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Effects of leaching parameters on the dissolution of nickel, cobalt, manganese and iron from Caldag lateritic nickel ore in hydrochloric acid solution

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ABSTRACT

The effects of leaching parameters on the metal dissolutions from Caldag laterite ore using hydrochloric acid at atmospheric pressure were investigated. The following leaching parameters were examined to understand their effects on the dissolution of the metals: hydrochloric acid concentration, solid/liquid ratio, particle size, leaching temperature and time. Extractions of 95.8%Ni, 94.5%Co and 94.3%Mn into the leach solution were obtained along with a substantial amount of iron (81.5%) under the following conditions: 3.0 M HCl concentration, 90°C leaching temperature, 8 h leaching time, 1/5 solid/liquid ratio and −0.053 mm particle size. The hydrochloric acid consumption under these optimum conditions was found to be 543 kg t^{-1} ore. The results indicated that hydrochloric acid concentration and leaching temperature were the most important parameters affecting metal dissolutions. It was found that the dissolution of nickel did not exhibit a good linear correlation to that of manganese, which suggested that considerable amounts of nickel were not hosted in asbolane phase but also in other mineral phases such as goethite, haematite and clays. It was, however, found that most of the cobalt appeared to be hosted in asbolane. The semi-quantitative mineral analyses revealed that mineral dissolution order was as follows: calcite > goethite > haematite > lizardite ≥ chlorite-serpentine > asbolane > albite > kaolinite.

On a investigué les effets des paramètres de lixiviation sur les dissolutions de métaux du minerai de latérite de Caldag à l'aide de l'acide chlorhydrique à la pression atmosphérique. On a examiné les paramètres de lixiviation qui suivent pour comprendre leurs effets sur la dissolution des métaux: concentration d'acide chlorhydrique, rapport solide/liquide, taille des particules, température et temps de lixiviation. On a obtenu des extractions de 95.8% de Ni, 94.5% de Co et 94.3% de Mn dans la solution de lixiviation ainsi qu'une quantité substantielle de fer (81.5%) dans les conditions suivantes: concentration de 3.0 M du HCl, température de lixiviation à 90°C, temps de lixiviation de 8 h, rapport solide/liquide de 1/5 et taille de particule de –0.053 mm. On a trouvé que la consommation d'acide chlorhydrique dans ces conditions optimales était de 543 kg/t de minerai. Les résultats ont indiqué que la concentration d'acide chlorhydrique et la température de lixiviation étaient les paramètres les plus importants affectant les dissolutions de métaux alors qu'il n'y avait pas d'effet bénéfique d'une taille de particule plus fine. On a constaté que la dissolution du nickel ne présentait pas une bomme corrélation linéaire avec celle du manganèse, ce qui suggère que des quantités considérables du nickel n'étaient pas retenues dans la phase asbolane mais également dans d'autres phases minérales comme la goéthite, l'hématite et les argiles. On a cependant constaté que la majeure partie du cobalt semblait retenue dans l'asbolane. Les analyses minérales semi-quantitatives ont révélé que l'ordre de dissolution des minéraux était le suivant: calcite > goéthite > hématite > lizardite > chlorite-serpentine > asbolane > kaolinite.

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Laterite; Caldag; hydrochloric acid; nickel dissolution; cobalt dissolution

1. Introduction

The importance of abundant low-grade lateritic nickel deposits as sources for future nickel production will increase as the conventional source of nickel, namely exploitable nickel sulphide ores, is dwindling. This is evident from the stagnant nickel production levels from sulphide ores and the exponential increase in nickel production tonnage from laterite ores in the last decade [1]. The laterite ores, however, require more complex processing as they are not as readily amenable to beneficiation as nickel sulphide ores, which are fed to smelters. Hence, the use of a hydrometallurgical route is becoming more important compared to the pyrometallurgical route as the proportion of nickel produced from lateritic

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ores increases. In addition, the hydrometallurgical route is the only one that can recover cobalt as a separate valuable product from these ores.

The only operating commercial hydrometallurgical process to treat laterite nickel ores to date is high pressure sulphuric acid leaching (HPAL). Research activities have increased to improve the sustainability, efficiency and economics of the process, which was first commercialised some sixty years ago. Despite this and many years of operational experience, this process remains marginal in terms of process economics as it requires expensive equipment such as titanium-lined autoclaves and high energy consumptions. Besides, it can only be applied to the processing of ores containing low concentrations of acid consuming minerals (magnesium, calcium, etc.) since acid is a major component of the process operating cost [2]. As such, HPAL is suitable only for treating limonite ores often leading to minimal utilisation of the saprolite component in the ore bodies. Given these problems, there is a growing interest in novel leaching techniques particularly those operating at atmospheric pressure conditions to make lateritic nickel ores more economically attractive [2–12].

