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Separation of Iron, Nickel, and Cobalt from Sulphated Leach Liquor of Low Nickel Lateritic Oxide Ore

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ABSTRACT

A two-stage selective precipitation technique was developed for the separation of iron, nickel, and cobalt from sulphated leach liquor of nickeliferous lateritic oxide ore obtained from Sukinda Valley, Orissa, India. In the first stage of precipitation, solid CaCO_3 is added to the sulphated solution at 90 to 95°C till the pH is adjusted in the range of 2.2 to 2.5. Most of the iron is precipitated out along with CaSO_4 . In the second stage, the residual iron and cobalt are separated from nickel by selective oxidation of cobalt. Bleaching powder and sodium oxychloride were separately used for this purpose. About 80 to 85% of the iron may be precipitated in the first stage, with practically no nickel or cobalt loss.

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Efficiency of iron precipitation in the first stage greatly depends on the initial iron concentration of the leach liquor. In the second stage, residual iron precipitation is complete along with 90% of total cobalt and 7 to 8% of total nickel.

Key Words: Iron; Nickel; Cobalt; Sulphated leach liquor; Low nickel lateritic oxide.

INTRODUCTION

Sulphuric acid leaching of nickeliferous lateritic oxide ores both at atmospheric and elevated pressure have long been identified as potential routes for the recovery of nickel and cobalt.^[1–4] During high temperature and pressure sulphuric acid leaching, iron is initially dissolved but subsequently most of it is hydrolyzed and precipitated out as hematite.^[4] In atmospheric pressure leaching, sulphuric acid consumption is more and understandably an appreciable quantity of iron also goes into the solution as sulphate along with nickel and cobalt.

Low-grade nickeliferous laterites of Sukinda Valley, Orissa, India, were extensively studied for both atmospheric and high-pressure sulphuric acid leaching.^[5–7] In both the cases, it was necessary to separate iron and cobalt from nickel to obtain purer products and value-added by-products.

Iron is generally separated from sulphate leach liquor of Ni and Co either by precipitation or solvent extraction.^[8–14] Precipitation processes most commonly used include goethite precipitation,^[9] jarosite precipitation,^[13] and phosphate precipitation.^[11] Lateritic oxide of Sukinda Valley also contains a significant amount of cobalt (about one tenth of nickel content) in addition to nickel, which also goes into the leach liquor as sulphate. Recovery of this cobalt as by-product is extremely important for technocommercial viability of any nickel extraction process following this route. However, separation of cobalt from nickel in acidic solutions is not very straightforward because of the closeness of their chemical properties. A large number of techniques for separating cobalt from nickel in various matrices have been listed in early published research.^[15] Some of the existing practices include xanthate precipitation,^[16] cementation,^[17] ion exchange,^[18] selective oxidation, and precipitation of Co(II) using Caro's acid,^[19,20] solvent extraction,^[21] and separation by supported and hybrid liquid membrane.^[22]

A bench-scale, two-stage selective precipitation technique is described in the present communication in which iron and cobalt were separated from nickel in the sulphated leach liquor of low nickel nickeliferous lateritic oxide ore obtained from the Sukinda Valley, Orissa. Most of the iron was taken out



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in the first stage, while remaining iron and about 90% of the total cobalt were recovered in the second stage. The objective of the present work was to develop a simple process for separating iron and cobalt from nickel to extract cobalt value and obtain main product nickel in a purer form.

EXPERIMENTAL

Raw Material

A low-grade nickeliferous lateritic oxide ore obtained from Sukinda Valley, Orissa was used for sulphation. Chemical composition of the ore is given in Table 1.

Sulphated Leach Liquor

Fe, Cr, Ni, and Co contents of the sulphated leach liquor used in the present study are shown in Table 2. This stock liquor was used throughout with appropriate dilution for the separation study.

Separation of Iron in First Stage of Precipitation

Iron separation experiments were carried out in batches using the leach liquor mentioned above (see Table 2). AR-grade CaCO_3 was used as the precipitant in first stage of precipitation. The parameters optimized were initial Fe concentration of the leach liquor, pH, and CaCO_3 dosing. For

Table 1. Complete chemical analyses of the COB ore.

