

Key Factors Affecting Nickel Recovery during the Segregation of Laterite Ores

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The segregation process as applied to nickel laterite ores involves the formation of gaseous nickel and iron chlorides which are subsequently reduced to form ferronickel on the surface of a solid carbon reductant. A fundamental study of the process has been carried out using limonite, nontronite and garnierite ores. The study highlighted the significant impacts that ore mineralogy and carbon addition have on the amount of nickel which is segregated rather than retained within the ore due to in-situ reduction. These important aspects affecting nickel recovery and the success of the process are discussed in this paper.

Keywords: carbon reductant, chloride addition, in-situ reduction, laterite, mineralogy, nickel segregation

Introduction

Western Australian (WA) Ni laterite deposits account for ~20% of the world's laterite resource and largely remain undeveloped. Ni laterites typically consist of three increasingly weathered zones: a limonite zone, overlying a nontronite zone (also known as a transition, smectite or clay zone), and a lower garnierite zone (also known as a saprolite or serpentine zone). Limonite has the highest Fe and lowest MgO and SiO₂ contents and typically contains ~0.8-1.5% Ni; garnierite has the lowest Fe and highest MgO and SiO₂ and typically contains ~1.5-3% Ni; nontronite has an intermediate composition and typically contains ~1.4-4% Ni.

The segregation process offers a possible alternative to current processes (HPAL, Caron, RKEF and Electric Furnace) used for the treatment of Ni laterite ores.

Segregation involves extraction of Ni through chloridisation of NiO at ~1123-1273 K, followed by Ni concentration at a carbon surface through reduction of volatile Ni and Fe chlorides to form ferronickel. This product can be recovered from the segregated ore

by either flotation or magnetic separation.

A significant number of studies were carried out on the segregation of Ni laterites during the 70's and 80's; reported Ni recoveries¹ varied from 17-83% for limonite, 66-70% for nontronite (one study only) and 50-90% for garnierite ores. These studies provided important insight into the process mechanisms but many aspects remained unclear. For this reason, a detailed review of the segregation process and a fundamental study on its application to Western Australian laterites was recently carried out¹; the results discussed in this paper form part of that study.

It is well known that Ni and Fe oxides can be reduced in-situ by carbonaceous gases during the segregation process. This will decrease Ni recovery since in-situ ferronickel cannot be chloridised for subsequent reduction onto carbon. There is little doubt that prevention of in-situ ferronickel formation is a key factor in achieving high Ni recoveries from the segregation process. The aim of this paper is to provide insight into how laterite composition and mineralogy, and carbon addition, affect in-situ ferronickel formation and hence the potential success of the segregation process.

Experimental

Complete experimental details are described elsewhere¹. Segregation of limonite, nontronite and garnierite Ni laterite ores was carried out under standardised (5% CaCl₂, 5% C reductant, relative to dried ore) rather than optimised conditions, to provide a clearer assessment of the effect of mineralogy on the process. Segregation was carried out in a vibrated, vertical tube silica reactor, similar to that used by Davidson², but fitted for continuous measurement of oxygen pressure using a SIRO₂C700® probe placed just above the bed, and for continuous measurement of

carbonaceous gas evolution using a Ritter TG05 fluid displacement meter; these additions allowed the monitoring of carbon consumption and oxygen removal.

Metal segregated from nontronite and garnierite ores was concentrated using a Davis Tube magnetic separator and the concentrate analysed for metallic content using a selective Br₂/methanol leach; metal segregated from limonite could not be magnetically concentrated and was leached directly from the calcine. Reduction of laterites to form in-situ ferronickel in the absence of chloride was also studied to contrast with segregation; Ni recovery was also determined using the selective leach.

Materials

Laterites

Nickel laterite ores (limonite, nontronite and garnierite) were provided as drill core by Heron Resources Limited from deposits associated with their Kalgoorlie Nickel Project in Western Australia. The samples were dried, split into representative samples and then roasted in air for 30 minutes at the temperature of subsequent segregation. The purpose was to reduce H₂O from the 10-13% present in dried ore, down to 0.5-0.8%, and also to dissociate any carbonates and to oxidise any sulphides. Chemical analyses of the resulting limonite, nontronite and garnierite calcines are shown in Table 1.

Table 1. Chemical analyses of dry nickel laterite calcines*

wt%	NiO	CoO	Fe(t)	SiO ₂	MgO	Al ₂ O ₃	CaO	Cr ₂ O ₃	MnO	Na ₂ O
Limonite	1.39 (1.1% Ni)	0.05	59.8	3.64	0.56	5.23	0	2.40	0.15	1.12
Nontronite	1.72 (1.4% Ni)	0.08	27.3	45.5	2.26	4.42	0	6.78	0.19	0.10
Garnierite	1.75 (1.4% Ni)	0.05	11.7	57.7	16.0	2.79	2.13	0.98	0.24	0.32

*Assays adjusted to 0% H₂O; calcines actually retained 0.5-0.8% H₂O depending on roast conditions.