Dry [13] compared the economics of various laterite treatment processes using some of the established hydrometallurgical and pyrometallurgical processes including two processes that are still under development, namely the Neomet process with hydrochloric acid as lixiviant [14] and the DNi process with nitric acid as lixiviant [8]. He reported that HPAL offers the best financial solution for treating limonites if the profitable byproducts cannot be obtained by the Neomet and DNi processes. The Neomet and DNi processes, could, however, offer distinctly superior economics compared with HPAL if the magnesia and haematite byproducts were marketable, especially if the processed haematite can be used as a feed in the ironmaking industry. Furthermore, for treating saprolites, heap leaching with sulphuric acid offers the best economics among all the considered processes but the Neomet process offers better economics if the byproducts are saleable.

It has been demonstrated that atmospheric pressure acid leaching using hydrochloric acid as lixiviant, such as the Neomet process, can produce nickel and cobalt recoveries comparable to HPAL [4]. One of the major drawbacks of the use of this acid is that it has poor selectivity against iron and therefore, dissolves essentially all iron in the ores along with nickel and cobalt resulting in high level of ferric chloride in the pregnant leach solution [9,14,15]. It is, however, possible to recover the acid from the ferric chloride while producing haematite in the process by heating the ferric chloride solution at about 185°C at atmospheric pressure in a circulating matrix of an inert molten salt hydrate [14]. Hence, it appears that hydrochloric acid leaching is capable of treating dissimilar ores using a single flowsheet without prior heat treatment or high pressure which lead to high energy consumption and thus offers an economical alternative for the extraction nickel and cobalt [16].

Although the corresponding author has published a number of papers on the dissolution of nickel and cobalt from Caldag laterite ore in sulphuric acid or organic acid solutions, the dissolution order of mineral phases in hydrochloric acid solution have not been previously studied by this research group. In the current paper, the effect of various operating parameters in atmospheric pressure acid leaching with hydrochloric acid, namely acid concentration, temperature, leaching time and ore feed size were investigated. In addition, mineralogical characterisation of the leached residues, an indispensable step to understanding the leaching process, was carried out using X-ray diffraction (XRD) analysis to understand the order of mineral dissolution during the hydrochloric acid leaching process.

2. Materials and method

The nickel laterite ore was supplied by Caldag Nikel in Turkey. The ore was crushed in a laboratory jaw crusher and then ground in a laboratory ball mill. The milled ore was dry-sieved to obtain different size fractions. An X-ray fluorescence spectrometry (XRF, Philips PW-2404) was used for the determination of the chemical composition of the size fractions. An X-ray diffractometer (Bruker Discover) was utilised to identify the mineralogical composition of the different size fractions. The mineral phases were determined by using Diffrac Suite EVA software equipped with the current PDF-2 database. In order to verify the XRD results, a representative ore sample was analysed by field-emission scanning electron microscopy with energy dispersive X-ray (FESEM-EDX, Zeiss GeminiSEM 300).

The tests were conducted using a 250-mL Erlenmeyer flask which was placed on a digital temperature controlled hot plate equipped with a magnetic stirrer. In order to minimise evaporation, a condenser was fixed to the flask. The test set-up is schematically illustrated in Figure 1. During a test, 100 mL of a pre-determined concentration of hydrochloric acid solution was put into the flask. The temperature of the hot plate was increased to the target level. Then, the ore sample was added into the flask to start the test. After the leaching was completed, the slurries were vacuum-filtered through Whatman 1 filter paper. The residues were washed with deionised water. The elemental analysis of the filtrate was carried out using atomic absorption

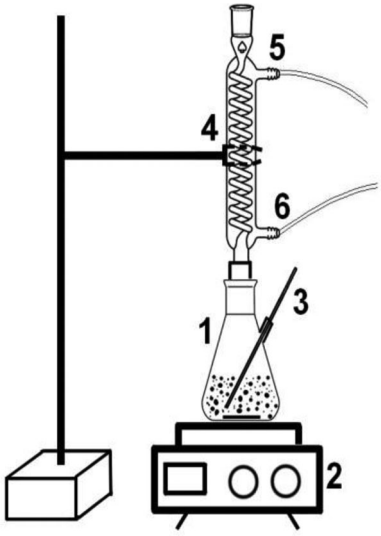

Figure 1. Schematic of the experimental setup (1 – Erlenmeyer flask, 2 – Hot plate, 3 – Glass thermometer, 4 – Glass condenser, 5 – Tap water outlet, 6 – Tap water inlet).

spectroscopy (AAS, Thermo Scientific ICE 3300). For mass balance calculation, the washed and dried leach residue was digested in aqua regia. The changes in the mineralogical composition of the solid phase were identified by XRD analysis.

