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RESEARCH PAPER

Two-Stage Reduction of Iron Scale with Intermediate Magnetic Separation for High-Purity Iron Powder Production

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ABSTRACT

A two-stage process is proposed for reducing iron-containing mill scale waste, involving an initial carbon-based reduction step, followed by hydrogen reduction, with magnetic separation in between. The purpose is to produce high-purity iron powder while optimizing hydrogen usage. The carbon stage, using powdered charcoal as a reducing agent, reduces most of the iron oxides economically at 900°C for 60 minutes. Intermediate magnetic separation, conducted using a permanent magnet to attract the iron-rich phase, removes non-magnetic impurities (e.g., silicon, calcium, tungsten oxides). The final hydrogen stage, carried out at 900°C for 60 minutes under flowing hydrogen gas, achieves nearly 100% iron purity as confirmed by EDS analysis. This process reduces hydrogen consumption and promotes the sustainable recycling of metallurgical waste for high-quality iron powder applications.

Keywords: Iron scale; Hydrogen reduction; Carbon reduction; Magnetic separation; Iron powder; Metallurgical waste recycling.

INTRODUCTION

Iron scale (mill scale) is a common by-product of steel manufacturing, formed as iron oxides (primarily FeO, Fe_2O_3, and Fe_3O_4) flake off from the surface of steel during hot rolling and other processing stages. This material contains a high percentage of iron oxides, along with various impurities, including silicon, calcium, and other trace elements that are picked up during the process. Recycling such technogenic waste has become increasingly important due to both environmental pressures and economic incentives [1–3]. Landfilling or stockpiling mill scale not only represents a loss of valuable iron resources but also poses environmental risks; thus, many steel plants seek ways to reuse or convert this waste into useful materials [1,3]. From an ecological perspective, reprocessing iron-rich waste contributes to resource efficiency and reduces the need for mining new iron ore, aligning with sustainability goals in the metallurgical industry [2,3].

Various techniques have been explored to recover iron from mill scale and similar steelmaking waste. Traditional approaches involve reducing the iron oxides either with carbon-based agents or with hydrogen gas, each method having distinct advantages and drawbacks. Carbon (typically in the form of coke, coal, or charcoal) is a cost-effective and readily available reductant that can reduce iron oxides by forming carbon monoxide (CO) and carbon dioxide (CO_2). Carbon-based reduction processes (as used in blast furnaces or direct reduction with syngas) are well-established and economical. Still, they introduce carbon into the product, generating CO_2 emissions and potentially leaving impurities in the iron product [4]. Hydrogen reduction, on the other hand, has gained attention as a cleaner alternative because its only reduction by-product is water vapour, avoiding direct carbon emissions [2]. Reduction of iron oxides with H_2 can achieve very high iron purity, as it does not carburize the metal and can potentially remove oxygen more completely under the right conditions [2,4]. However, pure hydrogen is relatively expensive and requires special handling (due to flammability and the need for infrastructure). In industrial practice, it often necessitates high temperatures or pressures to achieve fast kinetics [2].

To leverage the benefits of both reductants while mitigating their disadvantages, a two-stage reduction process has emerged as a promising strategy [4–6]. In a two-stage approach, a carbon-based reductant is used in the first stage to perform an initial reduction of the iron oxides, converting a substantial portion of the Fe oxides to metallic iron (and possibly intermediate oxides) while producing CO/CO_2. This step is cost-effective and can be fueled by relatively cheap carbon sources (even renewable sources like biomass charcoal can be used). After this partial reduction, an intermediate processing step, such as magnetic separation, can be introduced to remove the non-magnetic residue (impurities and unreduced oxides) from the magnetic iron-rich fraction [5]. The second stage then employs hydrogen gas to reduce any remaining iron oxides in the purified fraction to metallic iron, yielding a high-purity iron product. This combined method leverages carbon's economic and reducing properties for the bulk of the material, and hydrogen's superior reducing capability for final purification [6]. Notably, by removing impurities before the hydrogen stage, the consumption of hydrogen is minimized, as it is not wasted on oxidized gangue or inert compounds. The concept of two-stage reduction for waste iron oxides has been explored in previous studies [7], showing that such hybrid processes can significantly improve metal yield and purity compared to single-stage treatments.

