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HIGH PRESSURE SULPHURIC ACID LEACHING OF LATERITIC NICKEL ORE

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Abstract: In this study, the effects of acid/ore ratio (0.1–0.6 g H₂SO₄/g dry ore), leaching temperature (200–240 °C) and leaching time (30–120 minutes) on the high pressure sulphuric acid leaching of a lateritic nickel ore sample having 1.37% Ni, 0.076% Co and 47.72% Fe₂O₃ were investigated. The leaching experiments showed that the increase of leaching temperature and leaching time increased continuously the amount of dissolved Ni and Co, and decreased continuously the amount of dissolved Fe from the lateritic nickel ore sample. With the increase of acid/ore ratio, dissolution ratio values of Ni and Co increased up to acid/ore ratio of 0.3, and above 0.3, lower dissolution ratio values were obtained. Although leach residues obtained after leaching at acid/ore ratio of 0.3 contained mainly hematite, quartz and anhydrite, the use of higher amounts of acid (e.g. 0.6) caused the formation of basic ferric sulphate, verified by the sulphur contents and the X-ray diffraction patterns of leach residues. The dissolution ratio values of 86.4%, 92.8%, 95.1% and 96.6% Ni were obtained after 30, 60, 90 and 120 minutes of leaching, respectively, at acid/ore ratio of 0.3 and leaching temperature of 240 °C. In this study, the leaching behaviours of Mg, Ca, Mn, Al and Cr were also determined.

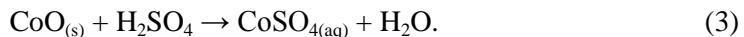
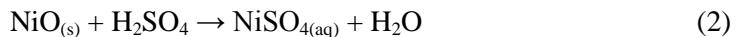
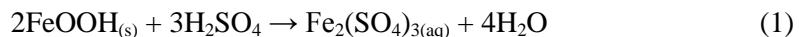
Keywords: lateritic nickel ore, pressure leaching, goethite, hematite, basic ferric sulphate

Introduction

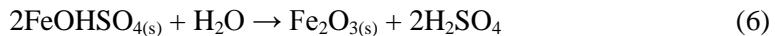
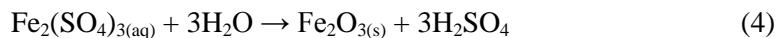
The lateritic deposits constitute the largest reserves (72%) of the world's nickel resources, but only 42% of the primary nickel production comes from these lateritic ores and the remainder is supplied from the sulphide ores (Oxley and Barcza, 2013). Because conventional mineral processing techniques (e.g. flotation) can not successfully be used for the concentration of lateritic nickel deposits, extractive metallurgical (e.g. hydrometallurgical) methods are applied to obtain nickel and cobalt from these type of ores (Zubryckyj et al., 1965; Queneau, 1970; Onodera et al., 1987; Whittington and Muir, 2000; Muir and Johnson, 2006; Quast et al., 2015a,b,c; Farrokhpay and Filippov, 2016). Among these, the high pressure sulphuric acid

leaching is one of the established and commercially used method to treat nickeliferous laterite ores (Carlson and Simons, 1960; Chou et al., 1977; Kuxmann and Landau, 1981; da Silva, 1992; Briceno and Osseo-Asare, 1995; Georgiou and Papangelakis, 1998; Rubisov et al., 2000; Whittington and Muir, 2000; Whittington et al., 2003; Whittington et al., 2005; Loveday, 2008; Chalkley et al., 2010; Guo et al., 2011; Kaya and Topkaya, 2011; Liu et al., 2012; Korkmaz, 2014; Onal and Topkaya, 2014).

In high pressure acid leaching processes, lateritic nickel ores are interacted with aqueous sulphuric acid solutions in acid resistant autoclaves at temperatures of 230–270 °C and under pressures of 3.3–5.5 MPa for 60–180 minutes. Under these leaching conditions, iron mainly follow a dissolution-precipitation path, forming insoluble solid oxides and sulphates, while nickel and cobalt dissolve and stay in the solution phase. By this way selective separation of nickel and cobalt can be achieved. As a main component in the ores, iron in the goethite dissolves to give ferric sulphate according to Eq. 1. Nickel and cobalt, present in the structure of goethite, are assumed in the form of oxides (or hydroxides), dissolve according to Eq. 2 and Eq. 3, respectively, and stay in the solution as sulphates



Following the dissolution of goethite, ferric ions produced hydrolyse rapidly and precipitate either as hematite (Eq. 4) or as basic ferric sulphate (Eq. 5), which can transform to hematite (Eq. 6) depending on leaching conditions. The formation of basic ferric sulphate is favoured under very acidic leaching solutions. If high salinity (e.g. Na₂SO₄, NaCl) process water is used, then jarosite forms (Eq. 7). These precipitation reactions regenerate the acid consumed during initial goethite dissolution (Voigt and Gobler, 1986; Georgiou, 1995; Georgiou and Papangelakis, 1998; Whittington and Muir, 2000; Kambossos, 2005; Jankovic, 2010; Liu et al., 2010; Crundwell et al., 2011)



In our previous study (Girgin et al., 2011), we investigated the effects of sulphuric acid concentration (5–95%), leaching temperature (20–95 °C) and leaching time (30–240 min) on the atmospheric pressure sulphuric acid leaching characteristics of a lateritic nickel ore. In this study, the effects of acid/ore ratio, leaching temperature and

leaching time on high pressure sulphuric acid leaching of a local lateritic nickel ore sample having 1.37% Ni were investigated by using chemical and X-ray diffraction analyses. Other than nickel, the dissolution ratio values of cobalt, iron, magnesium, calcium, manganese, aluminium and chromium were also determined.

