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54	PROCESS FOR LEACHING LATERITIC ORE AT ATMOSPHERIC PRESSURE

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ABSTRACT

An atmospheric leaching process in the recovery of nickel and cobalt from a lateritic ore, said lateritic ore including a low magnesium ore fraction and a high magnesium ore fraction, said process including the steps of:

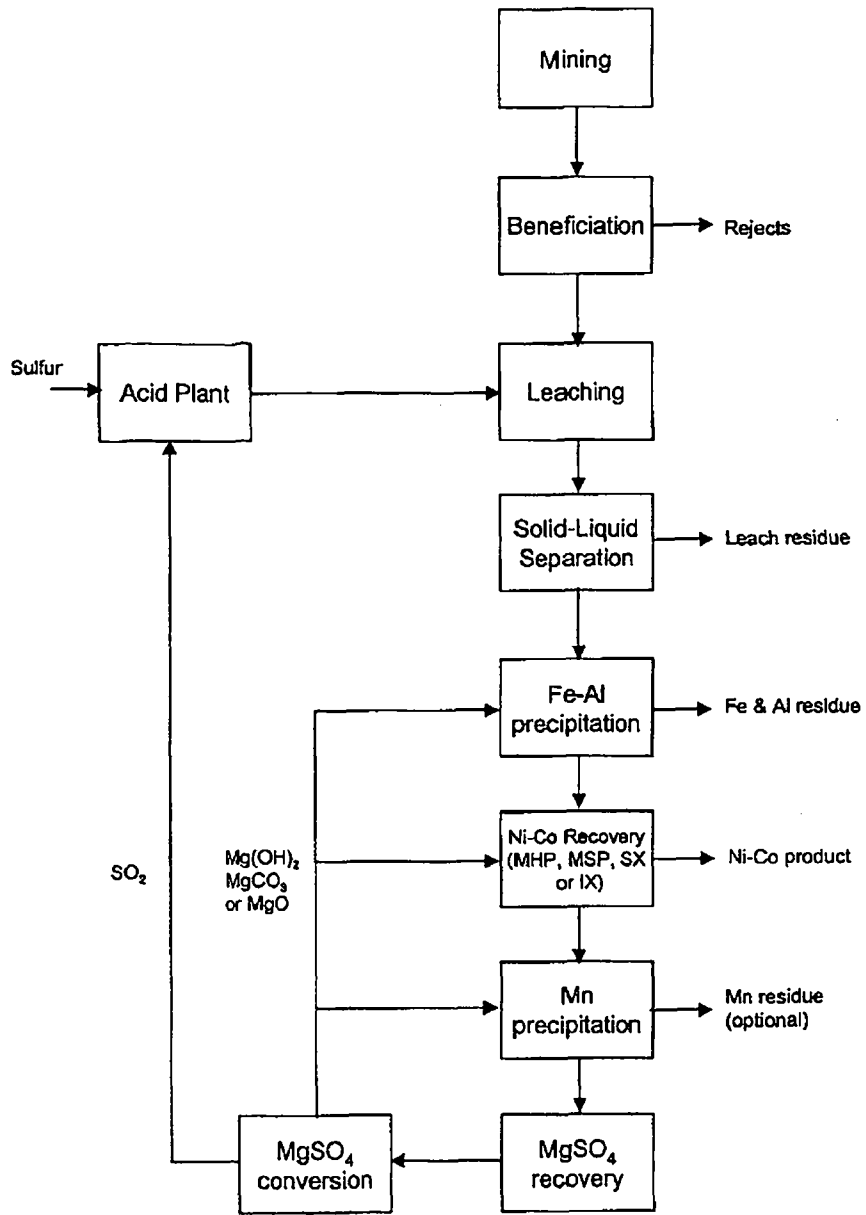
- (a) forming an aqueous pulp of said lateritic ore,
- (b) leaching said aqueous pulp with a concentrated mineral acid at atmospheric pressure to produce a slurry containing a pregnant leach liquor and a leach residue,
- (c) treating the pregnant leach liquor either separately or as part of said slurry to recover dissolved nickel and cobalt therefrom, leaving a magnesium containing barren solution,
- (d) treating said magnesium containing solution to recover a magnesium containing salt therefrom.

ABSTRACT

An atmospheric leaching process in the recovery of nickel and cobalt from a lateritic ore, said lateritic ore including a low magnesium ore fraction and a high magnesium ore fraction, said process including the steps of:

- (a) forming an aqueous pulp of said lateritic ore,
- (b) leaching said aqueous pulp with a concentrated mineral acid at atmospheric pressure to produce a slurry containing a pregnant leach liquor and a leach residue,
- (c) treating the pregnant leach liquor either separately or as part of said slurry to recover dissolved nickel and cobalt therefrom, leaving a magnesium containing barren solution,
- (d) treating said magnesium containing solution to recover a magnesium containing salt therefrom.