Constituents	Percentage
Fe_2O_3	38.37
NiO	0.51
CoO	0.04
Cr_2O_3	3.35
SiO_2	50.41
Al_2O_3	2.04
MgO	0.20
LOI	6.14



Table 2. Chemical composition of stock sulphated leach liquor.

Constituents	g/L
Fe	27.3
Ni	0.43
Co	0.02
Cr	0.58

optimization, six different leach liquors with varying initial Fe concentrations were prepared from the stock leach liquor with appropriate dilution and were subjected to CaCO_3 dosing. Precipitation reactions were carried out at a temperature of 90 to 95°C under constant and uniform agitation. pH was measured at the end of the reaction. The reaction mixture was cooled and filtered. Both filtrate and residue were analyzed for Fe, Cr, Ni, and Co by atomic absorption spectrometry to obtain the material balance.

Separation of Cobalt

Ca(OCl)Cl (bleaching powder) and 4% NaOCl solution were independently used for separating cobalt from nickel. Bleaching powder solution was prepared by dissolving a definite amount of bleaching powder in water. The filtrate was analyzed for the chlorine content and used for cobalt separation.

Instrumental

All the metal analyses were carried out using a GBC Avanta Atomic Absorption Spectrometer. X-ray diffractograms were obtained from SEIFERT XRD 3003 PTS x-ray diffractometer.

RESULTS AND DISCUSSION

Figure 1 shows the x-ray diffractogram of the COB ore. Only two major phases could be observed. Fe was present as goethite [$\alpha\text{-FeO(OH)}$], while silica was present as quartz. Complete chemical analyses of the COB ore used for sulphation are given in Table 1. Besides Fe and Si present as majors, minor elements present in COB ore were Ni, Cr, and Al, while Co, Mn, and Zn were present in traces.



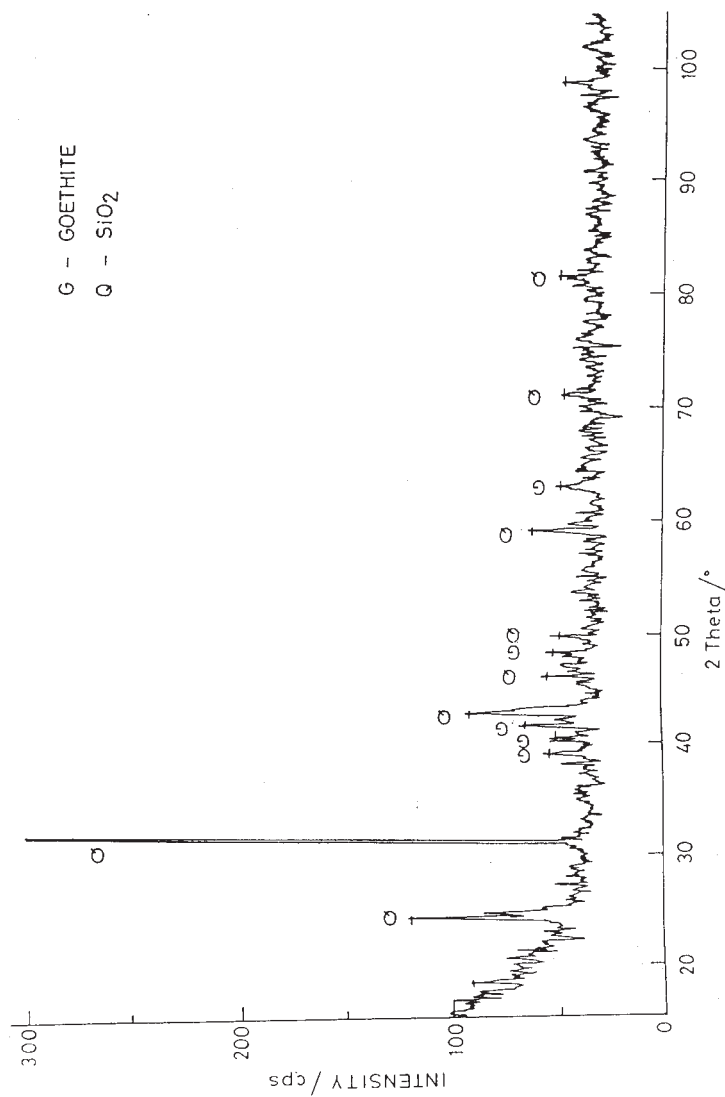


Figure 1. XRD spectrum of COB ore.