In this paper, we present an experimental study on the two-stage reduction of iron scale with an intermediate magnetic separation step, aiming to produce high-purity iron powder. Moreover, detail the reduction procedure (carbon reduction followed by hydrogen reduction) and the effects of the intermediate magnetic separation on the product quality. The results are analyzed in terms of iron content and impurity removal, and we discuss the applicability of this method for industrial-scale operations, potential limitations, environmental impacts, and underlying thermodynamic considerations. The overall goal is to evaluate whether this two-step process can be a viable and more sustainable route for recycling iron-rich metallurgical waste into valuable iron powder.

MATERIAL AND METHODS

The iron scale collected from a steel processing facility was used as raw material for the experiments. Before treatment, the scale was characterized as primarily composed of iron oxides (FeO, Fe_2O_3, Fe_3O_4) with minor components such as SiO_2 and CaO, consistent with typical mill scale composition. The reduction experiments were carried out in two sequential stages. In the first stage (carbon reduction), the iron scale was thoroughly mixed with a carbonaceous reducing agent (powdered wood charcoal) and heated in a muffle furnace at approximately 900°C for 60 minutes. Charcoal acts as a source of carbon monoxide when heated, which reduces the iron oxides in the scale to metallic iron (Fe) and possibly intermediate iron oxides of lower valence. The furnace atmosphere was kept inert or minimally oxidative to ensure the carbon preferentially reacts with the scale rather than burning in air. After cooling to room temperature, the

partially reduced material (a friable iron sponge intermixed with slag and charcoal residues) was manually crushed to liberate metallic iron particles from the non-metallic byproducts. This crushed product was then subjected to magnetic separation using a strong permanent magnet. During this separation, the metallic iron-rich particles (being ferromagnetic) were attracted and collected, while the non-magnetic fraction containing residual oxides, slag, charcoal ash, and other impurities was removed.

In the second stage, the magnetically purified iron material was subjected to hydrogen reduction. The iron-rich powder from the first stage was placed in a tubular furnace for the hydrogen treatment. Pure hydrogen gas flowed through the furnace at 900°C for 60 minutes, enveloping the sample. The hydrogen reacts with any remaining iron oxides, reducing them to metallic iron and producing water vapour (H_2O) as the only gaseous by-product. The continuous flow of hydrogen helped to flush out the water vapour, driving the reduction to completion by maintaining a low partial pressure of H_2O in the reaction zone. This second reduction step was performed for the same duration and temperature as the carbon reduction to allow comparison and ensure thorough reduction. Two independent runs of the hydrogen reduction were conducted to verify reproducibility.

After each stage of reduction (and after the magnetic separation), the products were analyzed to determine their chemical composition and purity. Energy-dispersive X-ray spectroscopy (EDS) attached to a scanning electron microscope was used for semi-quantitative elemental analysis of the samples, focusing on the content of iron and common impurities (such as Si, Ca, etc.). Additionally, X-ray fluorescence (XRF) analysis was performed on selected samples to verify the results of the bulk chemical composition. Visual and structural examination of the reduced iron (sponge iron) was also carried out to observe the morphology of the particles after each stage. Photographs of the sponge iron and the separated fractions were taken to document the physical appearance changes throughout the process.

RESULTS AND DISCUSSION

Carbon Reduction and Magnetic Separation

Fig. 1 shows the appearance of the iron sponge after magnetic separation. The corresponding elemental composition is presented in **Table 1**, showing 99.5% Fe content.