ADVERTISEMENT DRAWING



PROCESS FOR LEACHING LATERITIC ORE AT ATMOSPHERIC
PRESSURE

FIELD OF THE INVENTION

5 The present invention relates to a hydrometallurgical process to recover nickel and cobalt from nickeliferous laterite ores and, in particular, to the atmospheric leaching of both low magnesium fraction (eg limonite) and high magnesium fraction (eg saprolite) ores with a mineral acid to dissolve nickel and cobalt. In a preferred form of the process, the process also includes a step in which
10 magnesium values in the leach liquor are recovered.

BACKGROUND OF THE INVENTION

The known reserves of nickel and cobalt in nickeliferous oxide ores, e.g., those referred to as laterites comprising limonite and saprolite ore, are far greater than
15 the corresponding reserves in sulfide ores. An important disadvantage when processing laterite ores, however, is the inability to beneficiate these ores by conventional techniques.

A number of new hydrometallurgical processes are being developed for the
20 extraction of nickel and cobalt from nickeliferous laterite ores. Many of these processes require the dissolution of the metal values with sulfuric acid at high temperature (245.degree.-270.degree. C.) and pressure (525-785 psig), followed by solid-liquid separation and neutralization of residual free acid present at ambient pressure. This is the basic "Moa Bay Process", as described
25 by J. R. Boldt and P. E. Queneau in "The Winning of Nickel", Methuen, London, 1967. In this process, the nickeliferous ore is first made into a pulp having a solids content of about 40% before leaching at high temperature and pressure. During pressure leaching most metals dissolve and iron and aluminum are rejected by hydrolysis to hematite and alunite, respectively. After leaching, the
30 pulp is cooled and washed by counter current decantation and the solids are directed to tailing treatment. Excess acid is neutralized and the remaining iron and aluminum are precipitated as hydroxides with the addition of alkali. Nickel and cobalt are subsequently recovered via sulfide precipitation.

Several variations of the high-pressure acid leach (HPAL) method have been devised with the aim of improving the process and economical aspects. For example, U.S. Pat. No. 4,044,096 provides guidelines to optimize the high-pressure acid leaching of nickeliferous lateritic ores through a combination of operational steps to improve the economics and efficiency of leaching. The steps include scalping laterite ore to remove the coarse (high magnesium) fraction and thus lower the acid consumption.

The HPAL process is most amenable for high iron ores containing 40 wt % iron or higher. Lateritic ores with an iron content less than 40 wt % contain in general a higher amount of acid consuming minerals and are therefore not preferred for direct high pressure leaching. U.S. Pat. No. 3,804,613 teaches a method of high-pressure acid leaching of saprolite ore at relatively low acid/ore ratios by preconditioning the saprolite with leach liquor from the high-pressure leach step. No mention is made of concurrent limonite leaching.

U.S. Pat. No. 3,991,159 teaches the use of saprolite ore to neutralize acid resulting from the high-pressure acid leach of limonite ore. Leaching of the saprolite fraction is carried out at high temperature (150.degree.-250.degree. C.) and pressure for effective iron and aluminum rejection, but with relatively low nickel extraction from the saprolite ore. In another process, U.S. Pat. No. 4,097,575 teaches saprolite ore roasting at 500.degree.-750.degree. C. under oxidizing conditions to increase its neutralization capacity before neutralization of HPAL liquors. This process suffers from the additional need for roasting facilities.

While the prior art HPAL methods obtain a high extraction of nickel and cobalt, they require the use of expensive equipment and sophisticated materials of construction to withstand the use of concentrated acid at the high temperatures needed (200.degree.-300.degree. C.). Several alternatives to the HPAL process to recover nickel and cobalt from laterite ore have been proposed.

For example, U.S. Pat. No. 4,062,924 describes a method for leaching limonite ores in acidic media at temperatures up to 110.degree. C. and in the presence

of hydrogen sulfide gas to precipitate dissolved nickel and cobalt. Most dissolved iron is also reduced to the divalent oxidation state however, consuming very high amounts of the reducing gas in addition to high acid consumption. U.S. Pat. No. 4,065,542 teaches a similar method. In this process, ferrous iron produced by the method described above is used to leach metal values from manganiferous sea nodules. U.S. Pat. No. 4,511,540 illustrates a way to recover nickel and cobalt from ores with a manganiferous matrix by leaching with sulfuric acid in the presence of sulfur dioxide gas at temperatures below the boiling point of the liquid solution. None of these processes includes the treatment of saprolitic ores.

In the process of U.S. Pat. No. 3,793,432, limonite ore is leached with sulfuric acid at a pH below 1.5, while simultaneously adding alkaline iron-precipitating agents. The process is carried out at atmospheric pressures, but requires leaching times in excess of 40 hours and usually from 60 to 100 hours for efficient nickel extraction and iron precipitation. No use of saprolite is made in this process. U.S. Pat. No. 4,410,498 teaches a method to leach saprolite ore with sulfuric acid at atmospheric pressure, while adding a reducing agent to maintain the redox potential between 400 and 600 mV. In another process, described in U.S. Pat. No. 5,571,308, nickel and cobalt are leached from saprolite ore by contact with a mineral acid at room temperature or in the temperature range of 60.degree.-80.degree. C. The leaching mode can be conducted by heap, vat, or agitation leaching.

