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Computational reacting flow models for the pre-reduction of lumpy Nchwaning manganese ore with hydrogen

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Solid-state pre-reduction of manganese ores with hydrogen presents many potential advantages that include reduction of greenhouse gas emissions and lower energy consumption of the downstream smelting step. Before designing a pre-reduction reactor, it is crucial to investigate and understand the process kinetics and their influence on the overall pre-reduction reactor performance. Computational fluid dynamics (CFD) reacting flow models are used to predict the influence of kinetics, geometry and flow field on the chemical reaction rates. The current work employs the CFD models to predict the influence of temperature, flow field and kinetics on the degree of manganese pre-reduction with hydrogen. The models allow for the determination of the optimum reduction temperature and reduction time.

Keywords: CFD, kinetics, solid-state reduction, manganese, hydrogen

INTRODUCTION

Manganese has various industrial applications that include alloying in steelmaking, fertiliser production, batteries, glass making and in paints etc., (Sahoo *et al.*, 2001). Ferromanganese is produced by smelting of manganese ore in the submerged arc furnace utilising carbon as a reductant. The process is energy intensive and results in greenhouse gas emissions. Therefore, there is need to investigate alternative reductants aimed at minimising the CO_2 emissions and reducing the process energy consumption. Hydrogen is not only a source of clean energy, but is also a promising alternative reducing agent for metal production. Successful design of optimised industrial pre-reduction technologies based on hydrogen will accelerate the de-carbonisation of metal production processes.

The integrated pre-reduction of manganese with hydrogen with aluminothermic reduction in the downstream smelting step presents an attractive alternative towards decarbonising the manganese industry (Safarian, 2022). Moreover, while literature highlights the potential of using hydrogen for direct reduction in the downstream smelting step as a promising path to decarbonisation, this paper concentrates on the pre-reduction step. The pre-reduction of manganese with hydrogen presents many potential advantages such as the reduced energy consumption in the downstream smelting step and the reduction of greenhouse gas emissions. The reduction of energy consumption in the downstream step is attributed to the decomposition of carbonates present in the manganese ore during pre-reduction. The decomposition of carbonates is an endothermic process, thus it increases the energy consumption during smelting. Hydrogen as a reductant in the pre-reduction step results in water as the by-product, hence reducing the CO_2 emissions that are inherent in carbothermic reduction. The aluminothermic reduction in the smelting process does not result in the CO_2 emissions like is the case with the carbothermic smelting.

Studies on the reduction of manganese ore by hydrogen have been carried out by several authors. The reduction rate of low grade manganese ore briquette under hydrogen reducing atmosphere was investigated by (El-gawad *et al.*, 2014). The results from their study indicated that the reduction rate increased with increasing hydrogen flow rate. The increase in reduction rate can be attributed to the increased gas diffusion across the boundary layer, and increased hydrogen adsorption at high flow rates. The degree and rate of manganese ore reduction increased with increasing temperature due to increased rate of desorption and mass transport by diffusion. In addition, the dependency of reaction rates on temperature means that the reaction rates are controlled by kinetics. Since the gas flowrate also affects the reaction rates, computational fluid dynamics (CFD) models can help predict the transition from kinetic regime to transport regime.

The influence of hydrogen on the pre-reduction of carbonate-rich United Manganese of Kalahari (UMK) was investigated by Davies *et al.*, (2023). The results from their study revealed that using hydrogen as a reductant for the carbonate containing manganese ores accelerates the decomposition of carbonates, reduces the iron oxide contained in the ore to metallic iron and reduces the higher manganese oxides to MnO. It is evident that hydrogen pre-reduction contributes to energy consumption reduction in the downstream smelting because it completes the endothermic carbonate decomposition. Compared to pre-reduction in CO/CO_2 atmosphere, the introduction of hydrogen resulted in an improved rate and degree of reduction. This can be attributed to hydrogen's small molecular size which makes it easier to diffuse through the pores.

One of the earlier works on the pre-reduction of manganese with hydrogen investigated the reduction of synthetic pyrolusite at varying partial pressures and temperatures (Barner *et al.,* 1968). The authors conducted experiments in a vertical-tube differential flow reactor and studied the reduction of single porous pellets and a small bed of particles under hydrogen atmosphere. The equilibrium considerations predict that the reduction of MnO_2 by hydrogen end at MnO (Barner, 1967), because even at temperatures above the melting point of Mn, P_{H_2O}/P_{H_2} must be maintained below $1x10^{-4}$ to render the reduction to metal thermodynamically feasible. The findings from their study indicate that below 250ºC, the reduction rates are controlled by reaction kinetics. However, above 250ºC, the diffusion resistance starts to manifest as the Mn_3O_4 is formed and this phase has low porosity. The low porosity limits the access of reducing gases to the reaction sites. Finally, above 325ºC, the overall reaction rate was found to be controlled by chemical reaction kinetics.

The reduction of pure manganese oxides by H_2-Ar (20 vol% H_2 and 80 vol %) gas and CH_4-H_2-Ar (10 vol% CH_4, 20 vol% H_2 and 70 vol% Ar) gas mixtures was investigated by (Ostrovski et al., 2004). Their findings indicate that the reduction of pure MnO_2 by H_2-Ar starts at 305-320ºC and is completely reduced to MnO at 610-620ºC. On the other hand, the addition of CH_4 to the gas mixture resulted in carbon deposition when CH_4 exceeds 15 vol %. Carbon deposition has a retarding effect on reduction since it inhibits mass transfer of reducing gases.