First Stage of Precipitation

Six different leach liquors with varying Fe concentrations of 27.3, 19.5, 13.7, 9.7, 6.9 and 4.9 g/L were prepared by appropriate dilution of stock leach liquor to optimize the initial Fe concentration, pH, and CaCO_3 dosing vis-à-vis iron recovery. The entire Fe concentration range was divided into two groups, concentrate and dilute group. Concentrate group comprised first three concentrations (27.3 to 13.7 g/L), while the remaining three were clubbed in the dilute group. The rise in solution pH with increasing CaCO_3 addition in both the groups is shown in Fig. 2. In the concentrate group, pH above 1.0 g of CaCO_3 was rather slow, while in the dilute group, the slope of the curve was markedly steeper beyond 1.5 g of CaCO_3 addition. This clearly indicated that the pH response to CaCO_3 addition was sharper in the dilute group. This is also apparent from the pH curves in Fig. 2 that for the same amount of CaCO_3 addition, pH rise was always more with higher dilution.

Metal recovery in six leach liquors at different pH are shown in Table 3. It may be seen from Table 3 that in the leach liquor containing 27.3 g/L Fe, only 63.5% Fe separates at a pH of 2.12, corresponding to a 3 g of CaCO_3 addition. However, for the same amount of CaCO_3 addition, iron separation was 83% and 80% for 19.5 and 13.7 g/L Fe respectively, at pH 2.24 to 2.25.

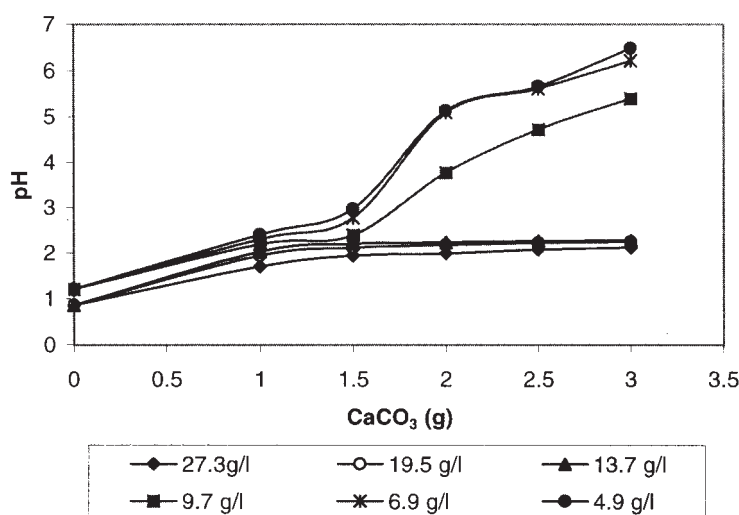


Figure 2. The pH curves for six different leach liquors.



Table 3. Metal recovery data in six leach liquors at different pH.