US Patent 6,261,527 also discloses a hydrometallurgical process for the recovery of nickel and cobalt from both limonite and saprolite ores, however in that process, iron is rejected as jarosite.

There are environmental concerns with this iron removal process as the jarosite compounds are thermodynamically unstable. Jarosite may decompose slowly to iron hydroxides releasing sulphuric acid. The released acid may redissolve traces of precipitated heavy metals, such as Mn, Ni, Co, Cu and Zn, present in the leach residue tailing, thereby mobilizing these metals into the ground or surface water around the tailings deposit.

Another disadvantage of this process is that jarosite contains sulphate, and this increases the acid requirement for leaching significantly. Sulphuric acid is a large input in acid leaching processing, so there is also an economic disadvantage in the jarosite process.

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The present invention aims to overcome or alleviate one or more of the problems associated with prior art processes.

10 The discussion of documents, acts, materials, devices, articles and the like is included in this specification solely for the purpose of providing a context for the present invention. It is not suggested or represented that any or all of these matters formed part of the prior art base or were common general knowledge in the field relevant to the present invention as it existed before the priority date of each claim of this application.

15

SUMMARY OF THE INVENTION

According to the present invention, there is provided an atmospheric leaching process in the recovery of nickel and cobalt from a lateritic ore, said lateritic ore including a low magnesium ore fraction and a high magnesium ore fraction, said
20 process including the steps of:

- (a) forming an aqueous pulp of said lateritic ore,
- (b) leaching said aqueous pulp with a concentrated mineral acid at atmospheric pressure to produce a slurry containing a pregnant leach liquor and a leach residue,
- 25 (c) treating the pregnant leach liquor either separately or as part of said slurry to recover dissolved nickel and cobalt therefrom, leaving a magnesium containing barren solution,
- (d) treating said magnesium containing solution to recover a magnesium containing salt therefrom.

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An advantage of the invention is the provision of an efficient and economical method to leach both low magnesium (eg limonite) and high magnesium (eg saprolite) ores in a single process stage at atmospheric pressure, to obtain high percent dissolution of nickel and cobalt. A further advantage of the method is

that it avoids the high capital costs associated with sophisticated autoclaves. Another advantage of a preferred form of the method is that it also avoids the production of jarosite. An advantage of a preferred form of the invention, is that the magnesium containing barren solution produced from the leaching process is treated to recover magnesium sulphate, which is then processed to give MgO, Mg(OH)₂ or MgCO₃ and SO₂. The SO₂ is advantageously used to regenerate H₂SO₄. The MgO or MgCO₃ may be fed back into the leaching process as a neutralising agent, disposed of as a stable residue, or sold as a commercial product.

DETAILED DESCRIPTION OF THE INVENTION

The low magnesium containing ore fraction includes the limonite fraction of the laterite ore (Mg wt % approximately less than 6). This fraction may also include low to medium level magnesium content smectite or nontronite ores which generally have a magnesium content of about 4 wt % to 8 wt %. The high magnesium containing ore fraction includes the saprolite fraction of the laterite ore (Mg wt % greater than approximately 8). This fraction may also include smectite or nontronite ores. The formation in step (a) of an aqueous pulp of both the low magnesium and high magnesium containing ore fractions is generally carried out in sodium, alkali metal and ammonium free water at solids concentration from approximately 20 wt % and above, limited by slurry rheology.

The ratio of acid to combined ore is typically at least 0.5. Preferably, the ratio is about 0.5 to 1.0, such as 0.5 to 0.7.

The aqueous pulp is subjected to a leaching step in step (b) utilising a concentrated mineral acid at atmospheric pressure. Preferably leaching is conducted whilst agitating leach reactants. Typically the leaching step is carried out at a temperature up to the boiling point of the leach reactants at atmospheric pressure. Most preferably the reaction temperature is as high as possible to achieve rapid leaching at atmospheric pressure. A preferred leaching temperature is at least 60°C, more preferably at least 75°C. In a preferred embodiment, leaching is carried out at around 80°C or higher, such as

at least 85°C. In another preferred embodiment, leaching is conducted at around 95°C.

5 Preferably, the leaching of both the low and high magnesium fractions occurs in a single process stage, which may comprise a single step, in which the two fractions are leached simultaneously, eg in the same tank or reactor. Alternatively, the two fractions may be leached in sequential steps in the single process stage. In that case, preferably the low magnesium fraction (eg limonite) is leached in a first step, and then the higher magnesium fraction is
10 subsequently added to the slurry to be leached in a second step. The sequential leaching of the low and high magnesium fractions may be in accordance with the disclosure of WO 03/093517, the entire disclosure of which is incorporated herein by reference.

15 Leaching is conducted for a period of time sufficient to release at least a substantial portion of the nickel and cobalt from the laterite ore into solution. Typically leaching is conducted for up to 30 hours. However, preferably leaching is conducted for up to 5 hours. More preferably, leaching is conducted for up to 4 hours. In a preferred embodiment, leaching is conducted for about 2
20 hours.