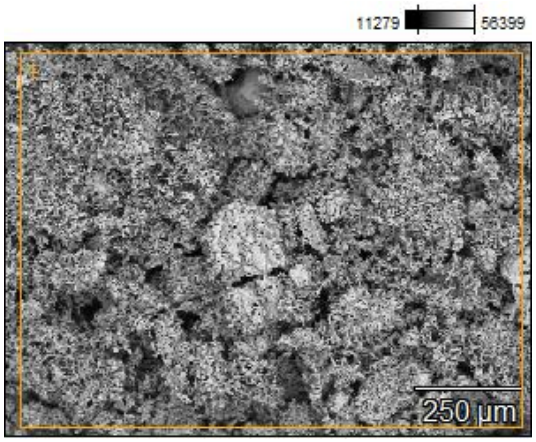

Fig. 1 Iron sponge obtained from the scale after magnetic separation

Table 1 Elemental composition after magnetic separation (Fe – 99.5%, Si 0.2%, Ca – 0.3%).–

Element	Si	Ca	Fe
Content, wt. %	0,2	0,3	99,5

By contrast, the sponge obtained without magnetic separation (**Fig. 2**) showed the presence of calcium, silicon, and an unexpected tungsten impurity (**Table 2**), likely originating from tool contamination or process equipment.

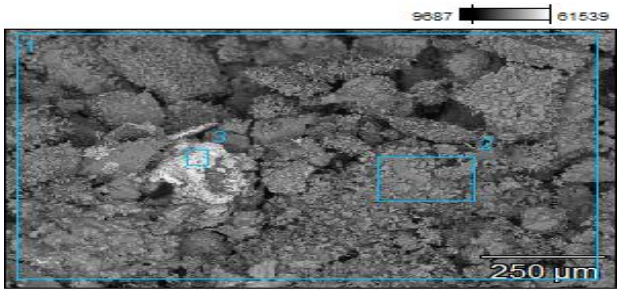

Fig. 2 Iron sponge without magnetic separation

Table 2 Elemental composition at different spots: Fe (9.6–90.4%), Ca (up to 12.3%), W (up to 55.4%).

Point	Element	O	Si	Ca	Fe	W
1	Content, wt. %	10,3	-	0,8	83,9	5,0
2	Content, wt. %	8,1	0,5	0,4	90,4	0,5
3	Content, wt. %	22,7	-	12,3	9,6	55,4

After the first-stage carbon reduction and subsequent magnetic separation, a metallic iron "sponge" was obtained from the iron scale. **Fig. 1** shows the appearance of this iron sponge product, which has a porous, metallic structure typical of partially reduced iron. Magnetic separation proved effective in isolating the iron-rich portion from the non-magnetic residue. Chemical analysis of the sponge after magnetic separation (**Table 1**) revealed that it contains approximately 99.5 wt% % Fe, indicating a very high iron content in the collected fraction. Only minor amounts of impurities were detected in this magnetically separated product—around 0.2 wt.% silicon (Si) and 0.3 wt.% calcium (Ca) are present, with other potential impurities below the detection limits of the EDS analysis. This result confirms that the combination of carbon reduction followed by magnetic separation can yield an iron product of high purity even before the hydrogen treatment.

The role of magnetic separation is crucial in this context. The initial carbon reduction likely converts most of the iron oxides to metallic iron or magnetite, which are ferromagnetic. At the same time, many impurities (e.g., silica, calcia, alumina) remain in oxidized forms that are non-magnetic. By applying a magnetic field to the crushed reduction product, the ferromagnetic iron and iron oxide particles are extracted, leaving behind non-magnetic waste.