In the leaching experiments, analytical grade of hydrochloric and nitric acids were used. Deionised water was used for dilution when needed. The hydrochloric acid consumption at the best condition was determined via titration method using 0.1 N sodium hydroxide titrant and di-potassium oxalate monohydrate to complex interfering ions, particularly iron [17]. In the leaching experiments, the solid/liquid ratio and mixing speed were fixed at 1/5 (w/v) and 300 rev min^{-1}, respectively. The leaching tests were carried out in duplicate to confirm the repeatability of the experimental results.

3. Results and discussion

3.1. Materials characterisation

The chemical compositions of the lateritic nickel ore at different particle sizes are presented in Table 1. The XRD analysis of the −0.053 mm fraction (Figure 2) shows that major phases are quartz, albite, calcite and goethite, while kaolinite, haematite, chlorite-serpentine, lizardite and asbolane are relatively minor or trace components. Note that the percentage mineral compositions produced by the semi-quantitative mineral analysis reflects only the percentage of the phases relative to all the other phases that were considered in the analysis. Arslan et al. [18] and Helvaci et al. [19] indicated that the nickel and cobalt bearing mineral in Caldag ore was asbolane. Onal and Topkaya [20] reported that nickel ore was also linked with the goethite/haematite and clay minerals. Basturkcu et al. [21,22] demonstrated that the nickel and cobalt hosting compounds in the laterite ore fall in the limonite group, iron hydroxides, hydrosilicates, clay, oxy/hydroxide and chromite, while the gangue minerals were determined to be in the chromite group minerals, quartz, clay minerals (smectite, chlorite, halloysite, montmorillonite), haematite, magnetite, calcite, feldspar, muscovite and dolomite. Semi-quantitative mineral analysis of the −0.053 mm fraction is shown in Table 2. A representative SEM-EDX map is given in Figure 3. The presence of sodium in the sample confirms that albite is present. Cobalt was excluded from the mapping analysis as it was a trace element in the ore sample as well as embedded in the coarse and compact manganese oxide particles [23]. FT-IR analysis on the same laterite ore has been reported by Kursunoglu et al. [24].

3.2. Effect of hydrochloric acid concentration on metal dissolutions

The experiments were carried out using the −0.053 mm ore sample varying the hydrochloric acid concentration from 1.0–3.0 M at 90°C for 8 h. Figure 4 depicts the effect of hydrochloric acid concentration on the metal dissolutions. The dissolutions increased with as hydrochloric acid concentration increased. The dissolution of iron rose significantly from 12.2% at 1.0 M HCl to 81.5% at 3.0 M HCl. The dissolution of nickel, cobalt and manganese also raised though to a lesser extent than iron from 43.1, 47.8 and 65.1%, respectively, at 1.0 M to 95.8, 94.5 and 94.3% at 3.0 M. These results

Table 1. Chemical composition of the lateritic nickel ore at different particle size fractions from XRF analysis.

	Chemical content (% mass)												
Particle sizes (mm)	MgO	Al_2O_3	SiO_2	SO_3	K_2O	CaO	TiO_2	Cr_2O_3	MnO	Fe_2O_3	Co_2O_3	NiO	LOI[a]
−212 + 150	2.14	2.02	57.97	0.08	0.24	2.59	0.08	1.19	0.25	25.31	ND	0.92	7.00
−0.150 + 0.106	3.01	1.95	53.01	0.09	0.27	2.91	0.06	1.23	0.33	28.18	0.09	1.14	7.73
−0.106 + 0.075	3.37	2.01	51.12	0.09	0.27	2.99	0.09	1.08	0.37	28.54	0.09	1.23	8.11
−0.075 + 0.053	4.17	2.53	48.82	0.08	0.29	3.10	0.13	1.14	0.37	29.14	0.10	1.28	8.58
−0.053	4.59	2.54	45.12	0.10	0.30	3.41	0.09	0.86	0.37	31.69	0.09	1.45	9.39

[a]Loss on ignition, ND: below detection limit (1 ppm).

