

# Segregation Roasting of a Saprolitic Ore - An Experimental Investigation -

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# Abstract

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- **The potential recovery of nickel from a South-American saprolitic laterite ore was investigated by applying the segregation roasting technique.**
  - Laterite ore was heated to temperatures that ranged between 900 and 1000°C in a rotary kiln. The laterite ore had been mixed with a known amount of calcium chloride and either coal or coke.
- **By varying the temperature and reductant type/addition rate in several tests, a preliminary set of optimal conditions was established for the specific laterite ore studied.**
  - The test procedure that was adopted and the results obtained are discussed in this presentation.
- **The benefits and challenges of commercialising segregation roasting are discussed.**

## Why Laterite Ore?

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- **Laterite ores account for an estimated ~70% of the world's nickel resources, with sulphide sources accounting for the remaining ~30%.**
  - Yet sulphide resources account for ~60% of the world's primary nickel production, while laterite resources account for the remaining ~40%.
- **Numerous reasons for this discrepancy exist, but the most important one is the mineralogical and compositional complexity that is associated with laterite ore deposits.**
  - This complexity currently offers only limited opportunities for upgrading, thereby creating capital intensive whole ore processing facilities.

# History Segregation Process

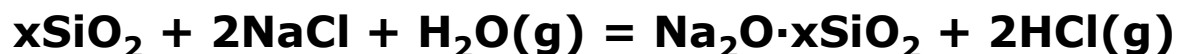
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- **Accidentally discovered in 1923 as a means of preparing Chilean copper ore containing copper silicate minerals and small amounts of sodium chloride for ammonia leaching by reductive roasting. After the roasting step, it was noted that :**
  - Copper had been “segregated” during roasting from the original copper mineral (a copper silicate chrysocolla).
  - Metallic copper had been formed and was deposited on the outer peripheries of the chrysocolla particles.
- **This discovery triggered a wave of research into this concept with particular application to the treatment of refractory copper ores found predominantly in the Zambian copper belt in Southern Africa.**
- **This process became known as the TORCO process (Treatment Of Refractory Copper Ores), and the research work culminated with the construction of a 500 ton per day pilot plant at Rhokana in Zambia.**

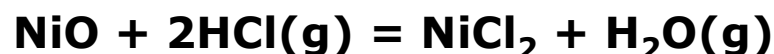
# Segregation Process Mechanism

The segregation process entails heating the minerals to temperatures far below their melting point in the presence of both a halide such as calcium chloride and a reductant such as coal or coke.

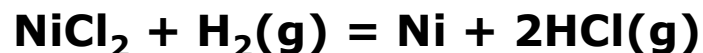
**Step 1: Initiation** - Salt is hydrolyzed by water in the presence of gangue minerals like silica, to produce hydrogen chloride.



**Step 2: Extraction** - HCl reacts with the oxide nickel minerals to produce nickel chloride.



**Step 3: Reduction and Segregation** - Hydrogen associated with the carbonaceous reductant reduces the nickel chloride to form the segregated nickel that can be recovered by applying physical separation techniques (e.g. magnetic separation).

