightarrow Fe + CO, \Delta H = +155.8\,\mathrm{kJ/mol} \quad (5)$$

Since the endothermic nature of reaction, it is necessary for the reaction to be held at higher temperature. If external heating source is interrupted, the reaction won't proceed. So, to keep the reaction moving forward it is necessary to maintain heating source. This means that higher Fe-metallisation degree (lower content of FeO) in smelting furnace results in lower energy needed for continuing the reaction. According to study conducted by outotec, 58% Fe-metallisation degree resulted in 27% reduction of specific power consumption. Since higher Fe-metallisation degree is obtained in the present work (up to 93.64%), H_2-pre-reduction is predicted to reduce specific power consumption even much higher than 27% recorded in outotec research. It needs other pilot for direct reduction and smelting with higher capacity to measure accurate power consumption reduction with higher metallisation degrees up to 93.64%. As mentioned above, pre-reduction results in lowering of specific energy consumption. So, at a constant power input in an electric arc furnace, the time needed for melting is reduced and therefore the slag has a lower metal iron content, so the total conductivity of slag is reduced and it makes possible for pre-reduced ilmenite pellets to be used in an AC open arc EAF. Further experimental investigation is needed to verify this theory.

4. Conclusions

This study investigated the microstructural changes and element distribution of a reduced ilmenite pellet using SEM and EDX techniques under different reduction conditions, including H_2/CO mixture and pure H_2 at varying temperatures and times. Based on the obtained results, the following conclusions were drawn:

(1) The degree of metallisation increases with an increase in temperature and reduction time.
(2) The iron metallisation rate can reach 93.64% with the hydrogen pure gas under the conditions of 890°C and 360 min.
(3) The EDS analysis of the samples indicates that the phase enriched in iron is depleted in titanium, confirming the high degree of metallisation of iron in the samples reduced by pure hydrogen gas.
(4) Higher metallisation degree (93.64%) reached in present work will result in lowering specific power consumption more than 27% for titanium slag production.
(5) Pre-reduced ilmenite pellets can be used in AC open arc EAF.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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