The leaching process typically also results in precipitation in at least some of the iron in the ore as one or more Fe containing compounds, such as a sulphate, a hydroxide or an oxide.

25 The mineral acid used in the leaching process is preferably sulphuric acid, more preferably it is concentrated sulphuric acid. The concentration of sulphuric acid added to the ore pulp is preferably greater than 90 wt %. The dose of sulphuric acid is preferably 100 to 140 % of the stoichiometric amount required to
30 dissolve approximately over 90% of nickel, cobalt, iron, manganese and over 80 % of the aluminum and magnesium in the ore.

The ratio of the high magnesium ore to low magnesium ore is ideally in a dry ratio of from 0.5 to 1.3. Preferably, the ratio is from 1 to 1.30. However, the

high/low magnesium ore ratio will largely depend on the laterite ore composition.

5 The leaching of both the high and low magnesium fractions may optionally be followed by a second leaching step. In the second leaching step, any unused acid from the first leaching step may be reacted with additional high magnesium ore fraction, such as saprolite. Leaching conditions of temperature, time and acid concentration are typically similar to those of the first leaching step.

10 Addition of saprolite can cause further precipitation of Fe containing compounds.

Conditions of temperature, time and acid concentration may conveniently be controlled to allow part or all of the iron and aluminum to be precipitated. The
15 acidity may be conveniently controlled by the addition of saprolite, MgO, Mg(OH)₂, MgCO₃ or another alkali. For example, the leach slurry may be treated in accordance with the method disclosed in WO 03/093517 (the entire disclosure of which is incorporated herein by reference), in which saprolite ore is added to a leach slurry in order to precipitate goethite or other relatively low
20 sulphate-containing forms of iron oxide or iron hydroxide. Alternatively, the leach slurry may be treated in accordance with the method disclosed in US 6,261,527 (the entire disclosure of which is also incorporated herein by reference) in which an iron precipitating agent selected from sodium, potassium ammonium ions and mixtures thereof is added to the slurry to precipitate
25 jarosite.

In a preferred embodiment, MgO is added to the slurry in order to precipitate iron containing compounds. Preferably, the MgO addition results in an increase of pH to a value of 3.0 or higher, causing iron precipitation.

30

The leached slurry is then treated to recover dissolved nickel and cobalt values therefrom. Such metal extraction treatment may be one or more of techniques known to those working in the art. Examples of such metal extraction techniques include ion exchange, resin-in-pulp, direct recovery by solvent

extraction, mixed hydroxide precipitation or mixed sulphide precipitation. Preferably the recovered nickel and cobalt values are recovered as mixed nickel/cobalt hydroxides or mixed nickel/cobalt sulphides.

- 5 Prior to or during recovery of nickel and cobalt from the leach liquor, the solid leach residue which usually includes precipitated iron compounds such as Fe sulphates eg jarosite or Fe hydroxides, eg goethite, may be removed from solution depending on the recovery process used. Alternatively, the solid residue may be retained with the leach solution during subsequent removal of
10 residual Fe and/or Al.

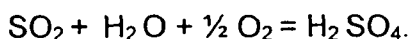
Prior to or after recovery of nickel and cobalt from the leach solution, the spent leach solution is preferably treated to remove any residual Fe and/or Al in solution. Typically, this step requires an increase in solution pH, such as by
15 adding a neutralising agent, such as MgO, Mg(OH)₂ or MgCO₃, and preferably addition of an oxidising agent such as air. Typically a sufficient quantity of neutralising agent is added such that the solution pH is increased to around 3 or above. A sufficient amount of the oxidising agent is also added to oxidise any residual Fe²⁺ in solution to Fe³⁺, which then precipitates out as goethite.

20 After removal of Ni, Co, Fe and Al from the spent leach liquor, the supernatant solution mainly contains dissolved magnesium, possibly together with a small quantity of manganese. The supernatant solution is then treated in order to recover the magnesium as magnesium salts. This is achieved typically by
25 evaporation until the magnesium salts crystallise out. Alternatively, reverse osmosis or precipitation by a strong alkali such as caustic soda, soda ash or lime, may be used.

The magnesium salt is typically a magnesium sulphate where the leaching acid
30 used was sulphuric acid. It has been the conventional practice to discard the magnesium salts as waste, meaning that metal values in the salts are therefore lost. Moreover, when the magnesium salt comprises magnesium sulphate, the sulphate component is also lost, which increases the acid requirement for the leaching process significantly. Sulphuric acid is usually an expensive input in

acid leaching, so there is an economic disadvantage in simply discarding a source of sulphate.