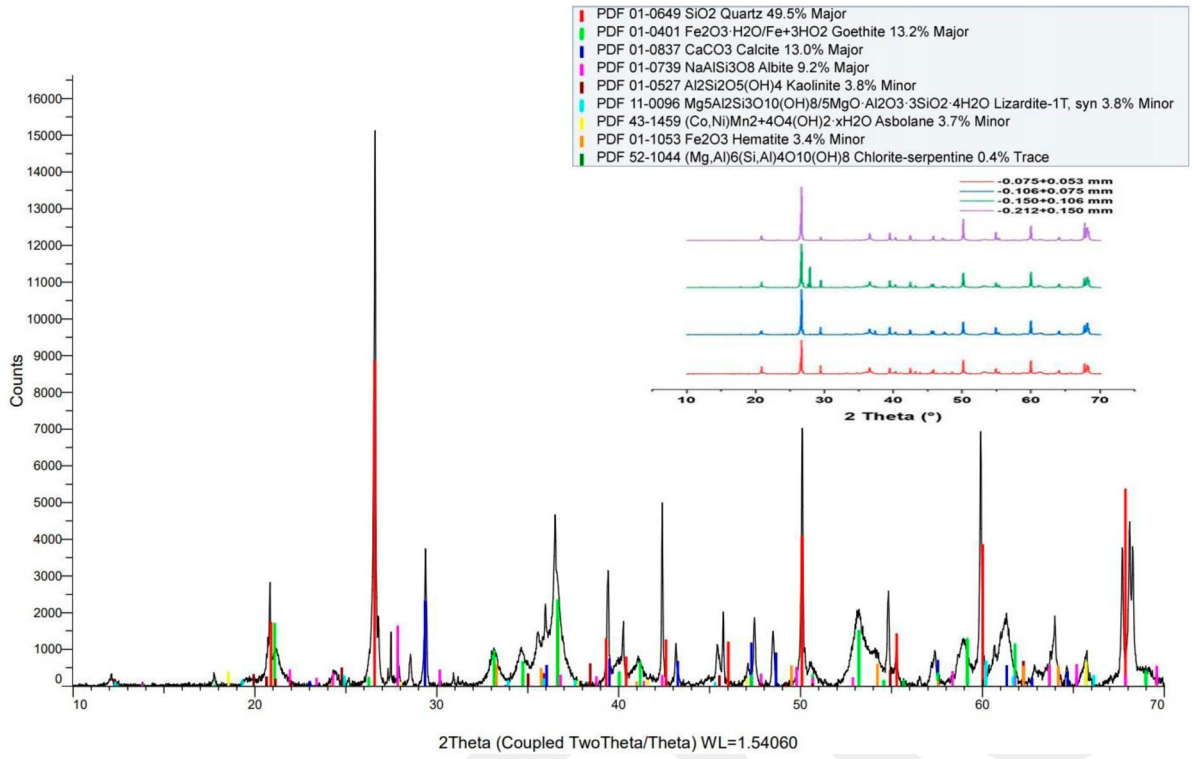

Figure 2. XRD patterns of the −0.053 mm size fraction and with identified minerals. The patterns for the other size fractions are shown as an inset.

are consistent with the mineralogical analysis of the residues, presented in Figure 5 which showed increasing dissolution of nickel and cobalt containing minerals as the acid concentration was increased. The semi-quantitative XRD analyses of the leach residues are given in Table 3. At 2M HCl concentration, the order of mineral dissolution from most to least was found to be calcite > lizardite ≥ chlorite-serpentine > haematite > kaolinite > asbolane > albite > goethite. When the acid concentration increased from 2 to 3 M, the dissolution order of the mineral phases was determined as calcite > goethite > haematite > lizardite ≥ chlorite-serpentine > asbolane > albite > kaolinite, which is close agreement with the findings of Li et al. [25]. In order to decrease the iron content in the leach solution for downstream processing such as solvent extraction (SX) and to avoid more residual acid recycling, the best acid concentration was determined to be 3 M. The iron, aluminium and chromium content in the leach solution can be removed by partial neutralisation before the SX step.

Table 2. Semi-quantitative mineral analysis of different size fractions by XRD.

	Particle sizes (mm)				
Minerals (%)	−0.212 +0.150	−0.150 +0.106	−0.106 +0.075	−0.075 +0.053	−0.053
Quartz	74.5	69.6	63.8	52.1	49.5
Calcite	6.8	8.9	15.6	12.3	13.0
Goethite	5.0	5.3	5.9	10.2	13.2
Albite	4.6	9.4	5.1	7.0	9.2
Asbolane	3.1	1.4	2.0	2.7	3.7
Kaolinite	2.3	1.7	2.7	8.2	3.8
Haematite	1.2	1.9	1.6	2.5	3.4
Lizardite	2.3	1.4	2.3	4.9	3.8
Chlorite-serpentine	0.2	0.4	1.0	0.2	0.4