Larssen *et al.*, (2022) Investigated the influence of hydrogen, and water-gas shift reaction on the pre-reduction of Comilog and Nchwaning manganese ores in the $CO-CO_2$ atmosphere. The gas compositions investigated are presented in Table 1. The pre-reduction of manganese ore is controlled by kinetics as opposed to thermodynamics. The authors found that in the case of Comilog ore, hydrogen resulted in higher reaction rates compared to water vapour (which produces hydrogen through water-gas shift reaction) and this was observed from reduction temperatures from 200ºC. On the other hand, for the Nchwaning ore, addition of hydrogen or water vapour during the heating of the ore from 25 to 600ºC did not promote reduction. However, at temperatures above 700ºC, hydrogen addition promoted the reduction of Nchwaning ore.

Table 1. Gas compositions investigated by Larssen et al., *(2022)*

	CO [L/min]	CO_2 [L/min]	H_2 [L/min]	H_2O [L/min]	Total [L/min]	Log($p(O_2)$) at T = 800°C
1a ($p(O_2)_1$)	2.00	2.00	0.00	0.00	4.00	- 18.4
1b ($p(O_2)_1$)	1.75	1.75	0.50	0.00	4.00	-18.4
1c ($p(O_2)_1$)	2.24	1.25	0.00	0.50	3.98	-18.4
2a ($p(O_2)_2$)	3.20	0.80	0.00	0.00	4.00	-19.6
2b ($p(O_2)_2$)	2.80	0.70	0.50	0.00	4.00	-19.6

The kinetics and mechanism for oxide reduction using molecular hydrogen was described by (Rukini *et al.*, 2022), who states that lattice oxygen in the oxide is removed, and the process is driven by H_2O removal. The dissociation energy of the hydrogen molecules facilitates the adsorption of hydrogen. The transformation of molecular hydrogen into the atomic state allows for diffusion into the metal oxide structure and subsequently the metal-oxide bonds break (Kung, 1989). This is followed by the formation of hydroxyl group and metal hydride. The metal hydride is volatile and as a result it releases hydrogen to form water with hydroxyl followed by water vapour desorption (Rukini *et al.*, 2022).

The CFD reacting flow models for gas solid systems can benefit the design and optimisation of pre-reduction reactors. This is because CFD models are able to predict the influence of flow field, geometry, particle size and temperature on reactor performance. The validated CFD models could also help reduce the number of experiments since only the optimum conditions predicted by the models can be investigated. The accuracy of CFD models depends on the physics incorporated and the kinetic data from experimental measurements. The kinetic parameters determination of carbonate rich ores necessitate that the ore is calcined before pre-reduction. This is because the decomposition of carbonates happens simultaneously with pre-reduction and results in the sample weight loss, and it would be a complex task to decouple the mass loss due to the reduction reactions and the mass loss from decomposition.

The objective of the current study is to use computational reacting flow models to predict the pre-reduction of Nchwaning manganese ore with hydrogen. The CFD models will help predict parameters such as the optimum reduction temperature and reduction time. One of the problems that confront the pre-reduction of manganese ores is the poor utilisation of hydrogen; therefore, the accurate prediction of the optimum flowrate will benefit the design of pre-reduction processes.

Reaction kinetics
The manganese higher oxides reduce to MnO in hydrogen atmosphere and the reaction mechanism is presented in Equations 1 to 3. The reduction of manganese ore with hydrogen does not proceed beyond MnO in part due to the low equilibrium constant for the reaction even at elevated temperatures. Metallisation is achieved in the smelting process where carbothermic reduction of MnO from the slag liquid takes place (Safarian *et al.*, 2006) .The iron oxide part of the ore is reduced according to the reaction mechanism presented in Equations 4 to 6. Given that the reaction system constitutes multi-step reactions, models such as the shrinking core model are not applicable.

$$2\,MnO_2 + H_2 \rightarrow Mn_2O_3 + H_2O \qquad\qquad [1]$$

$$3\,Mn_2O_3 + H_2 \rightarrow 2\,Mn_3O_4 + H_2O \qquad\qquad [2]$$

$$Mn_3O_4 + H_2 \rightarrow 3\,MnO + H_2O \qquad\qquad [3]$$

$$3\ Fe_2O_3 + H_2 \rightarrow 2\ Fe_3O_4 + H_2O \qquad\qquad [4]$$

$$Fe_3O_4 + H_2 \rightarrow 3\ FeO + H_2O \qquad\qquad [5]$$

$$FeO + H_2 \rightarrow Fe + H_2O \qquad\qquad \{6\}$$

Numerical method

The lumpy manganese ore particles in a thermogravimetric furnace are modelled as a fixed bed of particles and the flow in and around the fixed bed is resolved. In order to make the implementation easy, each computational cell is assumed to lie either entirely outside or inside the porous medium. The fixed bed is described as a collection of tensor and scalar properties. These properties include porosity, thermal conductivity and viscous resistance.

When the reduction reactions take place, the transformation from one phase to the other results in density changes and this leads to changes in porosity. Porosity is modelled according to Equation 7. The structural changes in the intermediates oxides may result in porosity changes which will affect the transport of reducing gases into the reaction sites. It is crucial that mathematical models capture this phenomenon to aid the design and optimisation of pre-reduction reactors.

$$\frac{d}{dt}\epsilon = -(1-\epsilon)\sum_{i=1}^{N}\frac{1}{\rho_i^s}R_i \qquad\qquad [7]$$

Where ϵ is porosity, t is the reaction time, ρ is density, R is the reaction rate and N is the number of species.