Accordingly, in a preferred embodiment of the process, the present invention is also concerned with treating the magnesium salt to recover magnesium compounds. Where the magnesium salt comprises $MgSO_4$, the recovery process also preferably includes a sulphate recovery stage. Preferably, the magnesium is recovered as a magnesium oxide, magnesium hydroxide or magnesium carbonate. More preferably, the magnesium is recovered as magnesium oxide. The magnesium recovery process may comprise that disclosed in co-pending Australian provisional patent application 2005900431 filed on 1 February 2005, the entire disclosure of which is incorporated herein by reference. Alternatively, the magnesium salt may be subjected to calcination. Where the magnesium salt is magnesium sulphate, calcination results in formation of MgO and/or $MgCO_3$ and SO_2 gas. The SO_2 gas may be captured and fed to a sulphuric acid production process, in which sulphuric acid is regenerated according to the following process:



20

The MgO , $Mg(OH)_2$ or $MgCO_3$ produced from the magnesium salt is a good source of alkaline compound, which can be fed back to the leach solution as a neutralising agent to effect precipitation, separately or in combination, metals such as Ni, Co, Al, Fe, Mn and other elements as desired.

25

Example 1

A mixture of limonite and saprolite ore in a dry ratio of about 1 is formed into an aqueous pulp. The aqueous pulp is then mixed with concentrated sulphuric acid, having a concentration of 93% H_2SO_4 , to form a leach slurry. The dose of acid is greater than 100% of the stoichiometric amount required to dissolve over 90% of the Ni and Co in the combined ore fractions. A first leaching process is conducted in a single reactor at a temperature of at least 80°C and for at least 2

30

hours. During the first leaching process, iron compounds precipitate out of solution.

5 Overflow from the leaching process is conveyed to a second reactor, where a saprolite ore slurry is added to the mixture. A second leaching process is then conducted, also at a temperature of at least 80°C and for a time of around 2 hours. During the second leaching process, further iron compounds precipitate out from solution.

10 After completion of the second leaching process, the solid residue is separated from the leached slurry. The pregnant leach solution is then subjected to a recovery process during which nickel and cobalt values are recovered.

15 The spent leach solution is also treated to remove any residual iron and aluminium. This is effected by the addition of a neutralising agent comprising MgO or MgCO₃. The pH of the barren solution is thereby increased, to a value higher than 3. The iron is precipitated largely as hydroxides, such as Fe(OH)₃.

20 At this time, the barren leach solution contains mainly dissolved magnesium. The spent leach solution is directed to an evaporation pond and excess water evaporated therefrom, causing crystallisation of magnesium sulphate.

25 The magnesium sulphate is then subjected to a magnesium recovery process. This comprises calcination to produce MgO, or MgCO₃, and SO₂ gas. The SO₂ gas is then used as a reactant in a sulphuric acid recovery process.

30 The following two Examples are concerned with the recovery of magnesium from magnesium sulphate crystals. Example 2 is a Comparative Example demonstrating calcination of MgSO₄·7H₂O under non-reducing conditions, which shows that MgSO₄ remains as the product. However, Example 3 demonstrates that calcination under reducing conditions achieves production of MgO at moderate temperatures, that is, at temperatures significantly lower than those at which calcination is conventionally conducted.

Example 2 – Comparative Example

A sample of magnesium sulphate heptahydrate (4.0353g) was placed in a small crucible and calcined in a thermogravimetric analyser (TGA) under a flow of dry nitrogen (5L/min). The temperature in the TGA was raised by 10°C/min from room temperature to 1000°C. The sample exhibited a weight loss of approximately 2.07g by 400°C and exhibited very little further weight loss. The resulting mass of the sample (1.9386g) corresponds closely with the formula MgSO_4 (theoretical weight of 1.9706g). A graphical depiction of the TGA run is shown in Figure 1.

10

Example 3

A sample of magnesium sulphate heptahydrate (4.0093g) was placed in a small basket and calcined in the thermogravimetric analyser (TGA) under a flow of dry hydrogen (5L/min). The temperature in the TGA was again raised by 10°C/min from room temperature to 1000°C. The sample exhibited a weight loss of approximately 2.03g by 350°C, corresponding to the loss of waters of crystallisation. The weight then remained stable until 630°C at which a further weight loss of approximately 1.06g. Rapid weight loss slowed at a temperature of 810°C. By the time 1000°C had been reached the total weight loss was approximately 3.29g. The remaining sample was carefully removed from the container and weighed. The mass of the weighed sample (0.63g) corresponds closely with the formula MgO (theoretical weight 0.655g). A graphical depiction of this run is shown in Figure 2.

25 Example 4

Example 4 is a Flowsheet, illustrated in Figure 3, which sets out the process stages in an embodiment of the present invention. In this Example, separation of the leach residue from the pregnant leach solution takes place prior to removal of residual iron and aluminium and recovery of Ni and Co metal values. The recovery of Ni and Co is effected using one of the techniques selected from mixed hydroxide precipitation, mixed sulphide precipitation, solvent extraction or ion exchange.

30

Example 5

Example 5 is a Flowsheet, illustrated in Figure 4, setting out the process stages in a further embodiment of the present invention. In Example 5, the Ni and Co metal values are recovered, using the Resin-in-Pulp (R-I-P) extraction technique, prior to removal of residual iron and aluminum, subsequent manganese precipitation and separation of leach residue from the barren solution.

The above description of the invention is illustrative of the preferred embodiments of the invention. Variations without departing from the spirit or ambit of the invention described herein are to be considered to form part of the invention.