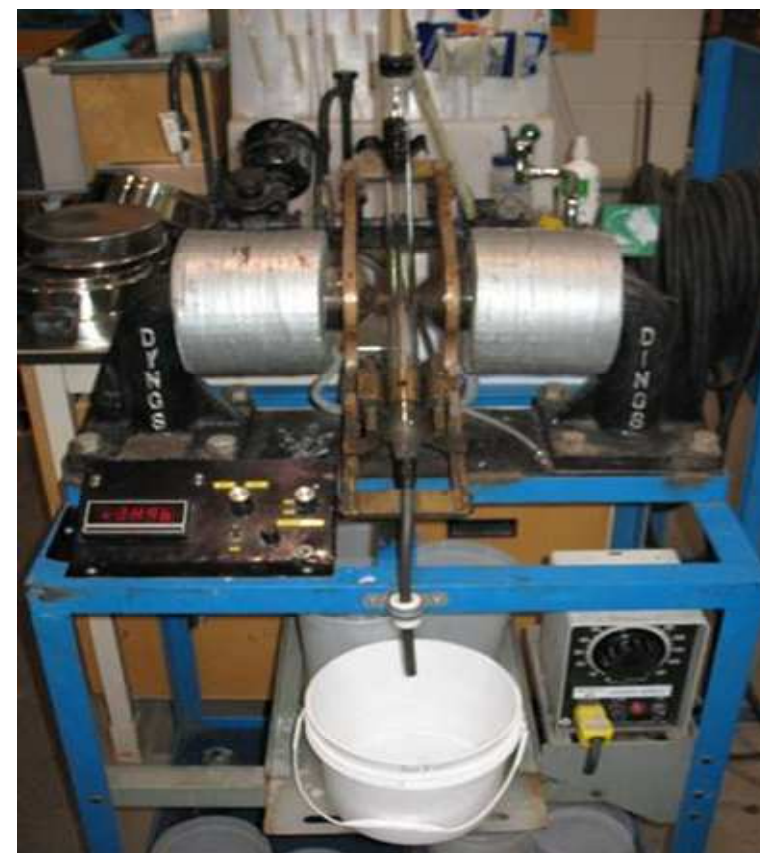


- Step 3 regenerates hydrogen chloride, which can react further with nickel oxide from the laterite ore.

# Experimental Apparatus



Rotary Reduction Kiln



Davis Tube Magnetic Separator

## Experimental Procedure (Cont..)

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- **In each test, a predetermined quantity of laterite ore was added to the rotary kiln to yield a total charge weight of 500g after addition of all other reagents.**
  - The ore was added to the reactor and then heated to the required temperature between 900 and 1000°C prior to the subsequent addition of halide and reductant.
- **The reagents consisting of weighed amounts of calcium chloride and coal/coke were added by means of a long handled spoon (ceramic weighing boat attached to a 20" ceramic rod) from the stationary end of the reaction tube.**
  - A nitrogen flow of about 1 L/min was maintained through the reactor during the test. Off gas was directed into a scrubber containing dilute aqueous NaOH solution to capture HCl/FeCl<sub>2</sub> evolved during the test prior to releasing it into a ventilated fume hood.

## Test Conditions

Test	Temp. ( °C)	Retention Time (hrs)	CaCl <sub>2</sub> (%)	Type of Reductant	% Coal/Coke	Kiln Rotation Speed (RPM)
1	950	1	7.5%	Coal	4.0%	2
2	950	1	7.5%	Coke	4.0%	2
3	950	1	7.5%	Coal	2.0%	2
4	900	1	7.5%	Coal	4.0%	2
5	1000	1	7.5%	Coal	4.0%	2

- Following the segregation tests, the product was allowed to cool gradually to room temperature.
- Product from the kiln was then collected from the reactor and subjected to magnetic separation to recover FeNi alloy.

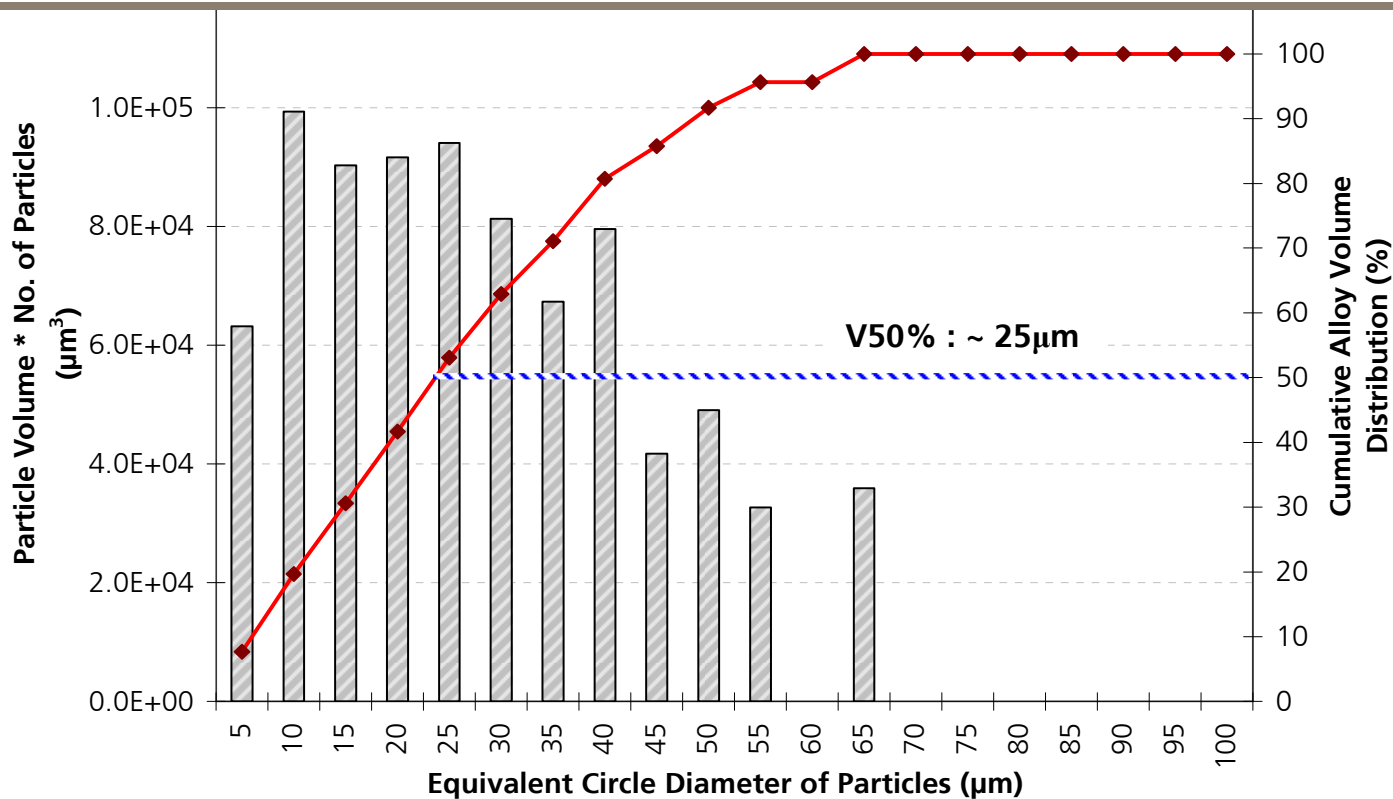
# Results and Comments: Grade & Recovery