In our experiments, this step dramatically improved the purity of the material destined for the second reduction stage. For comparison, a portion of the carbon-reduced sample was examined without performing the magnetic separation. The sponge iron obtained in that case (**Fig. 2**) showed visibly more slag and non-metallic inclusions. Elemental analysis at different spots on the un-separated sample (**Table 2**) indicated a significant presence of impurities: some regions of the sponge contained only 9.6 wt.% Fe (with the balance being other elements), and in certain areas, calcium reached up to 12.3% and an unexpected tungsten (W) impurity reached as high as 55.4%. The tungsten contamination is likely due to wear or abrasion of tungsten-containing components in the experimental setup (such as tungsten carbide from milling or furnace elements), which became mixed with the product when magnetic separation was not used to remove them. These findings underscore that without the intermediate separation; non-iron contaminants can significantly degrade the purity of the reduced iron. Magnetic separation thus serves as a critical purification step, ensuring that the feed into the subsequent hydrogen reduction stage is as iron-rich and impurity-free as possible [5].

Overall, the first stage achieved a substantial reduction of the scale: the iron content in the magnetically separated fraction was ~99.5%, meaning that most of the material in that fraction is iron (metallic or some magnetite), with only trace impurities remaining. This high Fe content after stage one is an excellent starting point for the second stage. It implies that most of the iron oxide in the scale was successfully reduced by carbon (charcoal) at 900°C, and that the majority of non-iron components were either left unreduced (and thus removed in the non-magnetic fraction) or were present in very small amounts. At this point, the

partially reduced iron sponge already meets the quality requirements for certain applications. However, to achieve the highest purity iron powder (suitable for demanding powder metallurgy applications), a second-stage hydrogen reduction is needed to eliminate the last remnants of oxygen and further increase the iron purity.

Hydrogen Reduction

Hydrogen reduction of the magnetically separated powder yielded a clean iron sponge, as shown in **Fig. 3**. Elemental analysis, as presented in **Table 3**, revealed a 100% Fe content, confirming the success of the method.

Fig. 3 Iron sponge after hydrogen reduction (two independent runs)

Table 3 Elemental composition: Fe – 100%

Element	Fe
Content, wt. %	100,0

The magnetically purified iron sponge from the first stage was subjected to hydrogen reduction in the second stage to remove any remaining oxygen and further improve its purity. After the hydrogen reduction (conducted at 900°C for 1 hour under flowing H_2), the resulting product was a clean, metallic iron powder with no visible non-metallic residues (**Fig. 3**). The two independent experimental runs of this hydrogen reduction yielded virtually identical results, demonstrating the consistency of the process. Elemental analysis of the hydrogen-reduced iron (**Table 3**) showed an iron content of approximately 100 wt.% – In other words, within the detection limits of the analytical methods used, the material is pure iron with no detectable impurities. This confirms that the hydrogen treatment successfully reduced any residual iron oxides (from the first stage) to elemental iron and did not introduce new contaminants.

Achieving ~100% Fe in the final product is a significant outcome, as it indicates complete metallization of the iron oxide content of the original scale. In comparison, if the scale were reduced in a single stage by carbon alone, it would be challenging to achieve such a high degree of purity, as some impurities and unreduced oxides would likely remain trapped with the iron, as evidenced by the analysis of the unseparated sample. Likewise, a single-stage hydrogen reduction of raw scale might achieve high purity. Still, it would require a large amount of hydrogen and careful control of conditions to ensure impurities are not simply included in the reduced mass. By splitting the process into two stages, we capitalised on the strengths of each reductant: the carbon reduction removed the bulk of oxygen and separated most impurities at a low cost, and the hydrogen reduction polished the product to ultra-high purity [4].

Hydrogen is known to be a very effective reducing agent for iron oxides, especially at elevated temperatures, due to its ability to penetrate porous solids and reduce oxides without leaving any solid residues [2,4]. Furthermore, hydrogen reduction avoids carbon contamination of the product (no carbon is dissolved in the iron, unlike in a carbon thermal reduction, where some carbon can dissolve or deposit). In our experiments, because the first stage had already produced a porous iron sponge, the hydrogen could easily permeate the material and react with any remaining FeO or Fe_3O_4. The high porosity and large surface area of the sponge iron accelerated the reduction kinetics in the second stage, allowing the reaction to complete within 60 minutes.