The claims defining the invention are as follows:

1. An atmospheric leaching process in the recovery of nickel and cobalt from a lateritic ore, said lateritic ore including a low magnesium ore fraction and a high magnesium ore fraction, said process including the steps of:
 - 5 (a) forming an aqueous pulp of said lateritic ore,
 - (b) leaching said aqueous pulp with a concentrated mineral acid at atmospheric pressure to produce a slurry containing a pregnant leach liquor and a leach residue,
 - (c) treating the pregnant leach liquor either separately or as part of said
10 slurry to recover dissolved nickel and cobalt therefrom, leaving a magnesium containing barren solution,
 - (d) treating said magnesium containing solution to recover a magnesium containing salt therefrom.
2. The process of claim 1, further including the step of increasing the pH of
15 the pregnant leach liquor and/or the barren solution to produce an iron containing precipitate.
3. The process of claim 1 or 2, wherein both said low and high magnesium fractions are leached simultaneously during said leaching step (b).
4. The process of claim 1 or 2, wherein said low and high magnesium
20 fractions are leached sequentially during said leaching step (b).
5. The process of claim 1, wherein said leaching step (b) is conducted at a temperature above 60°C.
6. The process of claim 5, wherein said temperature is at least 80°C.
7. The process of claim 1, wherein the ratio of high magnesium ore to low
25 magnesium ore is in a dry ratio of from 0.5 to 1.3.
8. The process of claim 2, wherein the step of increasing pH comprises adding a neutralising agent to the pregnant leach liquor and/or the barren solution to increase the pH to a value of approximately 3 or above.
9. The process of claim 2 or 8 wherein the step of increasing pH also
30 comprises addition of an oxidising agent in order to oxidise any residual Fe^{2+} to Fe^{3+} and cause its precipitation as a hydroxide, such as $\text{Fe}(\text{OH})_3$.
10. The process of claim 9, wherein said oxidising agent is air.
11. The process of claim 1, wherein said pregnant leach liquor is treated to recover nickel and/or cobalt using one or more of the following methods: ion

exchange, resin-in-pulp, direct recovery by solvent extraction, mixed hydroxide precipitation or mixed sulphide precipitation.

12. The process of claim 11, wherein said pregnant leach liquor is treated to recover nickel and cobalt using mixed hydroxide precipitation or mixed sulphide precipitation.

13. The process of claim 1, wherein said magnesium containing salt is magnesium sulphate.

14. The process of claim 1, wherein the magnesium salt is treated to recover magnesium.

15. The process of claim 13, wherein said magnesium sulphate is treated to recover MgO, Mg(OH)₂ and/or MgCO₃.

16. The process of claim 15, wherein said magnesium sulphate is subjected to calcination to produce MgO and/or MgCO₃ and SO₂ gas.

17. The process of claim 16, wherein said SO₂ gas is fed to a sulphuric acid production process.

18. The process of claim 15, wherein said MgO, Mg(OH)₂ and/or MgCO₃ is used as said neutralising agent in step (c).

19. The process of claim 15, wherein said MgO, Mg(OH)₂, and/or MgCO₃ is used to precipitate iron, aluminium, and/or manganese from the pregnant leach liquor from (b) and/or the barren solution from (c).