Governing equations

The governing equations for the gas phase are presented in Equations 8-11. The equations describe momentum, continuity, species conservation and energy balance respectively. The heterogeneous gas solid reactions are presented by the source term R, which is also used to effect the coupling between the solid and gas phase.

$$\frac{\partial}{\partial t}\rho^G u + \nabla.(\rho^G uu) + \varepsilon\nabla P - \nabla.(\mu_{eff}\nabla u) - \rho^G g = -\mu_{eff}\ D.u - F.u \qquad\qquad [8]$$

$$\frac{\partial}{\partial t}\varepsilon\rho^G + \nabla.(\rho^G u) = (1-\varepsilon)\sum_i R_i^{Reduction} \qquad\qquad [9]$$

$$\frac{\partial}{\partial t}\varepsilon\rho^G y^G + \nabla(\rho^G u y^G) - \nabla.(\varepsilon\rho^G D_{eff}\nabla Y^G) = \varepsilon w^G + (1-\varepsilon)R_i^{Reduction} \qquad\qquad [10]$$

$$\frac{\partial(\varepsilon\rho^G Cp^G T^G)}{\partial t} + \nabla.(\rho^G Cp^G T^G u) - \nabla.\nabla(\varepsilon k_{eff}^G T^G) = -\varepsilon\sum_i w_i h_{f,k}^0 - h_{conv}S_{Av}(T^G - T^S) +$$
$$+(1-\varepsilon)T^G\sum_i Cp_i\ R_i^{Reduction} + S^{G,Radiation} \qquad\qquad [11]$$

In the Equations (8)–(11), C_p is the specific heat, k_{eff} is the gas thermal conductivity, D denotes Darcy's resistance to flow, G is the gas phase, ϵ is porosity, D_{eff} is the effective diffusion coefficient, ρ denotes density, u is the gas velocity and μ_{eff} is the dynamic viscosity. The subscript *eff* denotes the contributions of correlations resulting from volume averaging.

Geometry and Meshing

A 2D axisymmetric geometry for a reactor height of 45 cm and radius of 5.8 cm was used as shown in Figure 1 (b) and the corresponding mesh in Figure 1 (c). The reactor used in the CFD model is an experimental Thermogravimetric Analysis (TGA) presented in Figure 1 (a). The manganese lumps were packed into the reactor from 0 cm to 4.5 cm. A uniform porosity was specified for a packed bed of pellets and this follows an example of porosity as observed in entities such as sand. The CFD model which

defines porosity as a field inside the main computational domain developed by (Jan Źuk *et al.*, 2022) was used. In this case, a special type of immersed boundary method named 'fictitious domain' is used to define manganese ore using its physical characteristics such as viscous resistance, thermal conductivity, density and porosity etc.
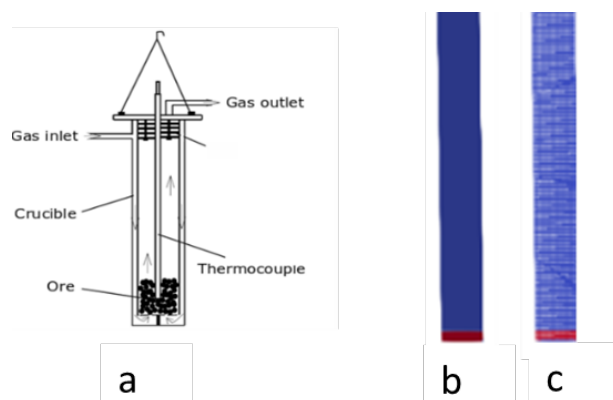

Figure 1. Geometry and meshing of a 2D axisymmetric representation of a TGA furnace.