Test No.	Test Conditions	Description	Weight (g)	Concentrate Grade (wt %)		Weight Distribution %	Normalized Recovery		
				Ni	Fe		Ni (%)	Fe (%)	
1	950 °C, 7.5% CaCl <sub>2</sub> , 4% Coal, 1 hr time	Product Head	53.1	1.27	15.6	6.84	32.8	9.4	
		Mags	3.35	3.98	12.3				
		Non-mags	45.6	0.60	8.70				93.2
2	950 °C, 7.5% CaCl <sub>2</sub> , 4% Coke, 1 hr time	Product Head	56.7	1.37	15.8	15.0	41.8	21.6	
		Mags	7.95	2.12	19.8				
		Non-mags	45.0	0.52	12.7				85.0
		Mags (Stage 2)	0.73	6.71	41.2				
		Non-mags (Stage 2)	2.83	1.04	15.4				62.5*
3	950 °C, 7.5% CaCl <sub>2</sub> , 2% Coal, 1 hr time	Product Head	194	1.20	8.90	2.66	58.5	3.2	
		Mags	4.76	<u>15.0</u>	11.4				
		Non-mags	174	0.29	9.40				97.3
4	900 °C, 7.5% CaCl <sub>2</sub> , 4% Coal, 1 hr time	Product Head	55.5	1.36	16.0	4.67	46.8	4.3	
		Mags	2.38	8.96	15.4				
		Non-mags	48.6	0.50	16.6				95.3
5	1000 °C, 7.5% CaCl <sub>2</sub> , 4% Coal, 1 hr time	Product Head	65.5	1.36	15.6	15.1	<u>84.6</u>	33.6	
		Mags	9.41	<u>6.76</u>	34.6				
		Non-mags	52.7	0.22	12.2				84.9
		Mags (Stage 2)	2.65	12.5	56.7				
		Non-mags (stage 2)	2.17	1.05	15.7				93.6*