DATED this 29th day of SEPTEMBER 2005

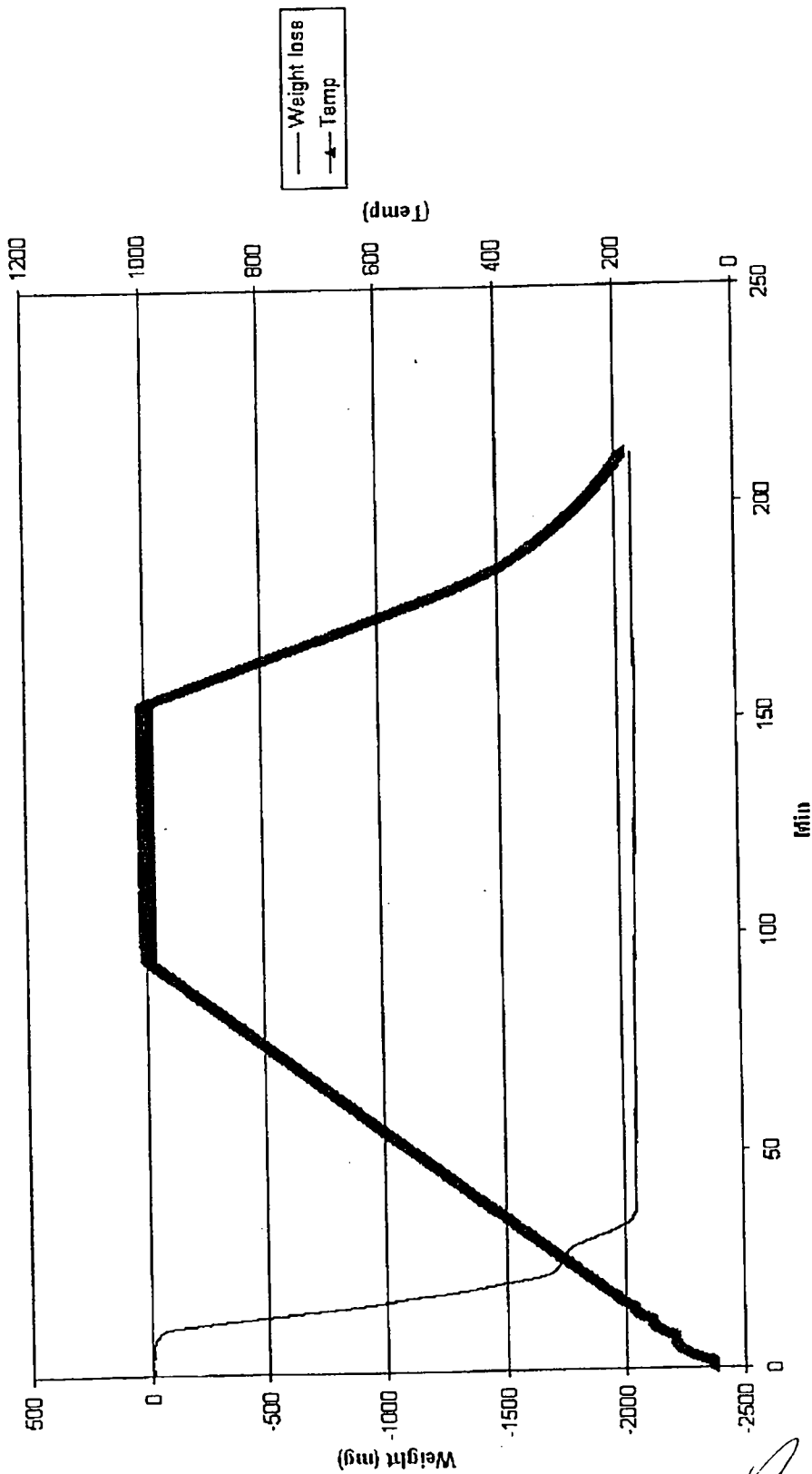
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Patent Agents for the Applicant

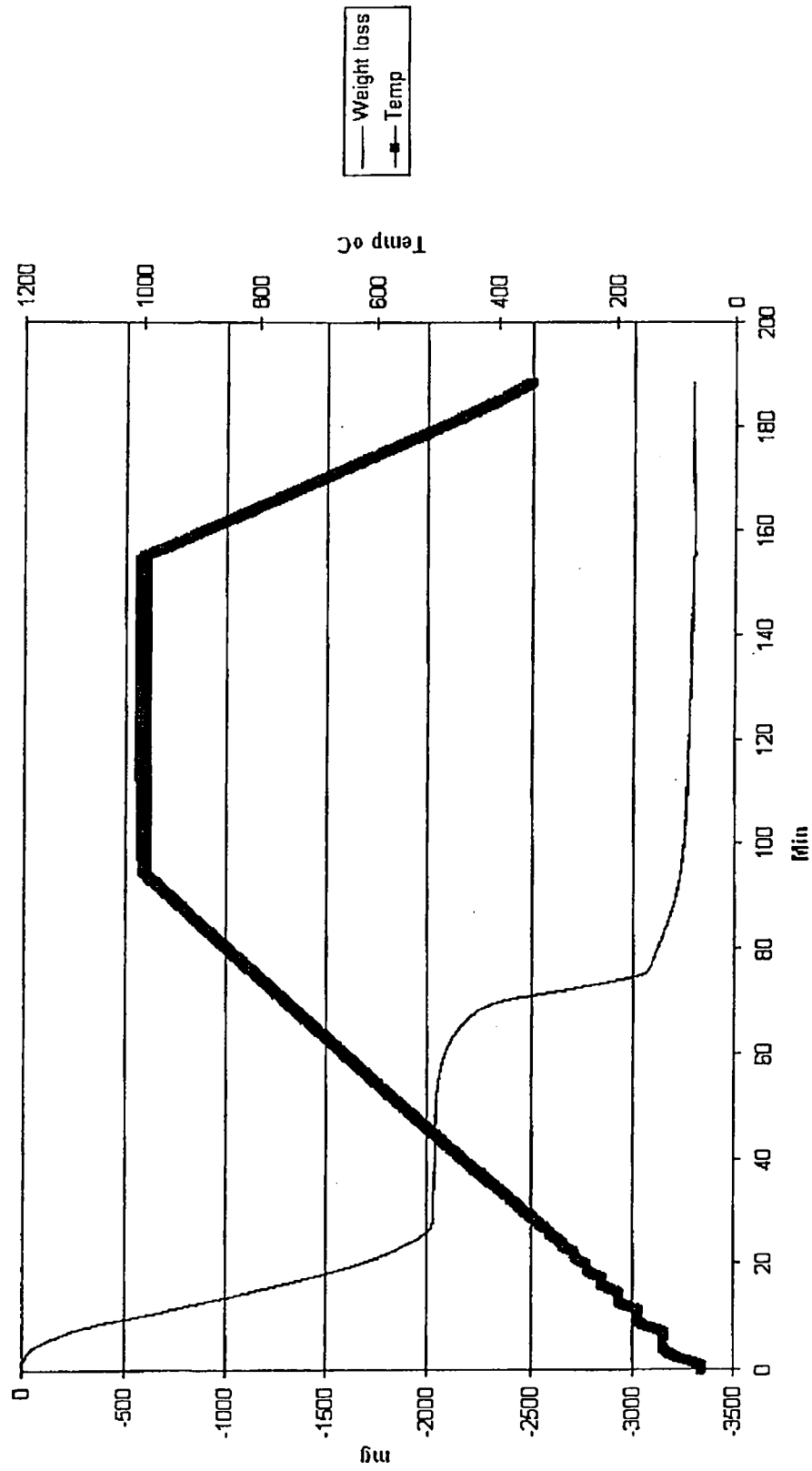
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Figure 1: MgSO₄.7H₂O calcined under N₂