Initial Fe/Cr/Ni/Co concentration (g/L)	CaCO ₃ (g)	pH	Fe (%)	Ni (%)	Co (%)	Cr (%)
Fe: 27.3	0	0.86	0	0	0	0
Cr: 0.58	1.0	1.7	42.75	0	0	14.53
Ni: 0.43	1.5	1.94	48.71	0	0	15.54
Co: 0.025	2.0	1.99	48.45	0	0	28.87
	2.5	2.07	56.17	0	0	38.09
	3.0	2.12	63.50	0	0	41.75
Fe: 19.5	0	0.86	0	0	0	0
Cr: 0.41	1.0	1.94	43.45	0	0	19.49
Ni: 0.31	1.5	2.02	48.13	0	0	35.12
Co: 0.018	2.0	2.17	50.54	0	0	37.18
	2.5	2.22	59.01	0	0	41.67
	3.0	2.24	83.0	0	0	50.53
Fe: 13.7	0	0.86	0	0	0	0
Cr: 0.29	1.0	2.03	43.05	0	0	37.15
Ni: 0.21	1.5	2.18	50.66	0	0	35.09
Co: 0.012	2.0	2.20	55.12	0	0	38.76
	2.5	2.24	66.56	0	0	41.04
	3.0	2.25	77.06	0	0	46.72
Fe: 9.7	0	1.21	0	0	0	0
Cr: 0.19	1.0	2.19	55.62	0	0	55.09
Ni: 0.20	1.5	2.55	98.44	0.27	0	81.86
Co: 0.01	2.0	3.77	99.88	90.11	85.21	100
	2.5	4.72	99.99	97.54	95.87	100
	3.0	5.4	99.99	99.50	100	100
Fe: 6.9	0	1.21	0	0	0	0
Cr: 0.13	1.0	2.30	87.55	11.55	3.16	56.42
Ni: 0.14	1.5	2.77	98.63	15.17	8.11	78.88
Co: 0.007	2.0	5.09	99.33	65.7	60.99	100
	2.5	5.61	99.99	94.29	90.52	100
	3.0	6.21	99.99	98	98.35	100
Fe: 4.9	0	1.21	0	0	0	0
Cr: 0.009	1.0	2.40	87.37	16.28	2.58	45.82
Ni: 0.1	1.5	2.97	98.77	18.16	13.68	95.97
Co: 0.005	2.0	5.13	99.99	87.43	81.08	100
	2.5	5.66	99.99	98.69	97.85	100
	3.0	6.48	99.99	99.25	99.03	100



It was further interesting to note that in the leach liquor containing 19.5 g/L Fe, while 83% Fe got separated at 2.24 pH, practically all of the Ni and Co remained in the solution. Further, one may also find that about 50% of the Cr was separated along with Fe at pH 2.24. Corresponding Fe and Cr figures for 13.7 g/L Fe are, however, slightly inferior. In the dilute group as pH response with CaCO_3 dosing increases, Fe–Ni separation figures change accordingly. The most notable Fe–Ni separation could be observed with the leach liquor containing 9.7 g/L Fe. Approximately, 98% iron was separated at a pH of 2.55 corresponding to a 1.5 g of CaCO_3 addition with practically no nickel and cobalt loss. This, however, was an ideal situation and even a small pH deviation caused a significant nickel and cobalt loss.

Thus, in the first stage of iron precipitation using CaCO_3 as precipitant, it was optimized to operate on a leach liquor containing Fe in the range of 13 to 19 g/L. About 80% of the total iron could be separated out at a pH of 2.25 without any significant cobalt and nickel loss. Ideally it is possible to separate about 98% Fe without losing any Ni or Co by proper dilution and pH control. However, on dilution, pH control becomes extremely crucial and even a slight deviation may cause a serious Ni and Co loss. These are important engineering data to decide on the volume of the tank shell, cost of operation, etc. in a large-scale production.

It must be specifically mentioned at this stage that the entire first-stage precipitation was carried out under aerobic conditions. No attempt was made to convert entire iron into +III oxidation state, which might change the precipitation pattern. Conversion of entire iron to its higher oxidation state, however, runs the risk of oxidizing Cr, Ni, and Co also to their respective higher oxidation states, resulting into Co and Ni loss and generation of hazardous Cr(+VI).

The precipitate obtained from the neutralization of the leach liquor (Fe: 19.5 g/L, Cr: 0.41 g/L, Ni: 0.31 g/L, Co: 0.018 g/L) with CaCO_3 was investigated in detail. Figure 3 shows the x-ray diffractogram of the precipitate and Table 4 gives the complete chemical composition. The precipitate on drying becomes a soft and porous mass. It may be very easily crushed into fine powders. Principal mineral phases present in the dried precipitate were gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), silica as quartz (SiO_2), and mixed hydroxides of Ca and Fe [$\text{Ca}_3\text{Fe}_2(\text{OH})_{12}$]. It was most interesting to note that iron did not separate as isolated hydroxide of Fe(III). On the contrary, it was a mixed hydroxide of Ca and Fe. Complete chemical composition, given in Table 3, corroborates the x-ray pattern. It may also be seen from Table 3 that very little Ni and Co were lost during precipitation with CaCO_3 .