Mesh sensitivity analysis

Mesh sensitivity analysis was done to determine optimum mesh size, the results of which are not resolution-dependent. The Richardson interpolation method for mesh independence studies is used because it ensures that the process is not trial error and helps determine the optimum grid size after a few iterations. The results in Figure 2 and Figure 3 reveal that Grid 2 is the optimum mesh size as at this grid size the species mass fraction distribution does not change with the number of cells. The number of cells in Grid 1, Grid 2 and Grid 3 is 3750, 7500 and 11250 respectively.
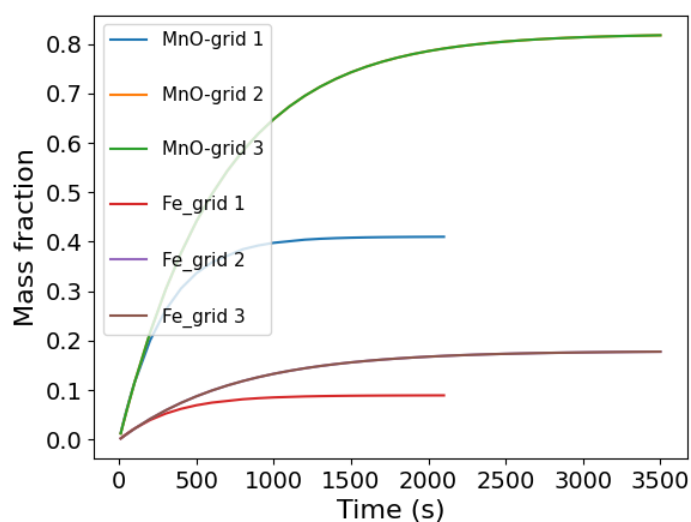
Figure 2. MnO and Fe mass fraction profiles at different mesh resolutions.
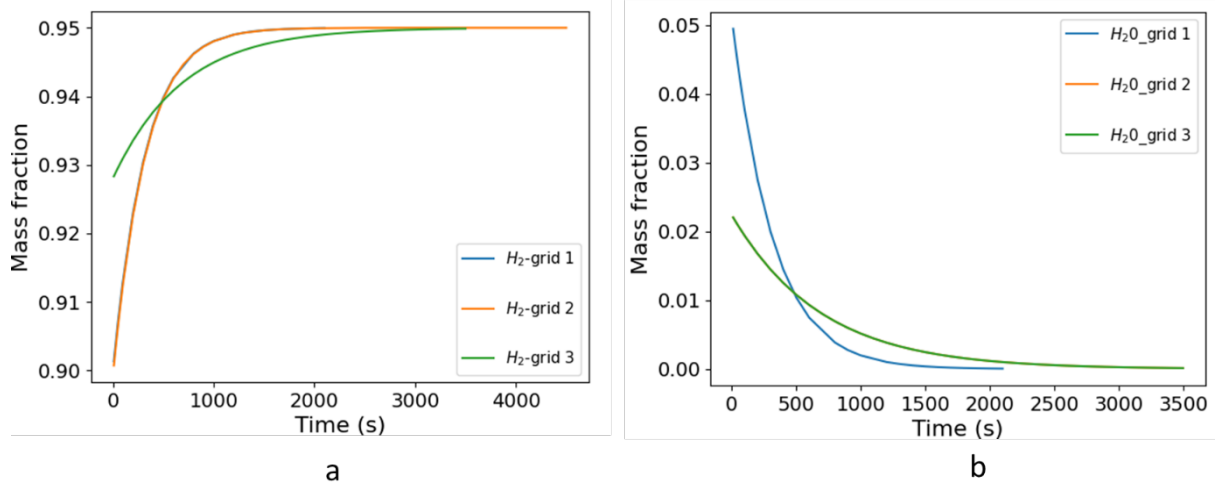
Figure 3. H₂ and H₂O mass fraction profiles at different mesh resolutions.

Results and discussions

The pre-reduction of manganese by hydrogen results in the metallisation of the iron from iron oxide part of the ore and formation of MnO from higher manganese oxides. The process is influenced by several factors such as temperature, reduction time and gas flowrate. The influence of these parameters on pre-reduction of Nchwaning manganese ore were investigated using the CFD model. The global reactions with the associated global kinetic data were used as opposed to the multi-step reactions with intermediate oxides. The global reactions were used because mass loss data from the TGA experimental measurements only allowed the determination of global kinetic data. A bed of particles after 10 and 1500 seconds of reduction at 700°C is shown in Figure 4 and Figure 5. As observed, at 1200 seconds, pre-reduction is complete and Fe_2O_3 and Mn_2O_3 are converted to Fe and MnO respectively. The corresponding mass fraction profiles are presented in Figure 6.
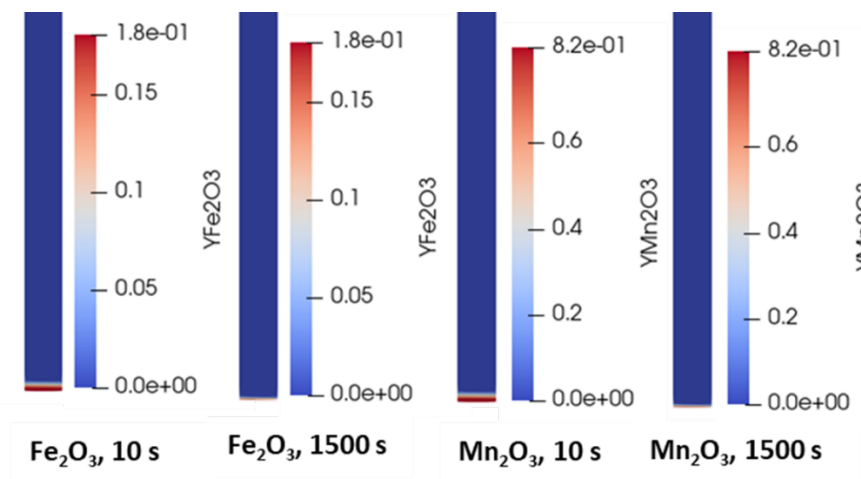
Figure 4. Fe₂O₃ and Mn₂O₃ mass fraction at 10 and 1500 seconds exposure time.
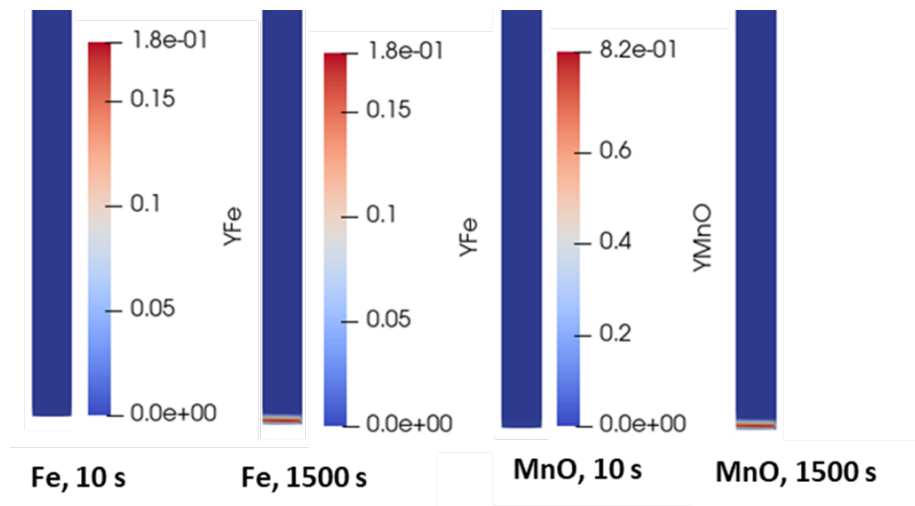
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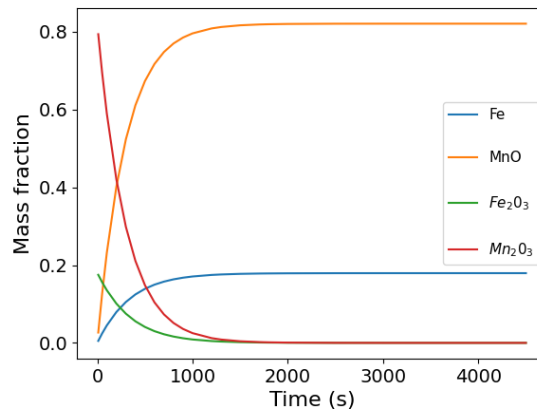*Figure 5. Fe and MnO mass fraction at 10 and 1500 seconds exposure time.*