The calcines were sized using a laser sizer and their mineralogy assessed using quantitative XRD, and for nontronite and garnierite, also using EDS-SEM analysis.

Limonite

The limonite laterite was a mixture of hydrated Fe oxides (goethite and limonite) with minor hematite and spinel (MgAl_2O_3). The limonite calcine was fine (80% passing 60 μm) and contained 86% hematite (60% Fe, Table 1). All Ni was considered evenly dispersed within hematite, based on extensive mineralogical analysis of calcined limonites from Western Australia³.

Nontronite

The nontronite laterite was essentially a mixture of the hydrated Fe silicate mineral nontronite (~46%), with quartz and minor chromite (FeCrO_4). The fine nontronite calcine (80% passing 100 μm) contained 39% Fe_2O_3 (27% Fe, Table 1) as hematite and minor maghemite. The calcine also contained 46% silica (Table 1) as both crystalline and amorphous phases.

Ni was contained within the nontronite mineral. After roasting, remnant nontronite was present as finely grained/amorphous dehydroxylate of intimately mixed hematite and silica. Ni was dispersed throughout the dehydroxylate at an average grade of 1.6%, most likely associated with Fe oxides. Coarser crystalline silica and chromite particles were present in the calcine but did not contain Ni.

Garnierite

The garnierite laterite was a mixture of quartz (~50%), hydrated Mg Al silicate (chlorite, ~16%), hydrated Mg Fe silicate (serpentine, ~7%) and hydrated Fe oxides (~8%), along with minor minerals including dolomite, diopside, chromite and kaolinite.

Ni was contained mainly within the chlorite (clinochlore and nimite) and serpentine (lizardite) minerals. After roasting, chlorite dehydroxylate was present as Ni rich

silicate veins within crystalline silica particles. The veins consisted of fine crystalline Mg-Al silicates (olivine and pyroxene) containing on average 7.7% Ni. Serpentine dehydroxylate was present as intimate mixtures of fine to amorphous Mg silicates, hematite and silica, containing on average 2.2% Ni, with appearance similar to nontronite dehydroxylate.

Overall, the fine garnierite calcine (80% passing 120 μm) contained 12% Fe (Table 1) present mostly as hematite ($\sim 18\%$ Fe_2O_3), both as coarse particles and within amorphous dehydroxylate. The 58% SiO_2 (Table 1) in the calcine was present as large crystalline quartz particles, with a lesser amount present within dehydroxylates as silica and silicates.

Carbon Reductant

Metallurgical coke and activated coconut carbon were used as reductants, the latter to provide a greater carbon surface area per gram to enhance reduction. The metallurgical coke (80% passing 280 μm) had a specific surface area of 4.3 m^2/g and contained 78.3% C. The activated carbon (80% passing 300 μm) had a specific surface area of 1160 m^2/g and contained 88.7% C.

Nickel Segregation

Figure 1 shows percent recovery of Ni versus time for all three calcines segregated at 1173 K, using 5% coke and 5% CaCl_2 relative to the dried ore. The curves are generated from integrated 'first order to a limit' rate equations fitted to experimental data¹. For nontronite and garnierite, segregation of Ni was rapid at first but then slowed to a limit; limonite behaved similarly but only after an induction period caused by the initially high concentration of hematite in the calcine. Under these standardised segregation conditions, the maximum recovery of Ni was greatest for

limonite (80%), intermediate for nontronite (63%) and least for garnierite (50%). Significantly higher Ni recoveries are predicted for nontronite and garnierite under optimised conditions, as discussed subsequently.

