



# Microwave carbothermic reduction roasting of a low grade nickeliferous silicate laterite ore



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## ARTICLE INFO

### Article history:

Received 19 June 2015

Revised 3 September 2015

Accepted 7 September 2015

Available online 14 September 2015

### Keywords:

Laterite

Ferronickel

Reduction

Carbothermic

Microwaves

## ABSTRACT

The known resources of nickel sulphide ores are quickly diminishing and in order to satisfy future nickel demands, nickel laterite deposits are being investigated as an alternative. Currently, expensive leaching and smelting processes are used to process the nickel laterite ores. The objective of the present research was to produce a high grade nickel concentrate via microwave carbothermic reduction roasting followed by magnetic separation. A thermodynamic model was developed for the roasting process in order to determine the optimum experimental conditions. The experimental variables investigated were: microwave energy and argon shrouding for the reduction tests and the magnetic field strength for the concentration stage. The behaviours of nickel and cobalt were studied in the reduction and magnetic separation processes. By optimizing the reducing and magnetic separation conditions, a high grade concentrate containing 9.2% nickel with a nickel recovery of 88.8% was achieved.

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## 1. Introduction

Nickel sulphide deposits are formed by either volcanic or hydrothermal processes and are mined at depth. Nickel laterite ores on the other hand, occur near the surface as a result of weathering in tropical climates, and are recovered using open pit mining. In 2004, laterite ores accounted for 72% of known nickel resources, yet only 42% of primary nickel metal production (Dalvi et al., 2004). Conversely, sulphides accounted for 58% of nickel production. This disparity is due to the technological challenges associated with upgrading the laterites. In the lateritic ores, the nickel is present as nickel oxide (NiO) in solid solution, whilst in the sulphide ores, the nickel is present as a distinct mineral commonly pentlandite ((Ni, Fe)<sub>9</sub>S<sub>8</sub>). Because of this, the laterite ores cannot be significantly beneficiated by mineral processing techniques. On the other hand, sulphide ores can be upgraded via flotation to produce a concentrate containing 6–15% nickel. Furthermore, this can be performed either at or close to the mine site before the concentrate is shipped for subsequent pyrometallurgical processing for metal extraction via roasting and smelting.

Nickel laterites can be classified into two main groups: limonites (60%) and saprolites (40%) (Butt and Cluzel, 2013). Most nickel laterite deposits contain both limonitic and saprolitic ores.

The limonitic laterite deposits are found closer to the surface and are primarily composed of goethite, (FeO·OH), and both the nickel and cobalt can substitute for iron in the goethite. The saprolite ore, found deeper in the deposit, is primarily composed of garnierite (Mg<sub>3</sub>Si<sub>2</sub>O<sub>7</sub>·2H<sub>2</sub>O) within which nickel may substitute for magnesium. The limonites are primarily processed using hydrometallurgical methods, whilst the saprolites are typically treated using pyrometallurgical processes. The hydrometallurgical processes require large amounts of acid whilst pyrometallurgical processing of the saprolite has a very high energy cost. These factors also contribute to environmental issues in the treatment of the nickeliferous laterites and some studies have shown that the processing of the nickeliferous limonitic laterite ores may have more of a negative environmental impact than the sulphide ores, despite the absence of sulphur in the oxide ores (Mudd, 2009). Considerable research effort is being devoted to developing new processes for the extraction of nickel from the laterites (Harris et al., 2009; Rao et al., 2013; Elliott et al., 2015). In a manner similar to the sulphide ores, it would be appropriate to produce a nickel concentrate from the laterite ores via selective reduction of the nickel to produce a ferronickel, followed by upgrading via magnetic separation, flotation or leaching. Also, it would be desirable to maximize both the nickel grade and the nickel recovery in the concentrate.

In most pyrometallurgical processes the major sources of energy remain carbonaceous or hydrocarbon based. Ultimately, the carbon in these materials is converted into carbon dioxide,

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which contributes to climate change. In this regard, microwave/millimeter wave processing techniques have potential applications in mineral processing and metal extraction systems (Pickles, 2009a, 2009b; Peng and Hwang, 2015). The electrical energy utilized in these processes can be relatively clean and there are other potential advantages, such as reduced dust production and gas throughputs. Another major advantage of microwave technology is the ability to overcome the limitations of the low thermal conductivities of oxides. Microwaves can heat specific materials rapidly and the technology is clean and easy to control. Regarding the microwave absorption characteristics of relevant materials: hematite is an active microwave absorber, magnetite and carbon are hyperactive, whilst magnesia and silica are inactive.

The objective of the present research was to carbothermally reduce the ore in a microwave system and then magnetically concentrate the resulting ferronickel product. Firstly the literature is reviewed regarding the microwave carbothermic reduction of nickel laterites. Secondly, an equilibrium model created using HSC Chemistry® 6.1 is discussed in order to determine the predicted nickel grades and recoveries as a function of temperature and the carbon to ore ratio. Thirdly, an experimental procedure is described for the microwave reduction and magnetic separation process for a low grade nickel silicate laterite ore. Fourthly, the effects of operating variables on the grade of the magnetic concentrate and also the recovery of the nickel to the concentrate are reported. Finally, the optimized conditions for the production of a high grade concentrate with a high nickel recovery are discussed.