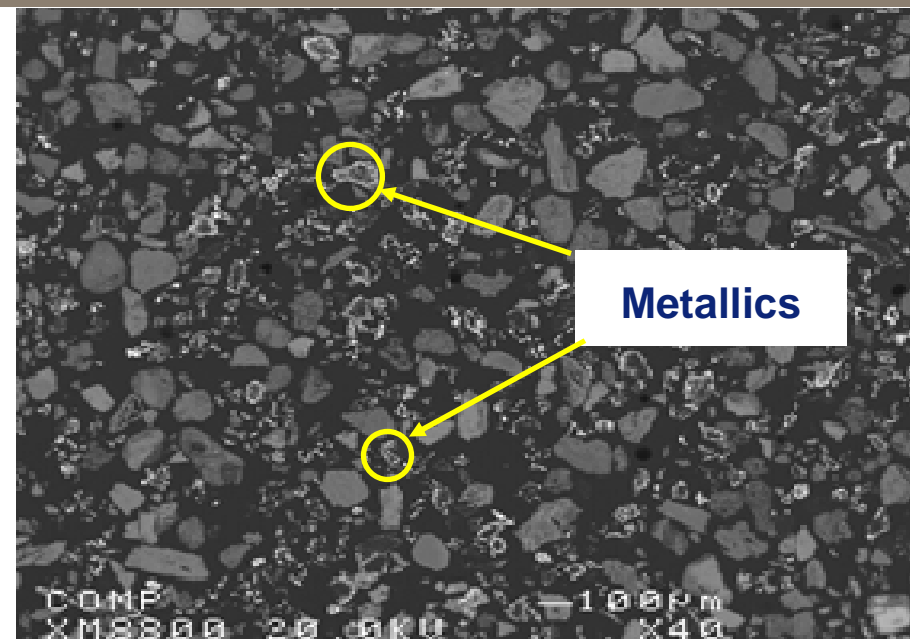
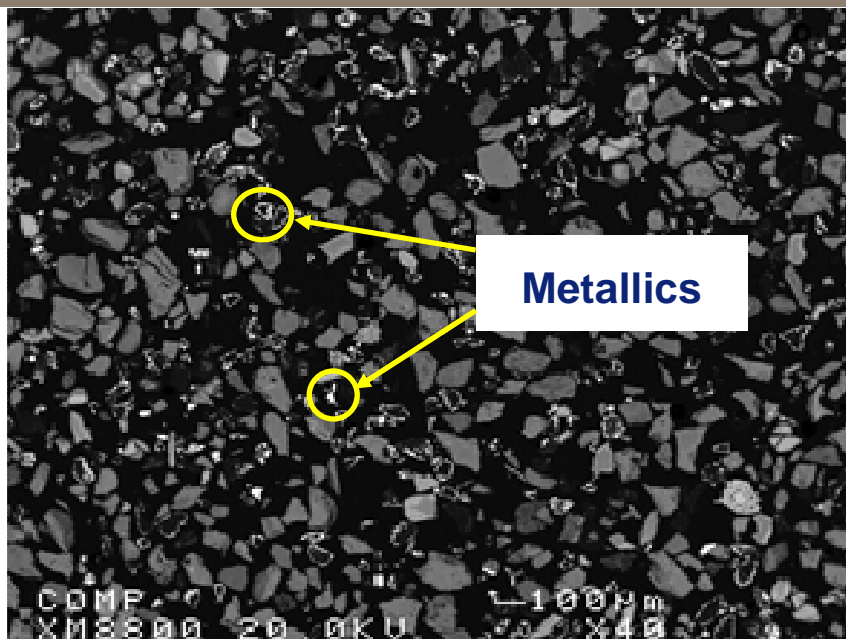
- The highest concentrate grade obtained was 15% Ni at a recovery of just under 60% (Test 3). A recovery of 85% was obtained at a grade of 6.1% Ni (Test 5).
- Increasing temperature has a significant beneficial impact on metal recoveries (comparison of Tests 4 & 5).

# Results and Comments: Particle Size Distribution



- In general, the FeNi particles observed are less than 85 microns in diameter.
- Additionally, the particle size diameter at the median particle volume for all of the tests ranged from 10 to 25 microns. It should be noted that these samples underwent partial size reduction during preparation.

# Results and Comments: BEI and MicroProbe Analysis



Above: Typical Micrographs Magnetic concentrate

Below : Microprobe assays for metallic prills

Description	Average Weight % (Analysis for Individual Metallic Prills)									
	Ni	Stdev	Fe	Stdev	Co	Stdev	Cr	Stdev	Si	Stdev
Test 1	61.2	21.6	38.1	21.7	0.67	0.31	0.03	0.02	0.00	0.00
Test 2	12.4	5.31	87.0	5.66	0.51	0.15	0.06	0.50	0.00	0.00
Test 3	73.8	4.80	25.2	4.18	0.95	0.35	0.10	0.13	0.01	0.01
Test 4	75.3	8.54	23.8	8.06	0.80	0.43	0.03	0.02	0.04	0.01
Test 5	34.0	18.8	65.4	18.5	0.60	0.16	0.01	0.01	0.00	0.00

## Results and Comments: FeNi Quality

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- **The microprobe assays indicate that the segregated metallics were found to mainly consist of ferronickel with small amounts of cobalt and chromium and almost no silicon.**
  - As expected, the nickel grade of the metallic prills decreased with an increasing amount of reductant addition
- **Most of the metallics were small, i.e. < 100 microns and the micrographs illustrate that further liberation (=grinding) is required to recover more FeNi particles.**
  - The creation of sufficiently large FeNi prills for commercial recovery is a key driver behind the success of this process, i.e. process control and reactor design are the engineering focus, not so much the metallurgy.