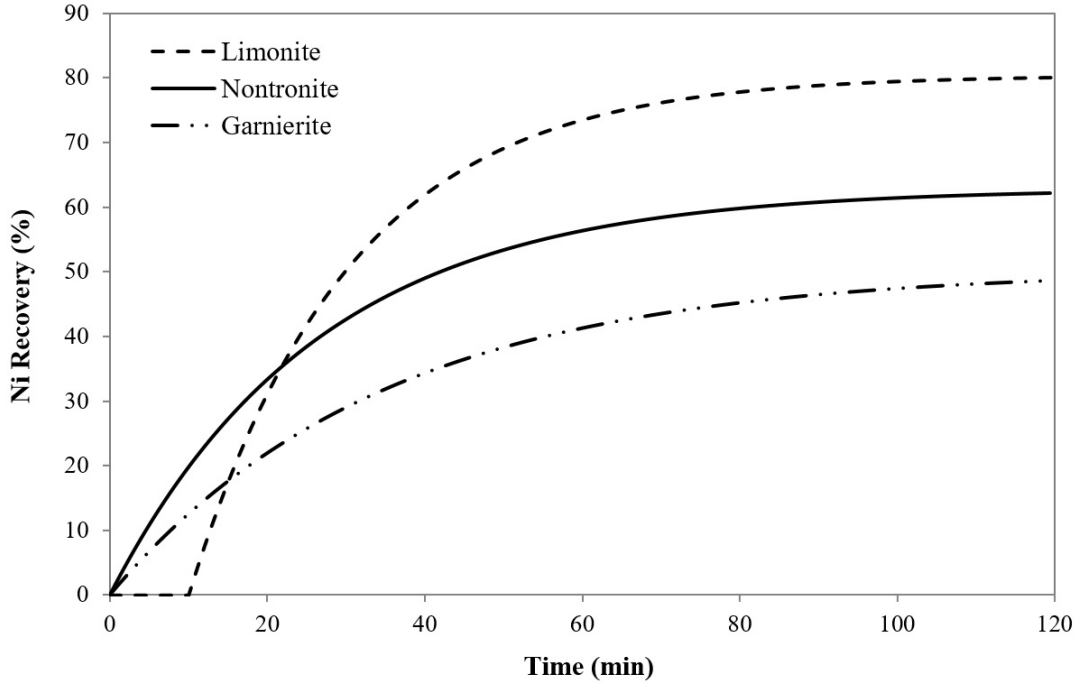
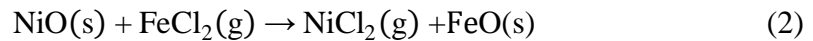
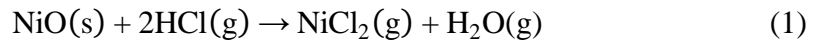


Figure 1. Ni recovery from laterites after segregation at 1173 K with CaCl₂ and coke, each added at 5 g/100 g of the dried ore.

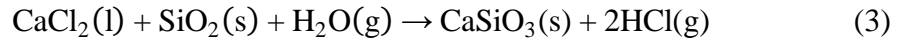
The rate of Ni segregation for all three ores was found to be dominantly controlled¹ by the rate of chloridisation of NiO from within the calcine (footnote¹), via the reactions:



The pressure of HCl in the gas is usually generated by the hydrolysis of CaCl₂ in the presence of silica (Reaction 3), and the pressure of FeCl₂ is generated by the

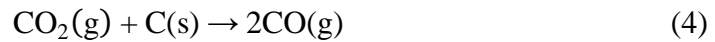
¹ For garnierite, the rate was also controlled by the rate of H₂ generation at the carbon surface by the water gas reaction.

chloridisation of FeO with HCl.

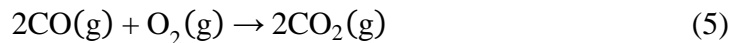


In-Situ Reduction

All three calcines (Table 1) contained Fe as hematite (Fe_2O_3), with the amounts decreasing in the order limonite (86%) > nontronite (39%) > garnierite (18%). While ferronickel is being reduced onto solid carbon through segregation, the reduction of Fe oxides by carbon also occurs via a CO intermediate within the gas. The reduction of hematite to magnetite with CO, for example, generates CO_2 which then reacts with C to regenerate CO through the Boudouard reaction:



Oxygen removed from the calcine by carbon reduction is thus evolved from the reaction bed dominantly as CO and CO_2 , with the average oxygen pressure within the bed being related to the CO_2/CO ratio through the equilibrium for the reaction:



The average CO_2/CO ratio in the evolved gas at any time during the reaction is expected to lie much closer to the equilibrium value for oxide reduction than for the Boudouard reaction. This is because oxide reduction with CO at segregation temperatures occurs at a much faster rate than the reaction of CO_2 with solid carbon, at least for pure oxides⁴.

The sequence of reduction of Fe oxides in the presence of silica, from least to most stable, is: Fe_2O_3 (hematite) \rightarrow Fe_3O_4 (magnetite), $\text{Fe}_3\text{O}_4(+\text{SiO}_2) \rightarrow \text{Fe}_2\text{SiO}_4$ (fayalite), $\text{Fe}_3\text{O}_4(+\text{SiO}_2) \rightarrow \text{FeSiO}_3$ (ferrosilite), $\text{Fe}_3\text{O}_4 \rightarrow$ 'FeO'(wustite) and 'FeO' \rightarrow Fe. The CO_2/CO ratio will decrease along with the oxygen pressure as Fe oxide reduction

progresses during segregation.

If all oxides are pure, and in the absence of any noticeable effect of the Boudouard reaction on gas composition, the oxygen pressure would remain constant during each Fe oxide transformation, and fall stepwise as each of the Fe oxide species is reduced.

However, the oxygen pressure falls continuously during reduction due to the influence of solid solutions on the activities of the species, since within the impure calcines, Fe_3O_4 is present within spinel, Fe_2SiO_4 within olivine, FeSiO_3 within pyroxene and 'FeO' within monoxide. The Boudouard reaction can also have some effect on the gas composition.