Figure 6. Species mass fractions for pre-reduction with hydrogen at 700ºC and 4 L/min.

The influence on temperature of manganese reactivity

Temperature influences the kinetics of pre-reduction of Nchwaning manganese ore by hydrogen. The rate of reduction increases with increasing temperature as observed in Figure 7. At 600ºC, the rate of reduction is slow as evidenced by the consumption rate of Fe_2O_3 and Mn_2O_3, even at 3000 seconds, the reduction is not complete. On the other hand, at 700 to 900ºC, the rate of reduction is faster and reduction is complete at 2000 seconds of reduction. The results show the dependency of reaction rates on temperature. However, the CFD model does not account for sintering; as a result the effects of sintering on the reactivity of Nchwaning ore at high temperatures is not captured by the model. The microscopic examination by (Sarkar *et al.*, 2023) reveal that above 700ºC, the pre-reduced Nchwaning manganese ore sinters resulting in low porosity and high density.
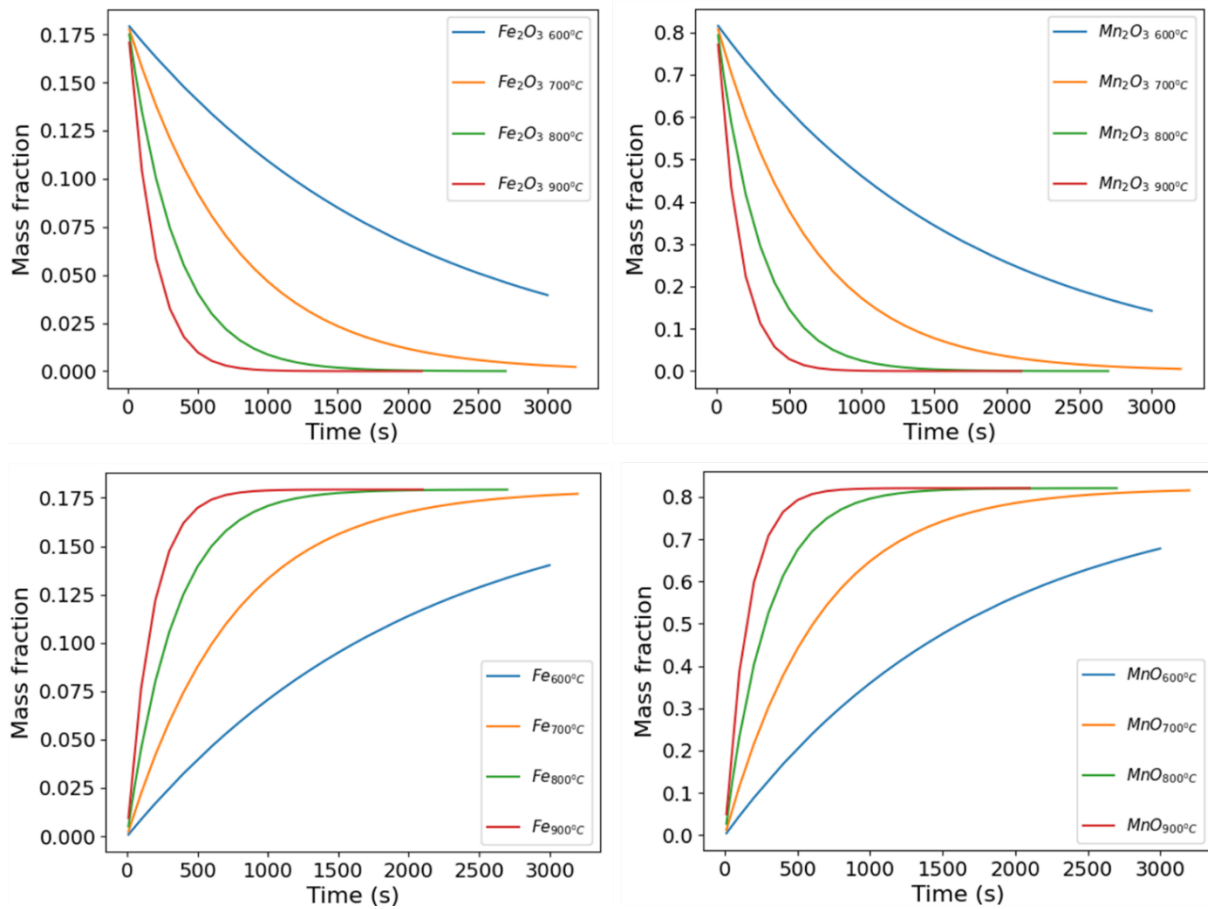
Figure 7. Species mass fraction profiles at various temperatures.