3.3. Effect of leaching temperature on metal dissolutions

The tests were carried out for 8 h at 3.0 M hydrochloric acid at various temperatures ranging from 30–90°C using the −0.053 mm nickel laterite ores. Figure 6 shows the effect of temperature on metal dissolutions over the studied temperature range. The increase in operating temperature had a significant positive effect on the metal dissolutions. It is known that high temperatures under atmospheric conditions achieve high nickel and cobalt dissolutions from laterites [18,21–23]. The nickel and cobalt per cent dissolutions were greater than for iron at all temperatures. After 8 h of leaching duration, 34.7% Ni, 39.7% Co, 62.4% Mn and 12.5% Fe were dissolved at 30°C. Dissolutions increased significantly to 95.8% Ni, 94.5% Co, 94.3% Mn and 81.5% Fe at 90°C. The nickel and cobalt dissolutions are below 90% when the leaching temperature is less than 90°C. Thus, to achieve high nickel and cobalt dissolution, a leaching temperature of 90°C was selected for further

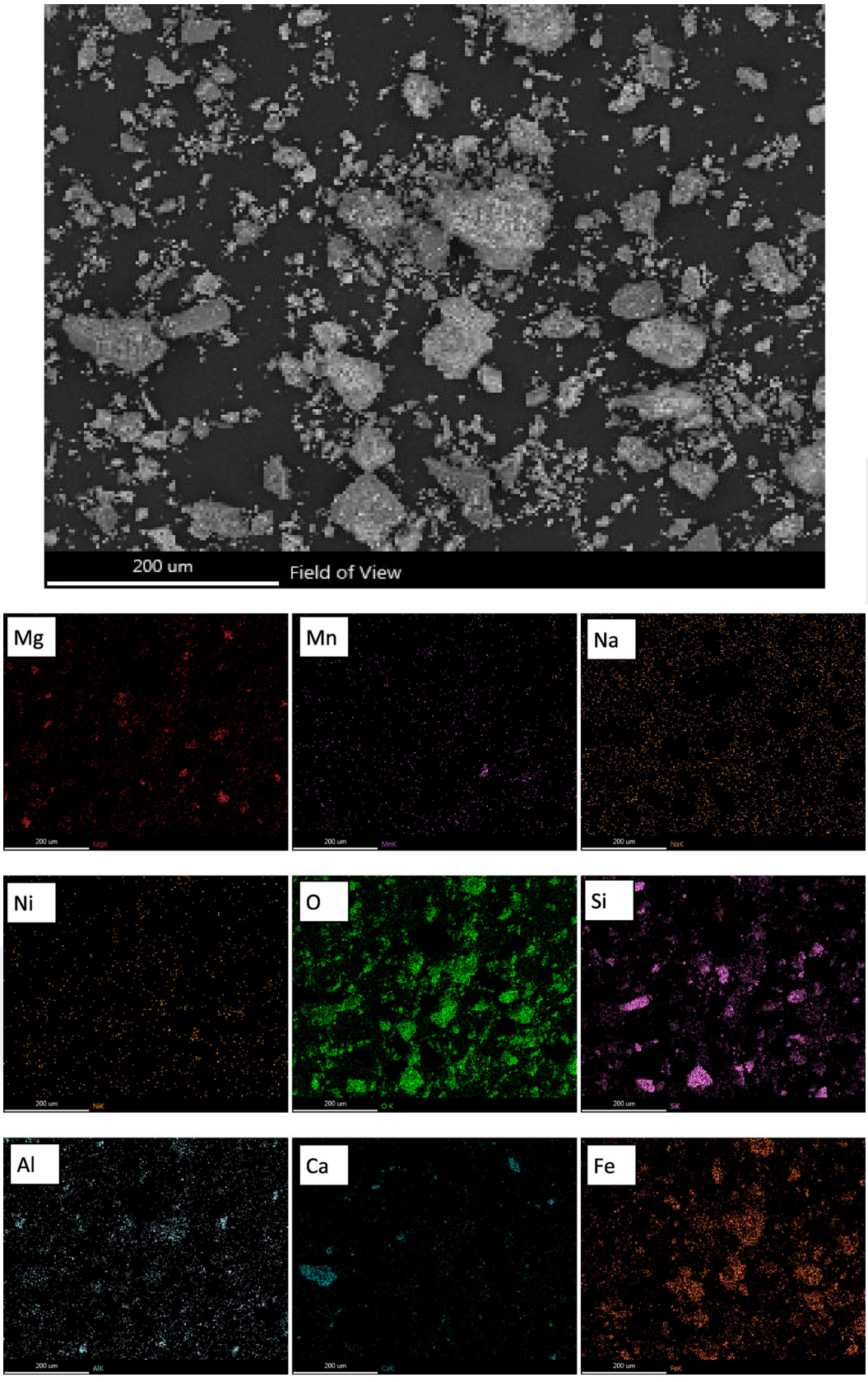

Figure 3. SEM-EDX mapping of the representative ore sample.