In the second stage of precipitation, attempts were made to separate out Co and residual iron from the leach liquor by oxidation of residual iron and selective oxidation of Co(II) using chlorine and hypochlorite separately.



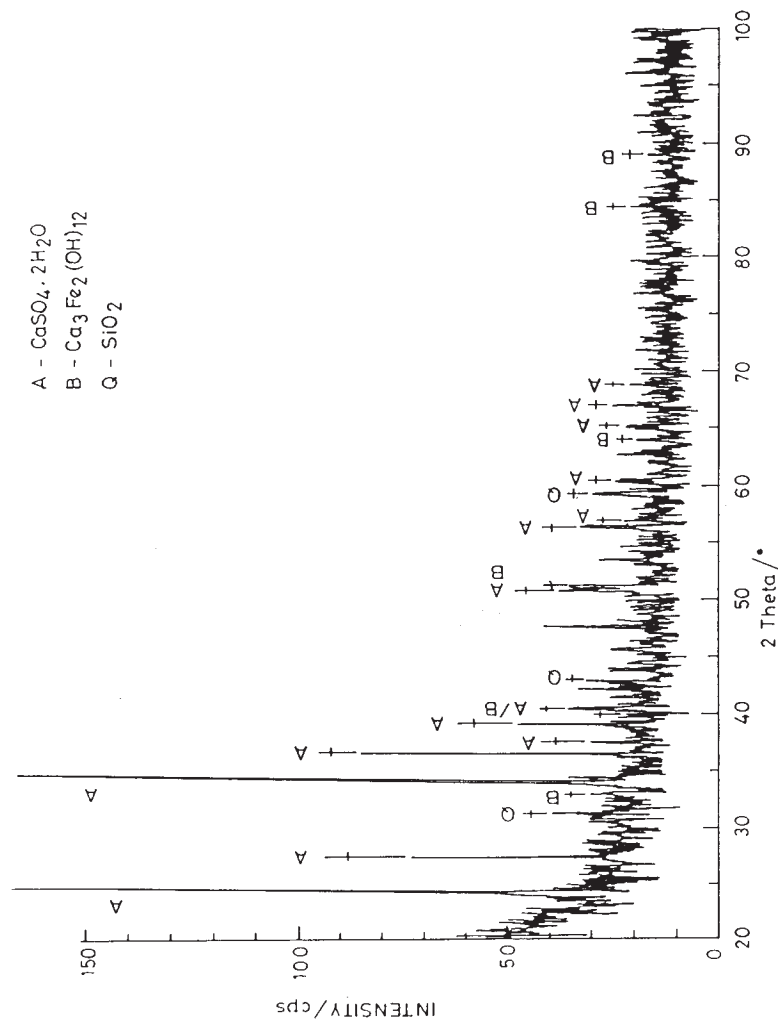


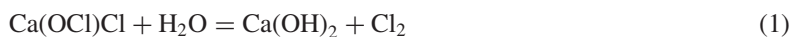
Figure 3. XRD spectrum of first-stage precipitate (after neutralization with CaCO_3).



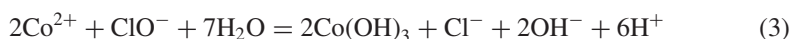
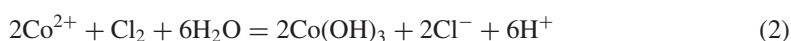
Table 4. Complete chemical analyses of the first-stage precipitate.

Constituents	Percentage
Fe ₂ O ₃	20.0
NiO	0.01
CoO	0.002
Cr ₂ O ₃	1.91
MnO	0.03
ZnO	0.009
CaSO ₄	47.74
CaO	12.04
SiO ₂	4.71
Al ₂ O ₃	0.49
MgO	0.16
LOI	12.35

Bleaching powder, Ca(OCl)Cl was used as the source of chlorine as given by the equation below,



and OCl⁻ was obtained from an aqueous solution of NaOCl. Oxidation of Co (II) to Co(III) with chlorine and hypochlorite are given by the following equations,