If the oxygen pressure becomes low enough, simultaneous reduction of FeO and NiO from within oxides and silicates will produce in-situ ferronickel alloy; this occurs well before reduction to form pure Fe is possible.

Limonite In-situ Reduction

Limonite calcine contains 86% Fe_2O_3 as hematite. Reduction involves the transformation of hematite to magnetite, followed by magnetite to fayalite as minor silica is consumed, and finally magnetite to wustite. Figure 2 shows the measured oxygen pressures within the gas versus reaction time for limonite calcine at 1173 K, for: (1) reduction with coke without chloride addition, (2) segregation with coke, and (3) segregation with activated carbon. Segregation with activated carbon was only carried out for 80 minutes compared to 120 minutes for both segregation and reduction with coke. The equilibrium oxygen pressures for the transformation of pure Fe oxide and silicate species are projected across the diagram for reference.

Figure 2 shows that the oxygen pressure profiles during reduction or segregation with

coke are similar. However, the profile for segregation with activated carbon lies below the other two, since the extent of reduction at a given time is increased because the carbon surface area is much larger for activated carbon compared to coke. All three profiles show an initial rapid decline in oxygen pressure, but this is an artefact from the heat up of the oxygen probe. The carbonaceous content of the gas would be virtually 100% CO₂ during the initial reduction of hematite to magnetite and the true initial oxygen pressure would lie close to the Fe₂O₃/Fe₃O₄ equilibrium at the top of the diagram.

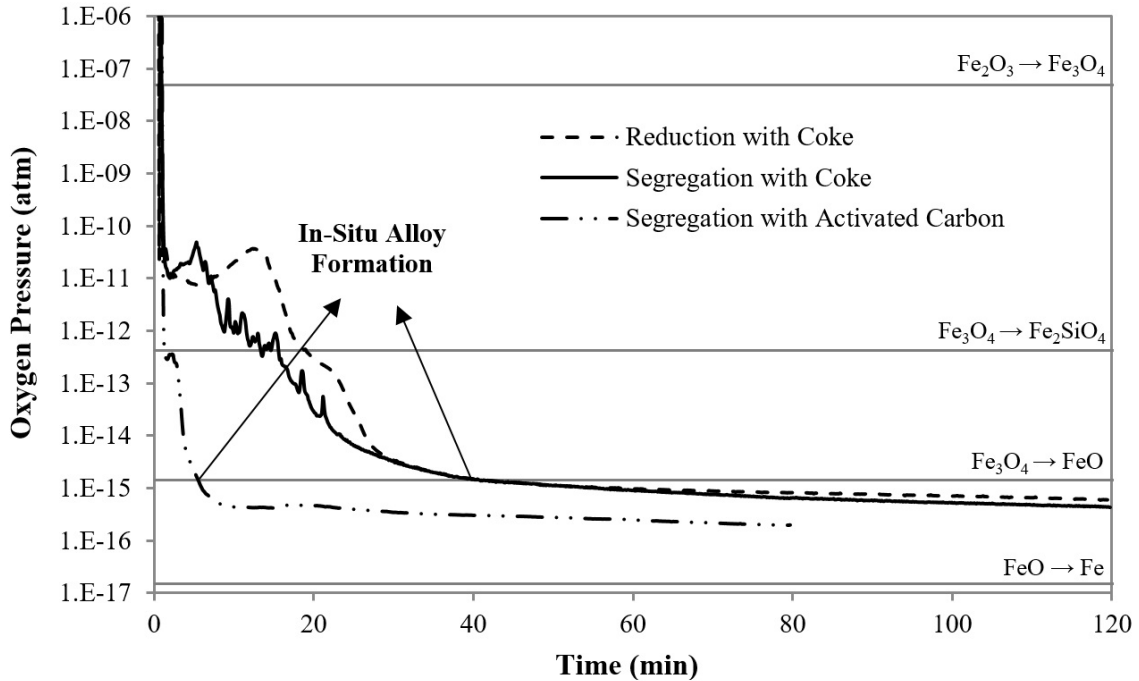


Figure 2. Oxygen pressure profile comparison, limonite, 1173 K. Oxygen pressures for segregation are a best fit from multiple experiments. Equilibrium values for pure oxides are for reference. On-set of in-situ alloy formation is a FACTSage (v6.4) equilibrium prediction¹.

The final oxygen pressure for all three reduction scenarios remains above that for the reduction of pure 'FeO' to Fe. However, equilibrium simulation¹ using FACTSage (v6.4) software predicted that in-situ reduction to form ferronickel can occur when the

oxygen pressure falls to 1.4×10^{-15} atm, assuming no prior removal of Ni from the calcine by segregation. Under this scenario, in-situ ferronickel is predicted to form after 5.5 minutes during segregation with activated carbon and after 41 minutes during segregation or reduction with coke at 1173 K (Figure 2).