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Figure 2: MgSO₄.7H₂O calcined under H₂



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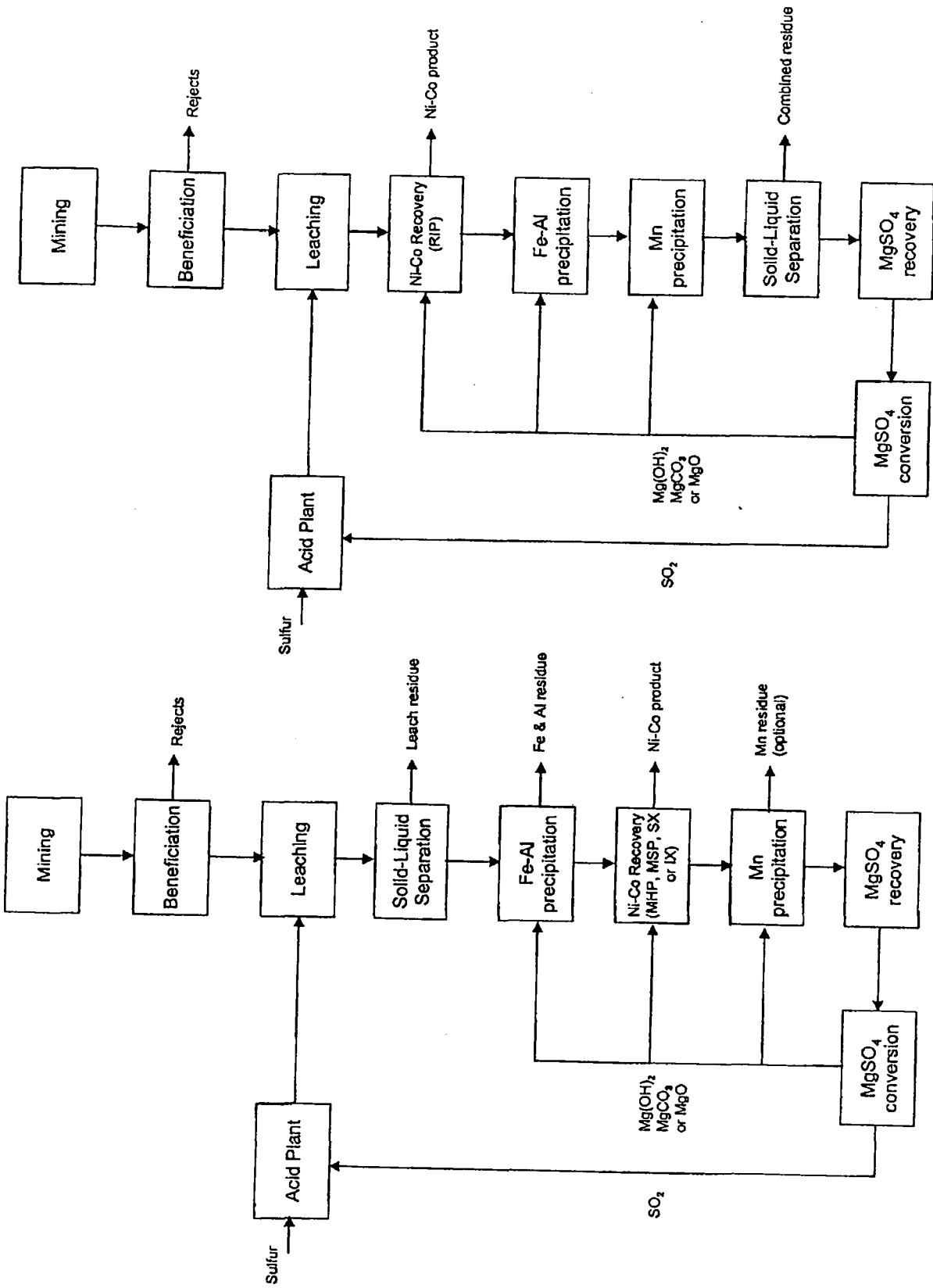


Figure 4 – RIP recovery of nickel and cobalt

Figure 3 – MHP, MSP, SX or IX recovery of nickel and cobalt

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