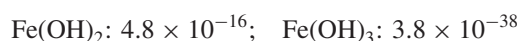
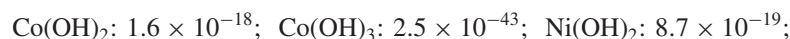
It may be seen that both oxidation reactions release H⁺ ions thus, lowering the pH of the resultant solution. It is imperative that the pH of the solution should be appropriately raised with a base to effect an efficient Co (OH)₃ precipitation which, however, is done in situ by Ca(OH)₂ in the first case and NaOH in the later. This may be noted in Eq. (3), that two equivalents of hydroxyl ions are also produced, which reduce the base requirement to raise the pH to a desired value. It is also understandable that the pH change in the case of Ca (OCl)Cl addition will be relatively slower as compared to NaOCl due to the sparingly soluble nature of Ca(OH)₂.



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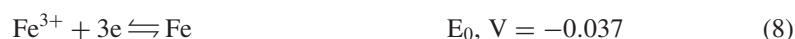
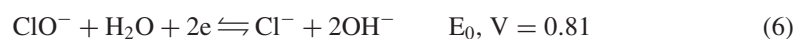
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The solubility products of hydroxides of Co(II), Co(III), Ni(II), Fe(II), and Fe(III) are given as follows:



It is evident from the solubility product data that under normal precipitation of hydroxides by raising the pH, it is not possible to separate Ni(II) and Co(II), though it may be possible to selectively separate the majority of iron from Ni(II) and Co(II) by proper pH control, as was done in the first stage of precipitation. It is also apparent from the solubility product data that if Co may be selectively oxidized to Co(III), then it may be possible to instantaneously precipitate out Co(III) keeping Ni(II) in solution.

It may be instructive at this point to look into the selective oxidation of Co(II) to Co(III) from the standard reduction potentials of the species involved as are given as follows:^[23]



It is evident from the present data that both Cl_2 and ClO^- can oxidize Co(II) to Co(III), albeit ClO^- can do it better. It is possible that Cl_2 or ClO^- may oxidize Ni^{2+} to its higher oxidation states, as nickel is known to exist in +III and +IV states also, though the +IV state is very rare.^[24] However, no research data were available on either reduction potentials of higher oxidation states of nickel and solubility products of their hydroxides and hydrated oxides.

It is apparent from the discussion that the most efficient separation of cobalt from nickel demands optimum combination of the right oxidant and pH. The amount of oxidant added should be just sufficient to oxidize the entire cobalt only and the pH should be the bare minimum for the precipitation of entire Co(III) and Fe(III). Results of our study on cobalt separation using bleaching powder and NaOCl are given in the following sections.



Cobalt Separation Using NaOCl

A 4% NaOCl solution was used for separating Co from the processed leach liquor obtained after first stage of precipitation with CaCO_3 . Different volumes of NaOCl solution were added to 25 mL of the processed leach liquor at pH 2.5 and pH changes were recorded. Co and Fe recovery and Ni loss were determined in the precipitate for every NaOCl addition. The results are shown in Fig. 4. It may be seen that at a pH of 4.7, corresponding to 15 mL of NaOCl, Co recovery was only 87.16% with 17.67% Ni loss. It is evident that 15 mL of 4% NaOCl was not sufficient to oxidize the entire Co(II), while at a pH of 4.7, some amount of Ni separated as hydroxide along with Co(III). It is also possible that some amount of Ni(II) was oxidized to Ni(III) and precipitated as hydrated oxide along with Co(III). To circumvent this problem, the following experiment was carried out. An excess of NaOCl solution was added to 25 mL of the processed leach liquor at pH 2.5 and the pH was raised to about 7. This was purposefully done to ensure the conversion of entire Co(II) to Co(III). A good amount of Ni also came out in the precipitate. However, the pH was again readjusted to a desired level by controlled addition of dilute HCl and Co, Fe, and Ni contents in the precipitate at the readjusted pH were determined. The results of this experiment are given in Table 5. It was gratifying to note that at a pH of 3.5, one might achieve about 99% Co recovery with about 9% Ni loss.