Experimental data were consistent with these predictions. For reduction with coke in the absence of chloride, 60% of Ni was present as in-situ ferronickel after 60 minutes, 19 minutes after in-situ alloy is predicted to form. Significant in-situ ferronickel was also found after segregation with activated carbon, consistent with the formation of in-situ alloy at 5.5 minutes, only 1 minute after the induction period for the start of limonite segregation¹. The in-situ alloy was present amongst remnant Fe oxides (Figure 3); Ni recovery from segregation was limited to 67% after 120 minutes as a result.

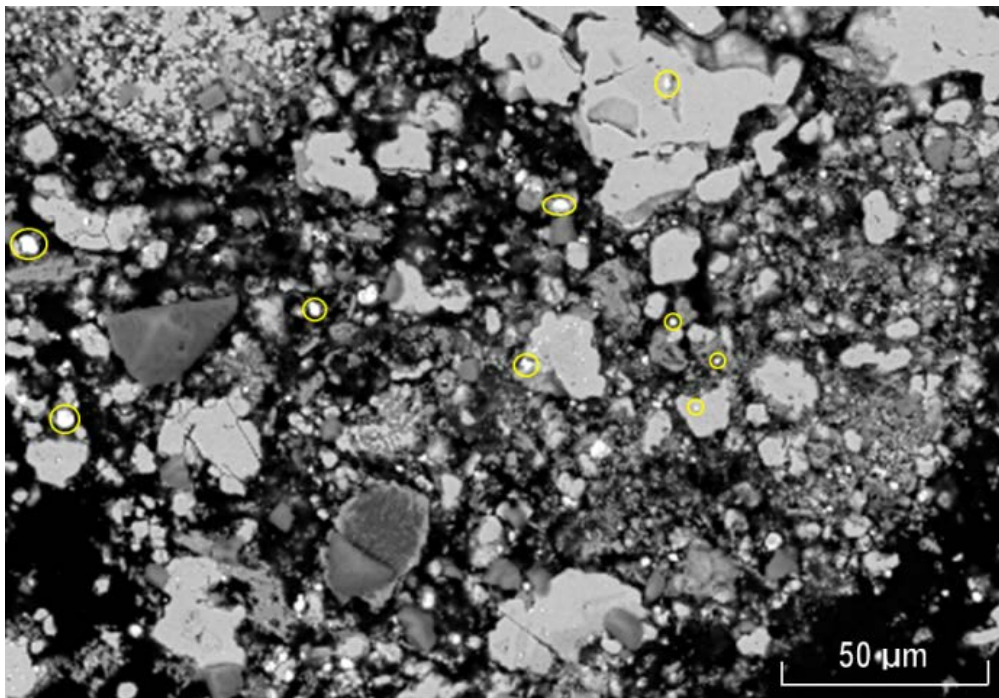


Figure 3. In-situ ferronickel (circled) within limonite calcine segregated with activated carbon at 1173 K.

Segregation with coke commenced after 10 minutes and removed 60% of Ni at 41

minutes (Figure 1), when in-situ ferronickel is first predicted to form, provided all Ni remains in the calcine. As Ni is removed by segregation, the critical pressure for in-situ alloy formation progressively decreases, making its formation less likely. Since in-situ ferronickel did not form during segregation with coke at 1173 K, as confirmed by experiment, early removal of Ni from the calcine by segregation completely suppressed the formation of in-situ alloy under these conditions. As a result, Ni recovery from segregation increased to 80% after 120 minutes when using coke at 1173 K, compared to 67% when using activated carbon.

Nontronite In-situ Reduction

Figure 4 shows the measured oxygen pressure profiles for nontronite calcine at 1173 K, for: (1) reduction with coke without chloride addition and (2) segregation with coke. No segregation was carried out with activated carbon at 1173 K. The figure shows that in contrast to limonite, much lower oxygen pressures were reached at a given time during segregation compared to reduction. It was also found that carbon consumption during segregation was 67% higher than for reduction after 120 minutes. This confirmed that the rate of carbon gasification via the Boudouard reaction (and hence the rate of Fe oxide reduction) was significantly enhanced for nontronite calcine when chloride was present; although likely related to mineralogy, the cause requires further investigation.

Nontronite calcine contains 39% hematite and 46% silica, significantly less hematite and significantly more silica than for limonite calcine, with a substantial portion of silica being amorphous and in contact with Fe oxides. As a result, there is substantial reduction of magnetite to form both fayalite (in olivine) and ferrosilite (in pyroxene), as confirmed by experiment.