It is also worth noting that the hydrogen consumption in this two-stage process is significantly lower than it would be if hydrogen alone were used to reduce the entire initial oxide mass. In our method, hydrogen was only needed to reduce the relatively small fraction of oxygen remaining after the carbon reduction and to refine the iron product. If one were to reduce iron scale directly with hydrogen from the outset, the hydrogen requirements would be much higher, as it would have to carry out the entire reduction of Fe_2O_3/Fe_3O_4 to Fe and potentially reduce or carry away other oxides present in the scale. By pre-reducing carbon (which is inexpensive and can be derived from renewable sources, such as biomass charcoal), the process conserves hydrogen. This not only cuts costs but also reduces the energy footprint associated with producing hydrogen (especially if using electrolytic or other energy-intensive hydrogen sources). Thus, the two-stage approach offers a more economical use of hydrogen while still delivering a high-purity product [6]. The implications of this hydrogen saving are important for industrial feasibility, given that hydrogen gas, especially if produced as "green hydrogen," can be a limited and costly resource.

Industrial Applicability and Environmental Aspects

The positive results from this two-stage reduction process suggest that it could be scaled up for industrial use in the recycling of iron-rich waste materials. In a steel plant or a dedicated recycling facility, a similar two-step process could be implemented to handle large volumes of mill scale or other iron-bearing residues. For instance, the first stage of carbon reduction could be carried out in a rotary kiln or a shaft furnace using a cheap carbon source (such as coke breeze, coal, or biomass charcoal), which can continuously reduce the oxide waste. The partially reduced material could then be passed through a magnetic separator (either a wet drum separator or a high-intensity magnetic separator, depending on whether the material is cooled or kept hot) to remove the non-magnetic slag. The second stage could involve a fluidized bed or a moving-bed reactor where the purified iron material is exposed to hydrogen gas at high temperatures. These unit operations (reduction furnaces, magnetic separators, hydrogen reduction reactors) are all within the realm of existing industrial technology, indicating that scaling up the process is technically feasible.

Previous studies on recycling steelmaking waste [1,3] have utilised processes such as direct reduction in furnaces or reintroduction of waste oxides into iron ore sintering/pelletizing. Still, these methods sometimes struggle with impurity removal. Our two-stage process, by design, specifically addresses the impurity issue, making the recycled iron product suitable for high-quality applications rather than just being reused as a lower-grade feed. Lebedev [6] notes that developing sustainable technologies for producing iron powder is a key area of interest in metallurgy, and the approach demonstrated here aligns with that direction by transforming waste into a valuable powder product.

Despite its promise, there are potential limitations and challenges in implementing this method industrially. One concern is the energy requirement: both stages operate at around 900°C, which means significant energy input (likely from combustion or electrical heating) is necessary. However, the two-stage nature might allow some energy integration; for example, the exothermic reaction of carbon oxidation in the first stage could supply part of the heat for that stage or preheat the materials. Process optimization would be necessary to minimize energy consumption per ton of scale treated. Another consideration is the supply and cost of hydrogen for the second stage. Although the process uses less hydrogen than a single-step hydrogen reduction, industrial-scale volumes of hydrogen could still be considerable. Viability may depend on the availability of low-cost hydrogen (potentially from excess industrial hydrogen or future green hydrogen production). Safety measures for handling hydrogen at a large scale (explosion prevention, proper ventilation, monitoring) would also be essential. Additionally, the multi-step process requires the material to be handled and transferred between stages, which can introduce operational complexity. Engineering the system to carry material from a carbothermic reduction unit to a magnetic separator and then to a hydrogen reduction unit (possibly under inert conditions to avoid re-oxidation during transfer) will require careful design. Nonetheless, these challenges are engineering issues that can be addressed with existing technology and proper process design.