Influence of porosity manganese reactivity

Surface area affects the reactivity of heterogeneous reactions, thus higher porosity results in increased reactivity. Although high porosity results in increased rate of reduction of manganese ores under hydrogen reducing atmosphere, it results in decreased thermal strength. Furthermore, high porosity may result in the ore cracking when it is rapidly heated due to high pressure from the evaporation of moisture because high porosity ores absorb moisture (Davies *et al.*, 2023). The influence of porosity on the ores reactivity was studied with CFD reacting flow model and the results are as presented in Figure 8. As observed, the reaction rates are high at high porosity and Fe_2O_3 and Mn_2O_3 are consumed more rapidly at a high porosity.
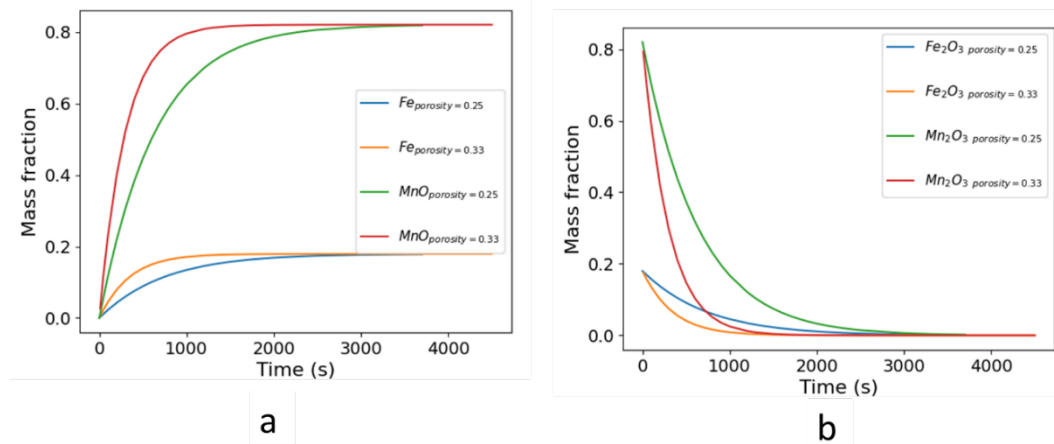
Figure 8. Species mass fraction profiles at varying porosity of manganese ore.

Comparison with experimental results

The CFD model considered a Thermogravimetric Analysis (TGA) reactor of the same height (45 cm) and diameter (5.8 cm) with the TGA used in the experimental measurements. In addition, the model used a bed height (0.45 cm) of manganese ore particles and hydrogen gas flowrate (4 NL/min) similar to the experimental test conditions. The model results indicate that the degree of reduction increases with increasing temperature and determines the time required for reduction to reach completion. It is crucial to compare the model results with experimental results to assess the accuracy and generality of the model. The verified and validated model can benefit the design and optimisation of pre-reduction reactors.

Experimental details for the pre-reduction with Hydrogen

For current research work, calcined Nchwaning ore (Calcination done in a muffle furnace at 900°C, air atmosphere) was used. Heating was carried out in an argon atmosphere with a flow rate of 2 NL/min up to the targeted temperature at a heating rate of 10°C/min and thereafter held at the targeted temperature for 10 min to obtain a uniform temperature distribution. Hydrogen was introduced with a fixed flow rate of 4 NL/min and a constant reduction time (90 min to 120 min). For all the reduction experiments, the reduction time was set to 120 min for the reduction temperatures 500°C to 700°C with 100°C intervals and the reduction temperature was set to 90 min for the reduction temperatures 800°C to 1000°C with 100°C intervals. Cooling was carried out under the argon flow (2 NL/min) to prevent manganese re-oxidation upon cooling. A graphical representation of the procedure is presented in Figure 9. For each test, 50 g of the calcined Nchwaning manganese ore sample was taken.
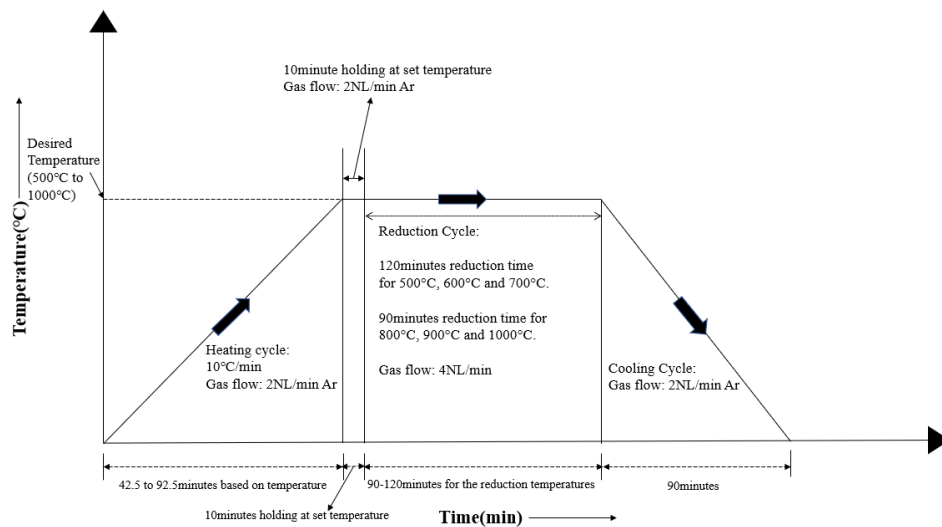
Figure 9. Schematic diagram of H₂ reduction.