Reduction of magnetite to form Fe silicates occurs at higher oxygen pressures than when the product is wustite and silicate formation can potentially buffer the oxygen pressure above that necessary for in-situ alloy formation. For reduction of nontronite with coke at 1173 K, without chloride addition, all hematite is reduced to magnetite after 20 minutes, and then silicates form. The oxygen pressure continues to fall as reduction proceeds but remains above the critical oxygen pressure for in-situ alloy formation (7×10^{-15} atm), even after 120 minutes of reduction with coke at 1173 K (Figure 4). This was confirmed by experiment since no ferronickel was found in this case.

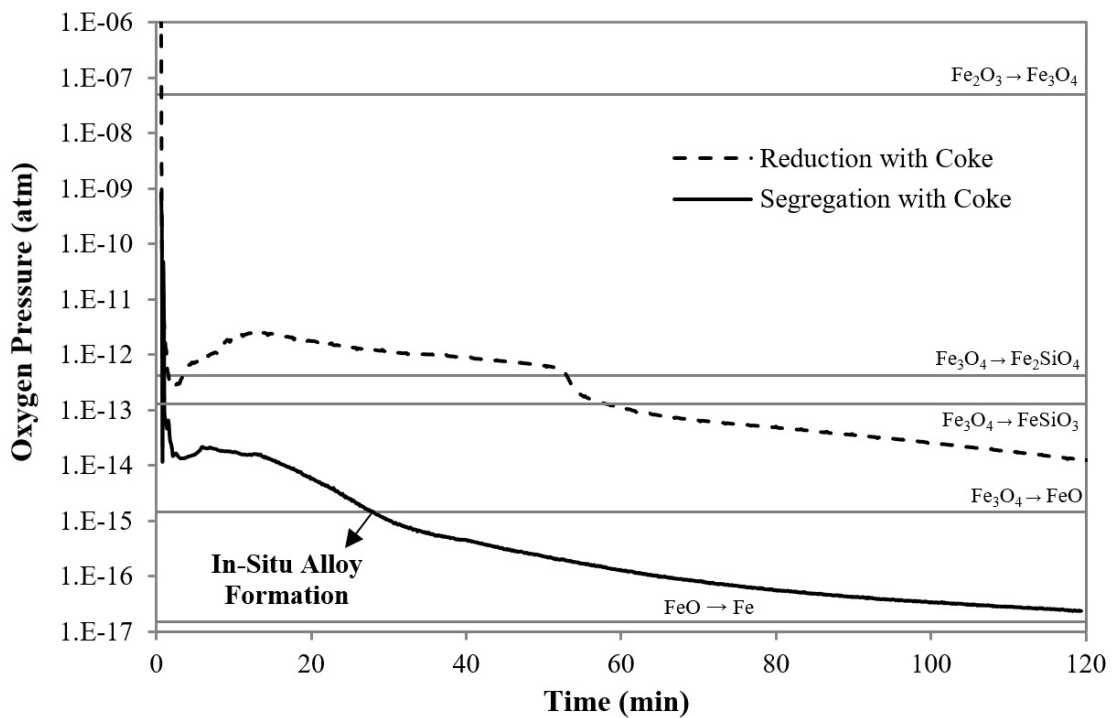


Figure 4. Oxygen pressure profile comparison, nontronite, 1173 K. Oxygen pressures for segregation are a best fit from multiple experiments. On-set of in-situ alloy formation is a FACTSage (v6.4) equilibrium prediction¹.

For segregation of nontronite with coke at 1173 K, the rate of Fe oxide reduction is significantly faster than in the absence of chloride; the conversion of hematite to

magnetite is complete after 5 minutes and the majority of magnetite is reduced to form silicates after 30 minutes. The oxygen pressure then continues to fall but with little gas evolution, as likely the remaining magnetite is slowly reduced from within spinel solid solutions.

The critical oxygen pressure for in-situ alloy formation is reached after 27 minutes (Figure 4) but by this time, 40% of Ni has been removed from the calcine by segregation (Figure 1). This removal completely suppressed the formation of in-situ ferronickel during segregation, as confirmed by experiment. Ni recovery was 63% after segregation with coke for 120 minutes at 1173K (Figure 1), with unsegregated Ni present as NiO within Fe silicates (ferrosilite and minor fayalite), in remnant nontronite dehydroxylate.

When the temperature of segregation with coke was increased to 1223 K, the recovery of Ni increased to 71%, with very minor in-situ alloy observed. When coke was replaced by activated carbon at 1223 K, the recovery fell marginally to 67%, and in-situ ferronickel was observed amongst the Fe silicates within remnant nontronite dehydroxylate.

Garnierite In-situ Reduction

Garnierite calcine contained the least amount of hematite (12%) and the highest amounts of silica (58%) and magnesia (16%); the latter present mainly as finely crystalline Mg-Al silicates within chlorite dehydroxylate and Mg silicates within serpentine dehydroxylate. At least half of the silica was present as coarse crystalline particles. The oxygen pressure profiles for both reduction and segregation with coke at 1173 K were similar, with minor enhancement of the carbon gasification rate during segregation. No segregation was carried out with activated carbon at 1173 K.