## Concluding Remarks

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- **The segregation process shows appreciable potential from a technical perspective, albeit some inherent challenges are associated with it.**
- **By varying the test parameters (reductant type and addition rate, halide addition rate, temperature and residence time), it is possible to establish an optimal set of operating conditions for a given laterite ore type.**
  - In the current test program, nickel concentrate grades ranging from 2.1% to 15% were obtained, with nickel recoveries ranging from 33% to 85%.
  - Microprobe analysis showed that the grade of the metallic particles ranged from 60-75% Ni at nominal particles sizes of less than 100 $\mu$ m.
- **Note: This was a proof of concept study and not an optimised process design**

# Potential Benefits Segregation Process



- **Provides an alternative process option for treatment of low-grade laterite ores.**
  - Potential to lower the FeNi smelting energy requirement since a large proportion of the gangue material is rejected during magnetic separation after segregation roasting.
  - Intermediate can be fed into existing FeNi smelter.
- **Production of a potentially saleable intermediate FeNi product with a lower capital cost investment. Especially viewed against the emergence of nickel pig iron as an alternate saleable product.**
  - Avoidance of constructing a large power plant for remote ore body locations and the supporting infrastructure requirements.

# Perceived Challenges Segregation Process



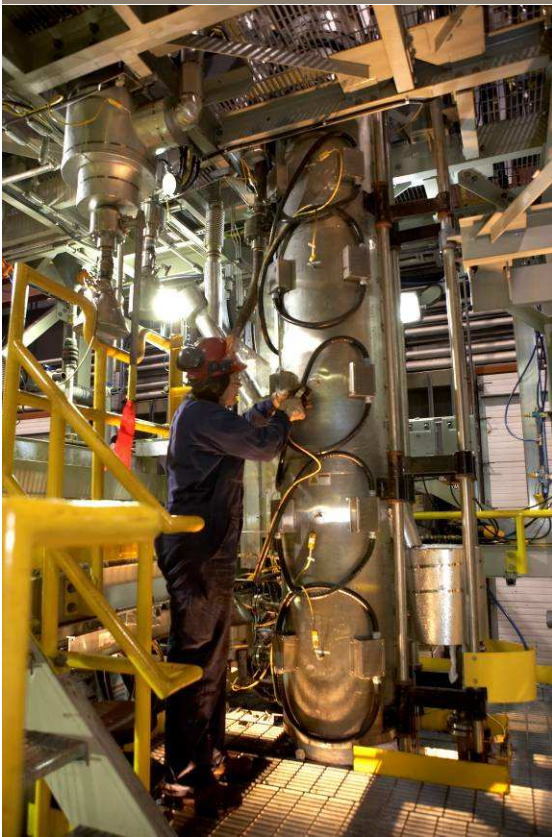
- **The use of chloride-based reagents is still perceived as a challenge on materials selection and reactor design.**
  - Chloride process practitioners such as Xstrata Nickel are well positioned to address this challenge (Nikkelverk refinery, Norway).
- **Carbon that is added to the ore is not completely consumed during segregation roasting, thereby potentially impacting downstream processing steps.**
- **No commercial plants based on this technology have been commissioned to date for nickel, making it difficult to benchmark with any commercial plant data.**
  - However, a number of fairly large scale plants have been commissioned for copper silicate ores using the same chemistry principles, e.g., the 500 tpd TORCO plant built in Zambia in the 1960's and the 4000 tpd plant built in Mauritania the 1970's, both by Anglo-American Corporation.
- **Due to the fine size of metallic prills that are formed, fine grinding may be required, depending on the technology selected to recover these metallics from the roasted ore matrix.**

# Known Challenges Segregation Process



- **Salts are required in (large) stoichiometric excess in order to attain appreciable kinetics.**
  - The residual earth-alkaline salts are hardly volatile and will remain in the residue, which commonly would need to be removed.
- **Most salts have melting points below 800 Celsius, which can cause melting phenomena in a charge yielding agglomeration or sintering effects; especially when assisted by poor heat transfer.**
  - Compared to HCl and Cl<sub>2</sub> salt is thermodynamically a poor chloridizing agent; but it's cheap and readily available.
- **Most salts have a significant tendency to hydrate, which makes it more challenging to handle them in storage and transportation.**
- **THE FOCUS is REACTOR DESIGN and ECONOMICS; not chemistry**

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Thank you