### 1.1. Microwave treatment of laterites

The heating behaviour and the permittivities of a limonitic laterite ore have been investigated by Pickles (2004). Thermogravimetric analysis (TGA) and derivative thermogravimetric analysis (DTGA) were performed on the ore. Free water was removed by about 100 °C and the conversion of goethite to hematite occurred in the temperature range of 250–400 °C as follows:



Preheating the sample in a conventional furnace improved the microwave heating behaviour. Susceptors such as charcoal and the use of a fireclay crucible also improved the heating process.

Pickles (2005) investigated the microwave reduction of a high grade nickeliferous silicate laterite ore containing 2.44% nickel. The process parameters that were studied included: power input, processing time, sample mass and charcoal addition. TGA/DTGA analysis showed that the decompositions of goethite and lizardite occurred at 240 °C and 500 °C, respectively. Charcoal improved the heating behaviour of the material. The absorbed microwave power increased with incident microwave power, sample mass and charcoal addition. These samples were processed at relatively high temperatures and were essentially smelted. A Davis Tube Tester (DTT) was used to separate the ferronickel as a magnetic concentrate. The maximum values for grade and recovery were found to be 13% and 90%, respectively. This work suggested that if microwave heating was combined with conventional heating as a hybrid process, the benefits of both could lead to an enhanced treatment method. Samouhos et al. (2012) studied the microwave reduction of a limonitic laterite and investigated the same variables as Pickles (2005). However, in their research, powdered samples were used instead of briquettes. The three power levels used were: 200, 400 and 800 W. The study showed that both increasing power and carbon addition increased the reduction degree of the laterite. This study recommended that future work be performed to improve this process including matching the sample mass to the microwave power used.

Chang et al. (2008) investigated the effects of charcoal content and processing time on the mineralogical phases of the processed limonitic laterite ore at a fixed power of 800 W. Charcoal contents of 1.9%, 5.2% and 7.6% were used with a maximum processing time of 12.5 min. It was found that the major mineralogical phase, goethite, was converted to hematite, then to magnetite (a hyperactive material) thus improving the heating behaviour of the sample. The magnetite would then convert to wüstite and finally iron. These transformations are summarized in the following sequence;  $\text{FeO} \cdot \text{OH} \rightarrow \text{Fe}_2\text{O}_3 \rightarrow \text{Fe}_3\text{O}_4 \rightarrow \text{FeO} \rightarrow \text{Fe}$ . At a charcoal addition of 1.9%, hematite and magnetite were the major phases present. At a charcoal addition of 5.2%, the predominant phases were magnetite and wüstite. Lastly, at a charcoal addition of 7.6%, a ferronickel phase was found to be present for the longer processing times.

Yi et al. (2011) performed an experimental study of the reduction roasting and magnetic separation of a nickel silicate laterite ore using microwave heating. The effects of processing time and coal additions were studied. A power level of 2 kW was used for all the tests. The reduced material was then subjected to grinding at different times before being magnetically separated at various magnetic field strengths. With regards to the effect of microwave processing time, an increase from 30 to 90 min did not significantly affect the nickel grade as it was found to remain relatively constant in the range of 3.5–3.8%. A time of 45 min was proposed to be the ideal roasting time for a coal blending amount of 22.7%, where a nickel grade of 5.2% and a nickel recovery of 72% were achieved. The authors stated that shorter grinding times were necessary to ensure that valuable minerals were not lost with the tailings. Hence, a grinding time of approximately three minutes was proposed. The fourth variable, the effect of magnetic field strength, was studied between the ranges of 1300–2500 Oe. In general, an increase in the magnetic field strength resulted in a gradual decrease in the nickel grade. The nickel recovery increased from 72% at 1300 Oe to 82.4% at 1900 Oe before decreasing to 62% at 2500 Oe. However, at 1900 Oe, the nickel grade was only 3.9%, which was lower than the grade of 5.2% at a magnetic field strength of 1300 Oe.

He et al. (2013) studied the microwave processing of a limonitic laterite ore and investigated the amount of charcoal used as a reducing agent. Three amounts of charcoal were used: 1.9%, 5.2% and 7.6%. The as-received laterite ore was not a good microwave absorber. For example, with no charcoal, the temperature at 5 min was only about 330 °C. However, for the same time, with charcoal additions of 1.9% and 5.2%, the corresponding temperatures were 687 °C and 922 °C, respectively. As the hematite and magnetite phases formed, the temperature increased rapidly, as they are better microwave absorbers than the goethite, originally present in the laterite ore. TGA studies showed that weakly bound water was removed at temperatures up to about 180 °C, whilst the mineral goethite decomposed up to 300 °C. A processing time of approximately 12.5 min was necessary to achieve some reduction of the laterite ore. At this time, the maximum temperature of the material began to level off, for all of the tests.

### 1.2. Thermodynamic model

In previous research, thermodynamic models have been developed using the Equilibrium Composition program of HSC® 6.1 for both the sulphidation (Pickles et al., 2013) and the reduction (Pickles et al., 2014) of the nickeliferous limonitic laterite ores. The model has been extended to the reduction of the nickeliferous silicate laterite ores (Elliott et al., 2015) and is applied in the present research to the carbothermic reduction of the Murrin Murrin ore. The ore was assumed to be comprised of Ni, Co, Fe, Mg, Si, Al and O. Carbon (C) was added as the reductant. Inputting these

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