From an environmental standpoint, the two-stage reduction route offers several advantages. First, it directly recycles a waste product (mill scale) into pure iron, thereby reducing the need for mining fresh iron ore and minimizing waste disposal. This contributes to a circular economy in the steel industry by recovering valuable iron units that would otherwise be discarded [3]. Second, by using a combination of carbon and hydrogen, the process can achieve lower overall greenhouse gas emissions compared to a purely carbon-based reduction. In the initial stage, if the carbon reductant is derived from biomass (as was the

case with wood charcoal in our experiments), the net CO_2 emissions can be partially offset by the renewable nature of the carbon source. The second stage uses hydrogen, which produces no CO_2 directly in the reduction reaction. If the hydrogen is sourced from renewable energy (green hydrogen), the carbon footprint of the reduction process can be very low. Even when using fossil-derived hydrogen, the efficiency gains and hydrogen savings in this two-stage process result in fewer fossil resources being used compared to all-hydrogen processes. Moreover, the elimination of impurities like sulfur (if present) during magnetic separation results in fewer pollutants in the product and potentially less environmental impact when the iron powder is later used or processed. Overall, this method aligns with the principles of cleaner production [3] and addresses both waste utilisation and emission reduction goals.

A brief consideration of the thermodynamics and kinetics provides further insight into why the two-stage process is effective. At around 900°C, the Gibbs free energy change for the reduction of iron oxides by carbon (through CO) and by hydrogen is both favourable [8]. Hydrogen is a highly potent reducing agent for iron oxides at high temperatures; for example, the reaction $Fe_3O_4 + 4H_2 \rightarrow 3Fe + 4H_2O$ is thermodynamically spontaneous at these temperatures, driving the conversion of the oxide to metal. Meanwhile, carbon (or CO) can also reduce Fe_3O_4 to Fe, although it produces CO_2 (or CO) as byproducts. The first stage likely reduces the iron oxides to a mixture of Fe and FeO/Fe_3O_4; completing the reduction to Fe requires either a stronger reducing environment or removal of the oxygen-bearing species. Hydrogen provides a stronger reducing atmosphere in the second stage. Because the material is porous and free of most gangue after magnetic separation, the remaining iron oxides are easily accessible to H_2. Kinetically, the porous structure of the partially reduced sponge iron shortens the diffusion distances for gaseous H_2 to react with internal oxide surfaces, leading to rapid reduction rates [9-11]. This explains why the hydrogen stage can fully reduce the material in 60 minutes. Thus, thermodynamically and kinetically, the strategy of using carbon first and hydrogen second is well-founded: the carbon stage takes advantage of inexpensive fuel and initial easy oxide reduction, and the hydrogen stage capitalizes on a highly favourable reaction to finalize the metallization.

CONCLUSION

The experimental investigation of a two-stage reduction process (carbon reduction followed by hydrogen reduction, with intermediate magnetic separation) for iron-containing scale has demonstrated its effectiveness in producing high-purity iron powder. Key outcomes of this study include:

- High Iron Purity: The final iron powder attained an iron content of approximately 100 wt.%, meaning essentially all iron oxides were converted to metallic iron by the end of the process.
- Impurity Removal: The intermediate magnetic separation and two-step reduction approach led to a significant reduction of impurities such as silicon, calcium, and tungsten in the iron product. Elements like W, which were introduced as contaminants, were effectively removed, yielding a cleaner iron powder than would be possible with a single-step reduction.
- Reduced Hydrogen Usage: By performing the initial reduction with carbon, the overall consumption of hydrogen gas was greatly decreased compared to a hypothetical one-stage hydrogen process. This makes the process more cost-effective and energy-efficient while still delivering a high-purity product.

The combination of these outcomes shows that the two-stage process is a viable method for recycling mill scale and similar iron-rich waste into valuable iron powder. The high purity of the product and the efficient use of resources suggest that this method could be implemented in powder metallurgy industries or steel plants aiming to improve sustainability. The approach addresses common challenges in waste recycling by removing deleterious impurities and limiting the use of expensive reducing agents. Overall, this work contributes to the development of sustainable metallurgical processes by demonstrating a practical route to transform industrial waste into a high-quality resource.

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