

EXAMENING EXPERIMENTAL PARAMETERS FOR ILMENITE REDUCTION WITH HYDROGEN

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ABSTRACT

The following paper looks to shed light on the effects different experimental parameters have on ilmenite reduction. Three parameters were chosen to vary, sample size, crucible type, and gas flow. From our findings sample size and gas flow has effects on the reduction rate, while crucible modification has no effect. The lower the sample size, i. e. 4 grams, the greater the reduction degree and rate. For low gas flow rates, 0.3 l/min, the reduction degree was low, while for rates between 2-4 l/min there was a plateau where the reduction degree remained constant. Chemical analysis suggested that the reduction followed the same path for all experiments, showing that only the reduction rate was affected.

Ilmenite ($FeTiO_3$) is one of the main raw materials used to produce white pigment[1]. In today's process, it undergoes multiple steps before ending up as high titania slag[2]. One of these steps is carbothermic pre-reduction. The pre-reduction metallizes the iron (Fe) optimizing the ilmenite pellets before being fed into a submerged arc furnace (SAF). The carbothermic reduction leads to carbon dioxide (CO_2) emissions. One way to avoid this is using hydrogen (H_2) as a reducing agent[3]. H_2 can be used in a fluidized bed, avoiding the need to pelletize the ore. The need to understand the interaction between gas and small particles increases. This abstract looks to shed light on the limitations when it comes to comparing results from different experimental setups.

The parameters of interest were sample size, gas flow, and crucible shape. 6 different sample sizes were chosen, 4, 6, 7, 8, 10, and 14 grams where an unmodified crucible was used. Investigating the effect of crucible modification, two sample sizes were chosen, 7 and 14 g. CFD modeling was performed on the setup to understand flow within the alumina furnace tube, Figure 1. From the modeling it could be seen that removing one of the crucible walls could increase gas flow in the crucible. For gas flow experiments, three flow rates were chosen, 0.3, 2, and 4 l/min and a sample size of 7 grams.

Figure 1 shows the % weight change recorded after reduction for all three parameters. From Figure 1a), the effects of % weight change as a function of sample size is plotted. One can see that as the sample size increases from 4 g the mass loss decreases. From previous results [4], 7 grams of sample has been used. Combining these findings, it is clear that, comparing results across experimental setups needs to be done with caution.

CFD modeling showed that the gas flow across the sample could be improved by removing one of the crucible walls, i.e. towards the gas inlet. Figure 1a) shows the results after modification. One can see for the two sample sizes, i.e. 7 and 14 g, there is little difference between unmodified and modified crucibles. This indicates that the supply of H_2 is sufficient to the sample and could indicate that the transport of H_2O away from the sample also is sufficient.

Figure 1b) shows the % weight change as a function of gas flow rate (l/min). As expected, for low flow rates, i.e. 0.3 l/min, the reduction decreases. The decreased flow rate leads to a lower supply of H_2 , as well as worse mass transport in and from the sample. This could be reasons for the lowered reduction degree. Wang et. al.[5] conducted similar studied, where H_2 was mixed with Ar gas, and for low vol% of H_2 i.e. 10-20 vol%, the reduction slowed down.

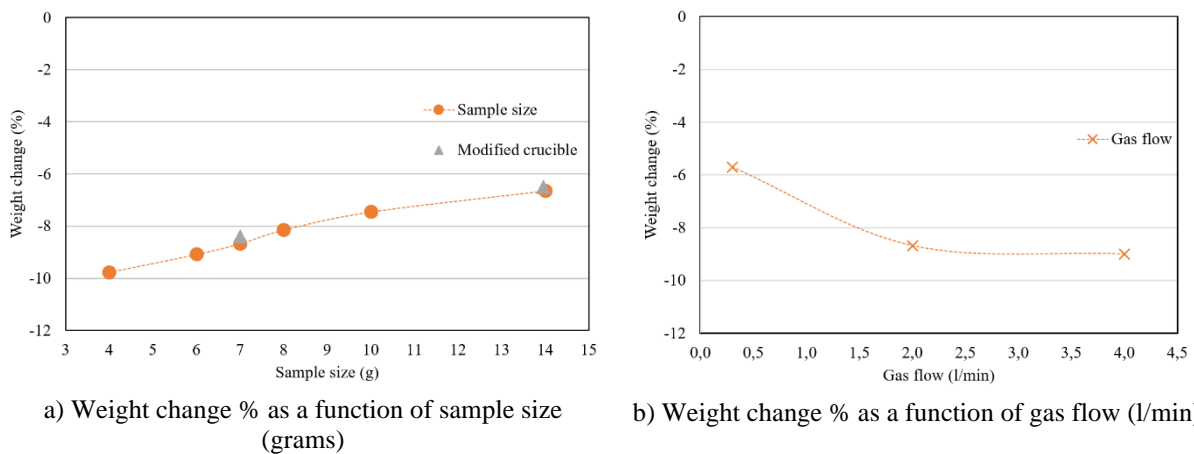


Figure 1: Reduced Senegalese ilmenite in hydrogen for 15 minutes at 806 °C

In conclusion, from the findings of this study one can see that comparing experimental results must be done with caution. For the reduction setup used in these experiments, the sample size has an effect on reduction degree and a sample size where a reduction gradient in the sample bed is avoided should be the optimal choice i.e. 7 grams, where no gradient was found. There seemed to be no difference in reactions occurring during reduction when changing these parameters. The rate was seen to be the only thing effected.

References

1. P. Mactaggart, A.M. *Pigment ID using Polarised Light Microscopy*. White Pigments 2007 June, 2007 [cited 2024 18.03]; Available from: <https://academicprojects.co.uk/white-pigments/>.
2. Rosenqvist, T., *The Tyssedal Ilmenite Smelting Process*, in *Terkel Rosenqvist Symposium*. 1988, Norwegian Institute of Technology: Norwegian Institute of Technology. p. 177-199.
3. Lobo, S.C., *Experimental Investigations and Modelling of Solid-State Ilmenite Reduction with Hydrogen and Carbon Monoxide*, in *Department of Material Science and Engineering*. 2015, Norwegian University of Science and Technology. p. 288.
4. T. P. M. V. Kaam, M.T., S. C. Lobo, *Hydrogen Reduction of Ilmenite: Effects of Oxidation*. To be published, 2024.
5. Wang, Y., et al., *Reduction Extraction Kinetics of Titania and Iron from an Ilmenite by H₂&Ar Gas Mixtures*. ISIJ International, 2009. **49**(2): p. 164-170.

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