The reduction with hydrogen experiments were conducted in a thermogravimetric setup. The setup consists of a vertical resistance furnace (Entech VTF 80/15), the position of which can be adjusted vertically to surround the crucible. The crucible is made of high temperature stainless steel (steel grade 253 MA) and was suspended in a balance (Mettler Toledo PR2003DR) to record the weight changes continuously throughout the experiment. Figure 10(a) shows a sketch of the crucible where it is shown that the gas passes through the double wall where it is pre-heated prior to interaction with the sample. The gas inlet and outlet are connected to the crucible by flexible synthetic rubber tubing, as can be seen in Figure 10(b) and 10(c)], and a thermocouple in an alumina tube is positioned such that the tip is in the centre of the sample material Figure: 10(a) and 10(c)]. Figures 10(b) and 10(c) show the crucible when the furnace is in its upper and lower position, respectively. Multiple gases can be used, and there are separate mass flow controllers for each gas (Bronkhorst F-201C). Ar, CO, CO_2, and H_2 with purity of 99.999%, 99.97%, 99.9992%, and 99.9%, respectively, were used in the experiments. Synthetic air containing 21% O2 and 79% N2 was used in some experiments. The gases are mixed before entering the crucible. The overall TGA furnace setup is shown in Figure 11.
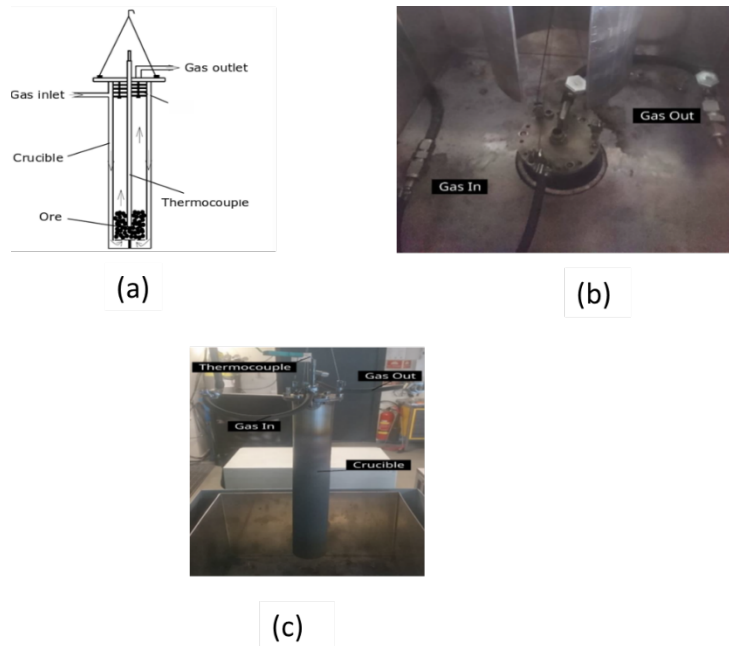
Figure 10. (a) Sketch of the crucible; (b) top view of the crucible when the furnace is in its upper position. Gas inlet and outlet is indicated; (c) Side view of the crucible when the furnace is in its lower position. Inlet and outlet gas, crucible and thermocouple input has been indicated.
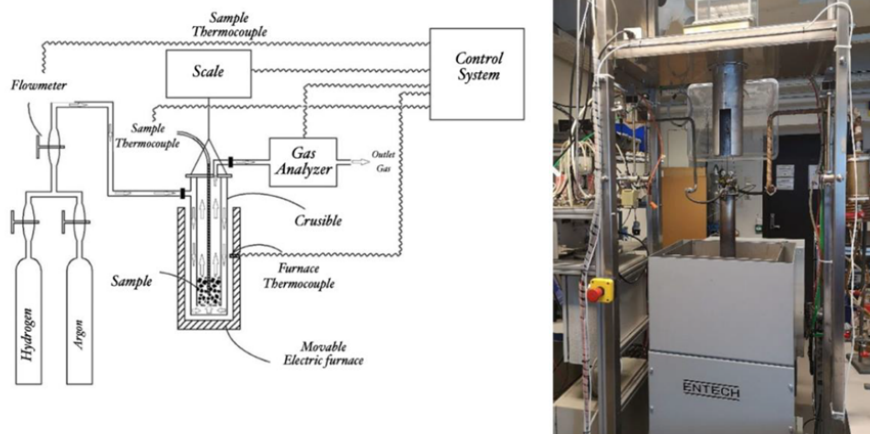

Figure 11. The TG furnace setup.