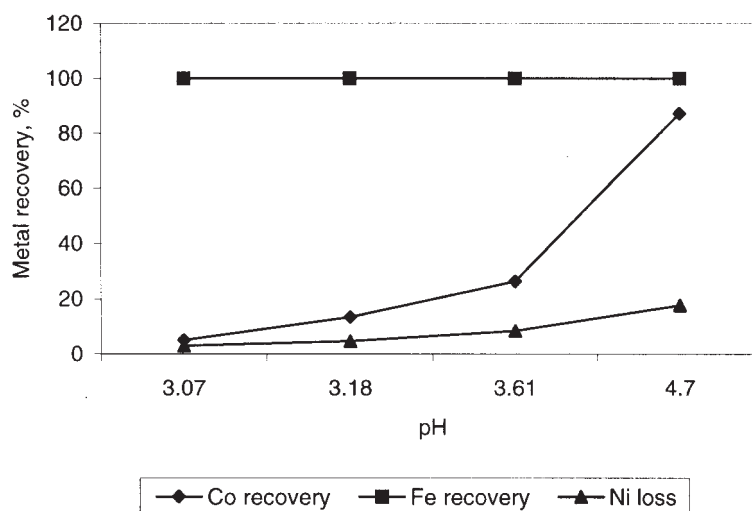


Figure 4. Metal recovery (%) with varying pH using NaOCl as oxidant.



Table 5. Cobalt and iron recovery in second-stage precipitation with NaOCl after pH re-adjustment.

Re-adjusted pH	Co recovery (%)	Fe recovery (%)	Ni loss (%)
3.0	94.81	100	9.61
3.5	98.83	100	9.15
4.0	99.73	100	14.83
4.5	100	100	14.88

The precipitate was dried at 105°C and calcined at 900°C and subsequently subjected to XRD analysis. The XRD pattern is shown in Fig. 5. It may be seen that all major peaks in Fig. 5 correspond to $\text{CoFe}_2\text{O}_4/\text{Fe}_2\text{O}_3$. It was gratifying to note that the calcined product was magnetic in nature indicating CoFe_2O_4 is the predominant phase as Fe_2O_3 is not magnetic.

Cobalt Separation Using $\text{Ca}(\text{OCl})\text{Cl}$

Aqueous solution of bleaching powder was used in the same manner as was done with NaOCl to separate cobalt and residual iron from the processed leach liquor obtained after the first stage of precipitation using CaCO_3 for oxidizing Co(II). Solid bleaching powder was not used to reduce contamination of cobalt and iron precipitate with Ca. The results are shown in Fig. 6.

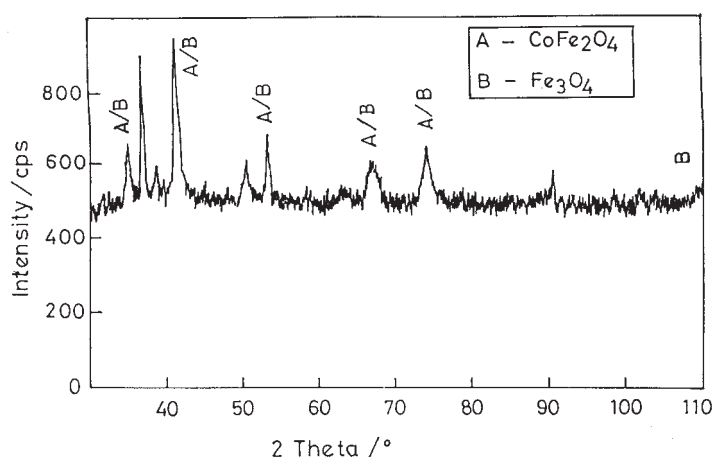


Figure 5. XRD spectrum of second-stage precipitate (after treating with NaOCl).