Figure 5 compares the oxygen pressure profile of garnierite with those for limonite and nontronite during segregation with coke at 1173 K. The oxygen pressure at which in-situ alloy can form (3.9×10^{-15} atm) is higher for garnierite compared to the other calcines and is reached earlier, after 7.5 minutes, when only 10% of Ni has been removed by segregation (Figure 1). As a result, significant in-situ ferronickel formed when segregating garnierite calcine with coke at 1173 K, as confirmed by experiment, with the recovery of Ni limited to 50% after 120 minutes (Figure 1).

The recovery of Ni increased to 65% for segregation with coke at 1223 K but fell to 48% when activated carbon was used at this temperature. In-situ ferronickel formed in all segregation experiments conducted with garnierite, using either coke or activated carbon, with the majority of unrecovered Ni present as in-situ reduced alloy amongst remnant chlorite and serpentine dehydroxylates.

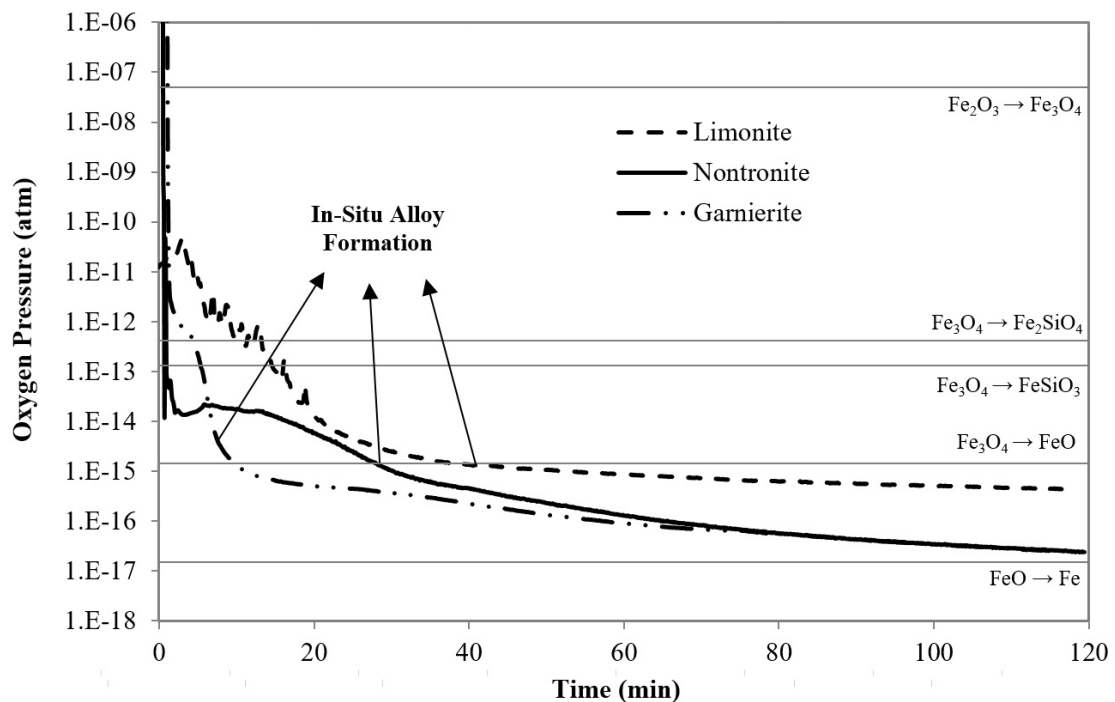


Figure 5. Oxygen pressure profiles from the segregation with coke of limonite, nontronite and garnierite at 1173 K.

Discussion

In-situ Reduction

The critical oxygen pressure at which in-situ ferronickel can form depends on the activities of both NiO and FeO within the calcine, which are related to the calcine composition and mineralogy. Oxide activities were not measured in this work; rather critical oxygen pressures were predicted as a first approximation using equilibrium simulations.

Since NiO is less stable than FeO, calcines containing higher concentrations of NiO will tend to have higher critical oxygen pressures which favour in-situ ferronickel formation. Nontronite calcine contained more NiO than limonite (cf. 1.72, 1.39%, Table 1) and was predicted to have a higher critical pressure at 1173 K (cf. 1.7×10^{-15} , 1.4×10^{-15} atm). In contrast, nontronite and garnierite calcines contained similar NiO (cf. 1.72, 1.75%), but the predicted critical pressure for garnierite at 1173 K was significantly higher (cf. 3.9×10^{-15} , 1.7×10^{-15} atm) due to the relatively high, localised concentration of Ni within chlorite and serpentine dehydroxylates of 7.7 and 2.2% respectively. Such high localised Ni concentrations may be present for other roasted garnierite ores, since chlorite and serpentine are the common minerals within garnierite which contain Ni.