The CO and CO_2 content of the off-gas was measured by infrared absorption (Uras 26, ABB). The gas exiting the crucible is run through wash bottles containing water to avoid uncontrolled condensation and to achieve a consistent temperature in the off-gas analyser. The sample temperature, off-gas analysis and weight loss were continuously recorded. The sample weight was measured on an external scale (Ohaus Pioneer PA4202) before and after the experiments.

Figure 12 shows the mass loss (%) of calcined Nchwaning manganese ore at 500°C, 600°C, 700°C, 800°C, 900°C and 1000 °C at a reduction time of 90 min to 120 min for a 50 g sample weight. The degree of

reduction and the rate of reduction is higher at at 900°C and 1000°C. The results indicate that mass loss increases with increasing temperature. However, the difference in mass loss at 900°C and 1000°C is insignificant, therefore there is no benefit of increasing the reduction temperature beyond 900°C.
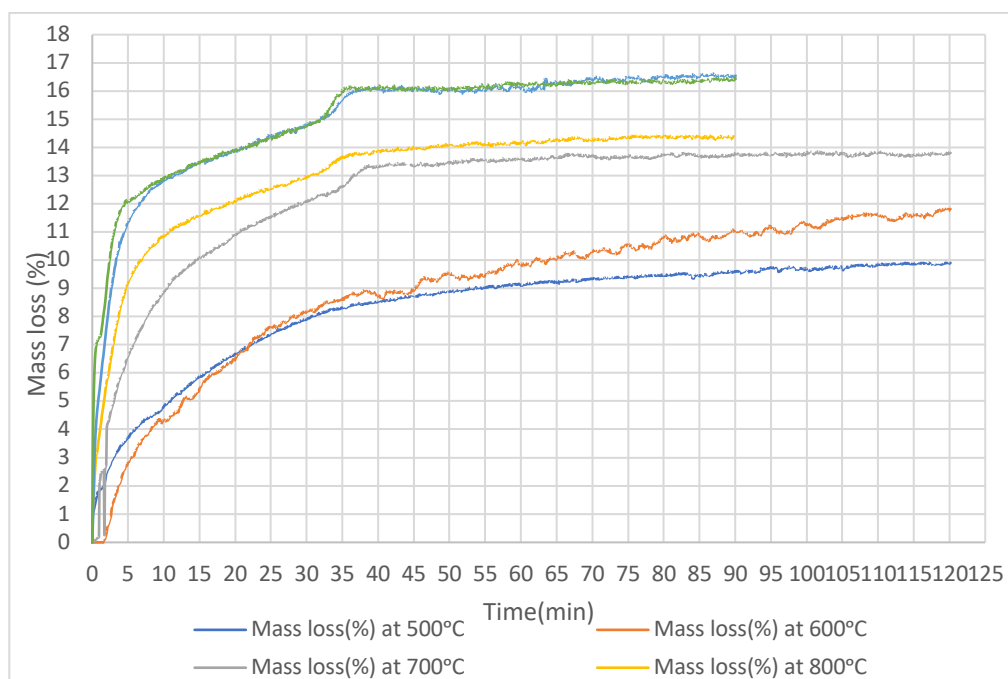

Figure 1. Mass loss (%) vs time(min) plot of pre-reduced samples.

Model verification

The model results were compared with experimental data in order to gauge whether its descriptions and predictions are viable. The mass loss data from the pre-reduction of calcined ore were used for model validation. This is because the model does not take into account the mass loss from carbonate decomposition, and the experimental data from pre-reduction of non-calcined ore would result in disagreement between the model and experimental results. The model results comparison with experimental data was performed for pre-reduction at 700°C as observed in Figure 13. The rate of mass loss from the model and the experiment follow the same trend until 2000 seconds and thereafter the discrepancy is observed. This can be attributed to some CO_2, which may have not been fully removed during calcination, and some volatiles in the sample. The CFD model can be used to scale-up and optimise the pre-reduction reactors given its accuracy in predicting pre-reduction behaviour for the demonstrated geometry and operating conditions.
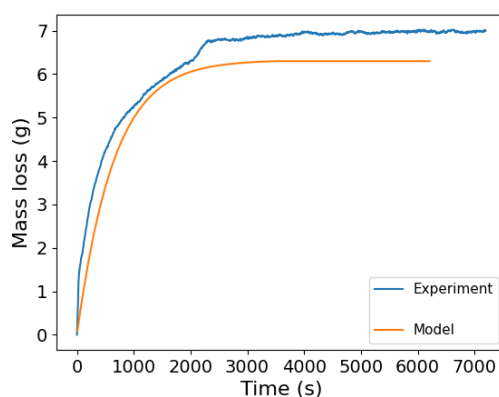

Figure 13. Experimental and model mass loss results evaluated at 700°C.

CONCLUSIONS

The study used CFD reacting heterogeneous model to investigate the pre-reduction of Nchwaning manganese ore by hydrogen. The findings revealed that the kinetics are influenced by temperature as the conversion of Fe_2O_3 to Fe and Mn_2O_3 to MnO was observed to be faster and high at higher temperatures. High porosity facilitates faster rate of pre-reduction as shown.

ACKNOWLEDGEMENT

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Mopeli Khama holds a PhD in chemical engineering from the University of Kwazulu Natal. His PhD work focused on the development of computationally efficient CFD-Hybrid models for heterogeneous systems. He has also worked on Population Balance modelling of bubble column reactors during his MSc studies at the University of Cape Town. His current research interests are in the Computational Fluid Dynamics model development and analysis of gas-solid pre-reduction processes with the view to aid design and optimization. These CFD models will help the de-carbonization initiatives in the metal production processes