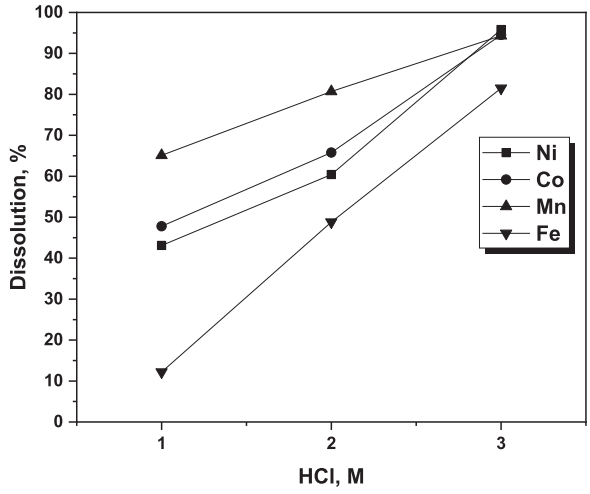

Figure 4. Effect of acid concentration on metal dissolutions.

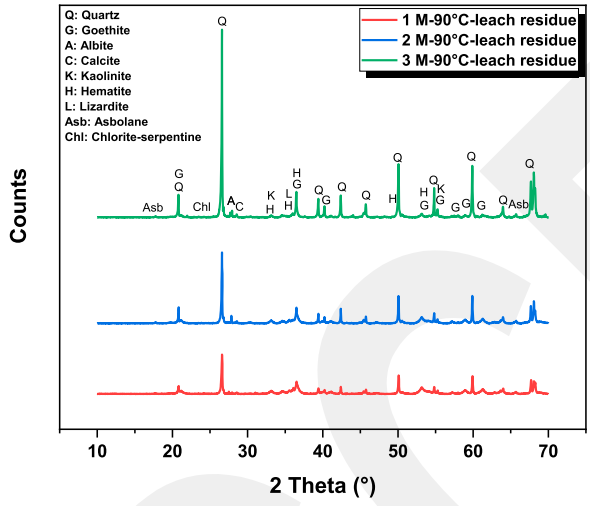

Figure 5. XRD patterns and semi-quantitative mineral analysis of the solid phase after leached using hydrochloric acid at a concentration ranging from 1.0–3.0 M and temperature of 90°C for 8 h.

studies. Figure 7 demonstrates the XRD pattern of the leach residues at different leaching temperatures. The semi-quantitative analyses of the leach residues are

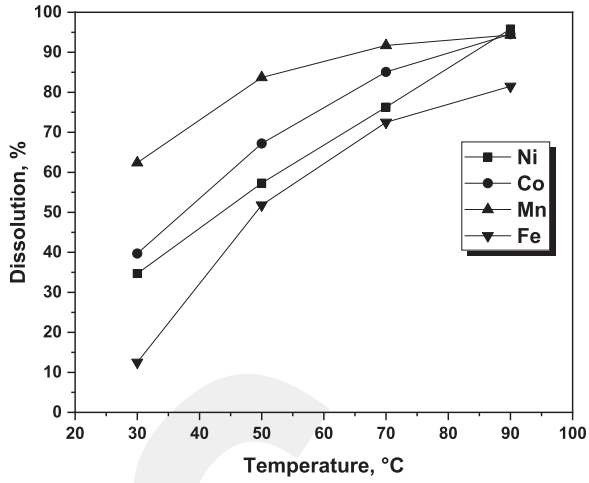

Figure 6. Effect of leaching temperature on metal dissolutions.

given in Table 4. Goethite, the dominant iron bearing mineral contents in the ore, was dissolved from 13.2% in the feed down to 4.6% in the leach residue. Haematite in the feed at 3.4% decreased to 1.2% in the residue. Lizardite decreased to 1.9% in the residue from 3.8% in the feed, a slightly lesser proportion dissolved than for haematite. Serpentine mineral phases significantly decreased from 0.4% in the feed down to 0.2%, very similar in proportion leached to lizardite. The leach residue was relatively enriched in quartz, increasing to 79.8% in the residues since this mineral is essentially not leached in the acidic solution. Calcite was leached at much larger extent than the other crytalline phases. Asbolane mineral phase significantly decreased from 3.7% down to 1.9%. From this table, goethite, haematite, lizardite, chlorite-serpentine and asbolane mineral phases decreased significantly with increasing leaching temperatures, which indicated that nickel, cobalt, manganese and iron were taken into the leach solution.

3.4. Effect of leaching time on metal dissolutions

The effect of leaching time on the metal dissolutions was studied at 90°C using the −0.053 mm particle size

Table 3. Semi-quantitative mineral analysis of the solid phases after leaching at various acid concentrations ranging from 1.0–3.0 M and temperature of 90°C for 8 h.