The extent of in-situ ferronickel formation for a particular calcine depends on both the oxygen profile generated within the carbonaceous gas during reduction, as determined by the sequence of Fe oxide reduction reactions, and the rate of carbon consumption. At a given carbon consumption, the oxygen pressure especially depends on the amounts of hematite and silica in the calcine and whether these have intimate contact such that silicates can form. Thus the potential for in-situ alloy formation during segregation is affected by the laterite calcine composition and mineralogy, as well as the carbon

addition.

Increase in temperature or carbon surface area enhances the carbon gasification rate via the Boudouard reaction. This increases the Fe oxide reduction rate and lowers the oxygen pressure at a given time. If the rate of carbon consumption is not too high, the formation of silicates can arrest the fall in oxygen pressure and suppress in-situ reduction, as observed for nontronite in the absence of chloride (Figure 4). It thus seems feasible that blending ores to promote silicate formation may be beneficial during segregation, as observed by Davidson².

For a specific reductant, an increase in C/Fe ratio within the charge will increase the rate of reduction of Fe oxides and lower the oxygen profile, thus increasing the potential for in-situ reduction. The C/Fe ratio for garnierite (0.42) is 5 times greater than for limonite (0.08) and as shown in Figure 5, the oxygen profile for garnierite is significantly lower than for limonite. Nontronite has an intermediate C/Fe ratio (0.18) and shows an intermediate oxygen profile, at least in the range 5-60 minutes; however, the oxygen profile is also affected by the catalytic effect from the presence of chloride, which appears unique to the nontronite mineralogy (Figure 4).

Segregation

The initial rate of Ni segregation is very rapid (Figure 1). This seems fundamental to the success of the process since removal of Ni from the calcine progressively reduces the potential for in-situ alloy formation by lowering the critical oxygen pressure required for its formation. The rapid initial segregation also minimises the amount of NiO that remains unsegregated within solid solutions or silicates. Such NiO is difficult to chloridise towards the end of the segregation process when the NiO

reactivity is low and the HCl pressure is minimal, since little H₂O remains for hydrolysis of CaCl₂.

All segregation was carried out using standardised additions of CaCl₂ and activated carbon or coke. Under comparable conditions, segregation recoveries of Ni were always lower when using activated carbon because of the formation of in-situ ferronickel. Ni recoveries were also lower for reduction in the absence of chloride compared to segregation, mainly because finely dispersed in-situ ferronickel was difficult to recover from calcine by leaching¹.

The highest Ni recovery achieved for limonite under standardised segregation conditions was 80% using coke at 1173 K. No in-situ ferronickel formed and this recovery appears close to the maximum for limonite, based on similar recoveries achieved by others for temperatures up to 1273 K⁵. Ultimately, segregation recovery from limonite appears limited by the large mass of Fe oxides into which remnant NiO can dissolve and become unreactive.

The highest standardised Ni recovery achieved for nontronite was 79% using coke at 1273 K (40 K above the initial calcine sintering temperature), compared to 63% at 1173 K, with no loss due to in-situ ferronickel. The higher temperature increases the rate of chloridisation (and hence segregation) which likely reduces the amount of NiO ultimately captured within crystallising Fe silicates. Higher recoveries from nontronite may also be possible by enhancing the initial rate of chloridisation through addition of MgCl₂ along with CaCl₂. MgCl₂ produces a much higher HCl pressure than CaCl₂ during hydrolysis; however, it is quickly consumed and CaCl₂ is still required to sustain segregation.

The highest standardised Ni recovery from garnierite was 65% using coke at 1223 K, with recovery mainly limited by in-situ ferronickel which remained within both serpentine and chlorite dehydroxylates. Others⁶ have achieved 90% Ni recovery from garnierite using 2% C as coke, much lower than the standardised 5% C used here. This likely minimised in-situ alloy formation by significantly raising the oxygen profile. Similar high recoveries seem possible using lower C with the present ore, since excluding the Ni lost in-situ as ferronickel, only ~13% Ni remained within silicates, leaving ~87% potentially amenable to segregation.

Conclusion

High recoveries of Ni are possible from laterite ores using the segregation process provided in-situ reduction of ferronickel by the carbonaceous reaction gas can be suppressed. The potential for in-situ reduction depends largely on the reactivity of NiO in the calcine and the oxygen profile which is generated within the reaction gas during the segregation process. If Ni can be removed from the calcine by segregation before the oxygen pressure in the reaction gas becomes low enough to reduce ferronickel in-situ, segregation can be successful. This paper provides an understanding of the impact that laterite composition and mineralogy as well as carbon addition and temperature have on this process requirement, as a guide to achieving high Ni recoveries from laterite ores using the segregation process.

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