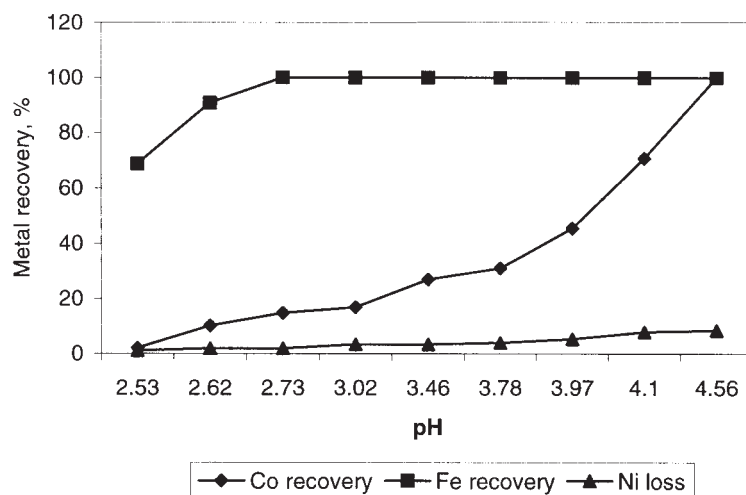


Figure 6. Metal recovery (%) with varying pH using $\text{Ca}(\text{OCl})\text{Cl}$ as oxidant.

It may be seen that the pH rise due to the bleaching powder addition was rather slow. For an addition of 9 mL of bleaching powder solution, equivalent to 110 mg of Cl_2 , almost complete cobalt separation could be achieved with about 8.49% Ni loss at a pH of 4.7. To reduce nickel loss without compromising cobalt recovery a similar experiment, as done with NaOCl , was carried out with $\text{Ca}(\text{OCl})\text{Cl}$. An excess of bleaching powder solution was added to 25 mL of the processed leach liquor to ensure complete oxidation of the entire cobalt, which raised the resultant pH to 7, causing a significant nickel loss. The pH of the solution was readjusted to lower values by the controlled addition of dilute HCl and Co, Fe and Ni contents in the precipitate at the readjusted pH were determined. The results are shown in Table 6. It was gratifying to note that at a pH of 4.0, nickel loss was 5.9% with Co recovery of 99.6%.

Table 6. Cobalt and iron recovery in second-stage precipitation with bleaching powder after pH re-adjustment.

Re-adjusted pH	Co recovery (%)	Fe recovery (%)	Ni loss (%)
3.0	86.76	100	0
3.5	93.47	100	4.63
4.0	99.58	100	5.88
4.5	100	100	8.42



pH of the filtrate obtained after separating Co for both $\text{Ca}(\text{OCl})\text{Cl}$ and NaOCl was raised to about 9 and a heavy black precipitate was obtained, which was in no way the commonly observed green $\text{Ni}(\text{II})$ hydroxide, at least by appearance. This black precipitate when dissolved in H_2SO_4 , however, regained the conventional green color of $\text{Ni}(\text{II})$ sulphate. This observation was somewhat similar to the oxidation of $\text{Ni}(\text{OH})_2$ with Br_2 solution in alkaline medium, which yielded $\text{Ni}(\text{III})$ oxide as $\text{Ni}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$ as a black solid that decomposed to NiO on dehydration.^[24]

Comparison of $\text{Ca}(\text{OCl})\text{Cl}$ and NaOCl performance clearly demonstrates that $\text{Ca}(\text{OCl})\text{Cl}$ is better than NaOCl though use of $\text{Ca}(\text{OCl})\text{Cl}$ severely contaminates both cobalt and nickel fractions with Ca, thus impairing the product purity. Ultimate technoeconomical evaluation of the process must consider this factor.

CONCLUSION

A two-stage precipitation technique based on the solubility products of hydroxides of Fe, Co, and Ni at different oxidation states was described for the separation of these elements in the sulphated leach liquor of a low nickel lateritic oxide ore obtained from Sukinda Valley, Orissa, India. In the first stage, iron was separated at a pH of 2.25 to 2.5 with CaCO_3 as the precipitant. Separation efficiency is greatly influenced by the initial Fe concentration in the leach liquor. More than 80% iron separation could be achieved with leach liquor containing 19.52 g/L Fe at a pH of 2.24 with practically no nickel and cobalt loss. Cobalt and residual iron in the processed leach liquor were separated from nickel through selective oxidation of iron and cobalt using $\text{Ca}(\text{OCl})\text{Cl}$ and NaOCl separately. Performance of bleaching powder was slightly better than NaOCl though cobalt and nickel fractions were severely contaminated with calcium.

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