		Residues		
Minerals (%)	Feed ore	1.0 M	2.0 M	3.0 M
Quartz	49.5	58.6	68.4	79.8
Calcite	13.0	4.7	2.4	2.1
Goethite	13.2	15.2	9.8	4.6
Albite	9.2	7.0	9.8	5.7
Asbolane	3.7	2.9	2.7	1.9
Kaolinite	3.8	4.3	2.6	2.6
Haematite	3.4	3.8	2.2	1.2
Lizardite	3.8	3.3	1.9	1.9
Chlorite-serpentine	0.4	0.2	0.2	0.2
For mass balance	Feed Weight:20 g	Residue Weight: 17.30 g	Residue Weight: 15.22 g	Residue Weight: 10.86 g

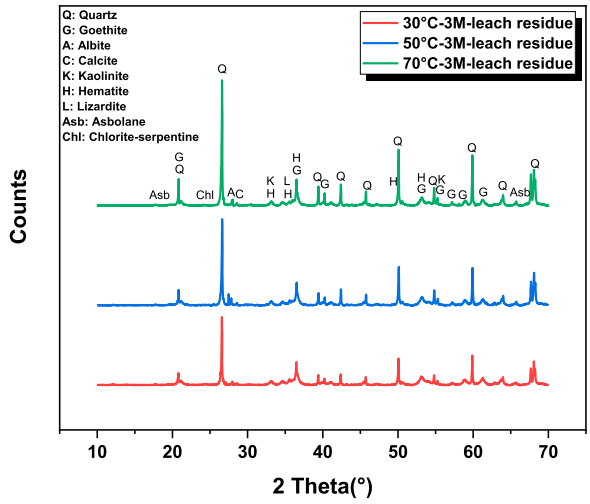

Figure 7. XRD patterns and semi-quantitative mineral analysis of the solid phase after leached using 3 M hydrochloric acid at a temperature ranging from 30 to 70°C for 8 h.

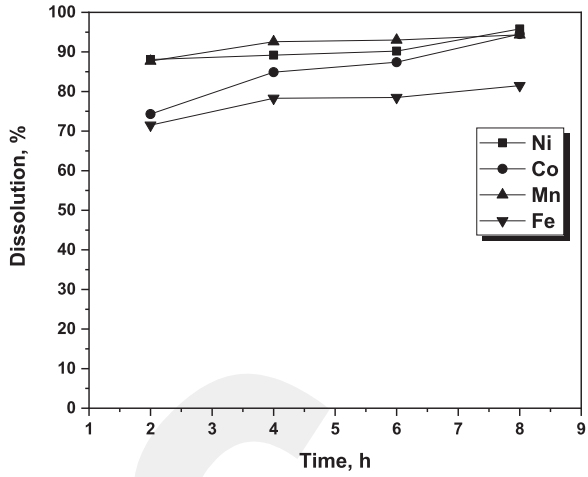

Figure 8. Effect of leaching time on metal dissolutions.

fraction and 3.0 M hydrochloric acid. Figure 8 shows the effect of leaching time on the metal extractions from the laterite ore. The nickel, cobalt, manganese and iron dissolutions slightly increased from 88.1 to 95.8%, 74.3 to 94.5%, 87.7 to 94.3% and 71.5 to 81.5%, respectively, when the leaching time increased from 2 to 8 h. This is probably because of refractory type haematite mineral phase [26] and forming a diffusion layer on particle surfaces by means of a substantial amount of silica in the ore [27]. The metals may also be inaccessible to the lixivant, e.g. locked within a mineral grain. The greatest nickel and cobalt dissolutions were achieved at 8 h. Figure 9 shows the nickel and cobalt dissolution as a function of manganese dissolution. It can be seen that nickel and manganese dissolutions did not have a good linear correlation against leaching duration suggesting that a considerable amount of the nickel was hosted in the phases other than asbolane such as goethite and haematite [18,20,23] and clay and/or serpentine mineral phases [21,22]. Cobalt dissolution, however, showed relatively linear correlation with that of manganese suggesting that most of cobalt is embedded in asbolane.

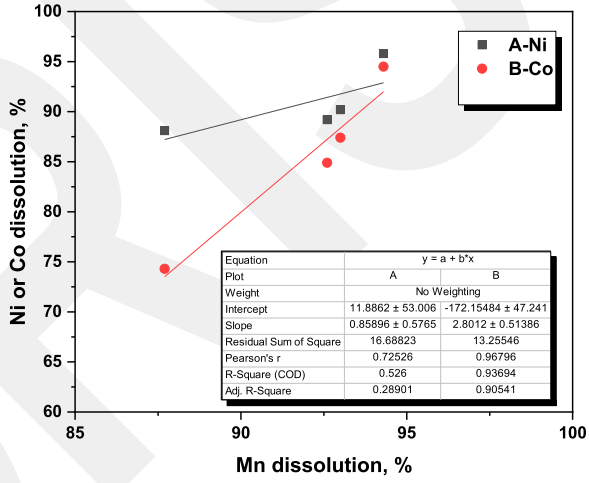

Figure 9. Nickel or cobalt dissolution versus manganese dissolution.

3.5. Effect of particle size on metal dissolutions

The effect of particle size on metal dissolutions was tested at 90°C and a hydrochloric acid concentration of 3.0 M. Figure 10 shows that there was no notable effect of particle size on metal dissolutions. This is in good

Table 4. Semi-quantitative mineral analysis of leached residues for different leaching temperatures.

		Residues			
Minerals (%)	Feed ore	30°C	50°C	70°C	90°C
Quartz	49.5	62.0	63.7	74.3	79.8
Calcite	13.0	3.5	3.1	2.5	2.1
Goethite	13.2	13.2	12.2	8.7	4.6
Albite	9.2	7.9	8.4	5.7	5.7
Asbolane	3.7	3.0	3.6	2.4	1.9
Kaolinite	3.8	3.9	3.2	2.5	2.6
Haematite	3.4	3.3	2.6	1.9	1.2
Lizardite	3.8	3.1	2.8	1.8	1.9
Chlorite-serpentine	0.4	0.3	0.3	0.2	0.2
For mass balance	Feed Weight:20 g	Residue Weight: 16.55 g	Residue Weight: 13.54 g	Residue Weight: 11.87 g	Residue Weight: 10.86 g

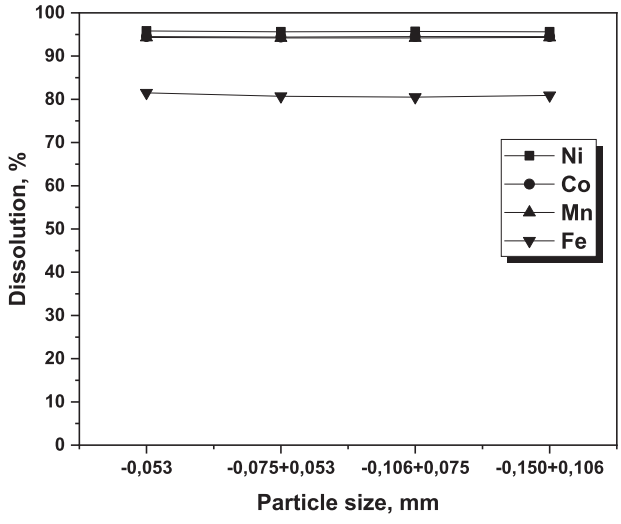

Figure 10. Effect of particle size on metal dissolutions.

agreement with the findings of Kursunoglu and Kaya [23] for sulphuric acid leaching of the same laterite ore. Previous researchers have indicated that in general, coarser laterite ore particles are easily leachable due to the irregular silica and porous goethite mineral phases [28–30]. The cobalt and manganese dissolutions showed a similar trend when particle size of the sample increased as the cobalt is most probably associated with asbolane mineral phases.

4. Conclusions

High metal dissolutions were achieved from the nickel laterite ore using hydrochloric acid under atmospheric pressure conditions. The effects of leaching time temperature, acid concentration and particle size on metal dissolutions were experimentally investigated. The highest dissolutions in the present study for −0.53 mm material using a solid/liquid ratio of 1/5 and 8 h leaching time were obtained with 3.0 M hydrochloric acid, at, 90° C. Under these experimental conditions, 95.8% Ni, 94.5% Co, 94.3% Mn and 81.5% Fe were extracted into the leach solution. The increases in the metal dissolutions as a function of time were relatively small after the first 2 h of leaching probably because the remaining metals were hosted in relatively refractory mineral phases. The obtained data demonstrated that hydrochloric acid concentration and leaching temperature had a strong effect on metal dissolution. The analysis of the correlation of nickel and cobalt with manganese dissolutions indicated these metals were not only hosted in the asbolane phase but were also presented in goethite, haematite and clay mineral phases. The semi-quantitative XRD analysis of the leach residue revealed that the dissolution order of the minerals was calcite > goethite > haematite > lizardite ≥ chlorite-serpentine > asbolane > albite > kaolinite.. The hydrochloric acid consumption at the preferred conditions from the range tested was found to be 543 kg t^{-1} ore. Based on the experimental results obtained from the present study, hydrochloric acid could be used as an alternative lixiviant for nickel and cobalt dissolutions from the Caldag laterite ore at atmospheric pressure leaching condition.

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Disclosure statement

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