Experimental

Materials

Merck grade concentrated sulphuric acid, distilled water and a ground lateritic nickel ore sample taken from mid-Anatolia region of Turkey were used in leaching experiments. The particle size analysis of the ground sample showed that 100%, 80%, 50% and 20% of the sample were finer than 305, 144, 84 and 46 μm , respectively. The main chemical components of the sample were 29.44% SiO_2 , 4.02% Al_2O_3 , 47.72% Fe_2O_3 , 1.56% MgO , 1.91% CaO , 2.38% Cr_2O_3 , 0.75% MnO , 1.37% Ni, 0.076% Co, 0.03% S and 10.20% loss on ignition. The low amounts of MgO and high amounts of Fe_2O_3 indicated the limonitic character of the ore sample. Mineralogically, the sample contained mainly goethite and quartz, together with lower amounts of carbonates (calcite, dolomite), smectite, hematite and asbolane (Fig. 1a). As seen in Fig. 1a, no X-ray diffraction (XRD) peak belong to any separate nickel mineral was observed, indicating that nickel is found in the structures and/or adsorbed on the surfaces of goethite, smectite and/or asbolane phases, as also observed in commercial nickel laterite ores (Whittington and Muir, 2000). The endothermic peaks at 118.52 °C and 297.22 °C in the DTA curve and corresponding weight losses of 4.62% and 5.61% observed between 25-200 °C and 200-450 °C in the TG curve (Fig. 1b) of the ore sample belong to moisture removal and goethite ($\alpha\text{-FeOOH}$) dehydroxylation, respectively (Schwertmann, 1984; Balek and Subrt, 1995; Mitov et al., 2002; Prasad et al., 2006; Lopez et al., 2008). Low intensity peaks between 450-1000 °C (corresponding weight loss: 2.85%) in the DTA curve are due to polymorphic transformation of quartz, dehydroxylation of smectite and decomposition of carbonates (Grim, 1968; Patterson and Swaffield, 1987; Papadopoulou et al., 2006). The moisture content of the ore sample was 3.71%.

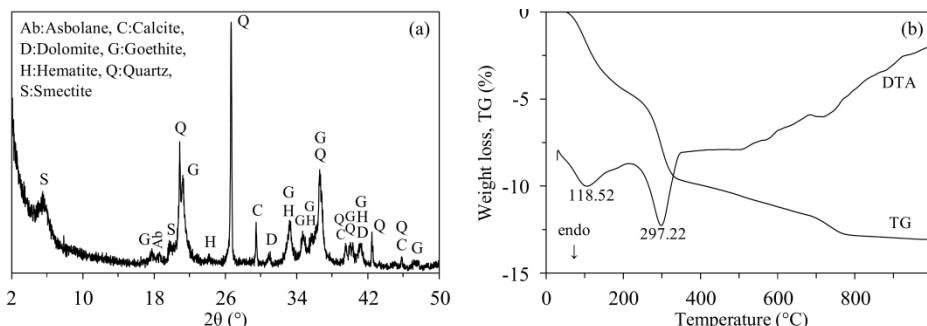


Fig. 1. XRD pattern (a) and TG/DTA (b) curves of lateritic nickel ore sample

Methods

Leaching experiments were performed in a 600 cm³ bench top Parr autoclave (Model: 4563) with a glass liner. The ore sample (40 g) dried at 105±5 °C was added to 120 cm³ aqueous sulphuric acid solution at room temperature in the autoclave, which was prepared by diluting Merck grade concentrated sulphuric acid with distilled water according to the desired acid/ore ratio (mass ratio H₂SO₄ / dry lateritic ore sample). The autoclave was closed properly, put into the heating mantle, made ready for a high-pressure leaching test. Then, the autoclave was heated up to the target temperature in the presence of constant agitation provided by a four-blade dual titanium impeller. When the target temperature was reached, the leaching time was started. At the end of the desired leaching time, the agitation was stopped and the autoclave was allowed cool naturally down to room temperature. The leach residues were separated from the suspension by filtration, washed, dried at 105±5 °C, weighed and then stored in sealed bottles for further XRD (Rigaku, CuKα) and chemical (Leco for S analyses and ICP-OES for other elements) analyses. The dissolution ratios (%) of nickel and other components in the ore sample were calculated according to Eq. 8. In order to ascertain reproducibility, some of the randomly selected leaching tests were repeated and the experimental error was found to be always less than ±4%.

$$\text{Dissolution ratio} = \frac{(\text{amount of Ni in ore sample}) - (\text{amount of Ni in leach residue})}{(\text{amount of Ni in ore sample})} \cdot 100 \quad (8)$$

Results and discussion

Effects of acid/ore ratio

Among the most important parameters that affect high-pressure acid leaching, the acid/ore (a/o) ratio was investigated in the first place at the values of 0.1, 0.2, 0.3, 0.4, 0.5 and 0.6 g H₂SO₄ per g of dry ore sample. The values were selected according to the existing literature. During these experiments, the leaching temperature and the leaching time was kept constant at T = 240 °C and t = 60 minutes, respectively. The changes in the dissolution ratios of Ni, Co and Fe, and the changes in the dissolved iron/dissolved nickel (dissolved Fe/Ni) ratio following high pressure leaching of the lateritic nickel ore sample at different a/o ratios were given in Fig. 2 and Fig. 3, respectively. It was seen from Fig. 2 that when the a/o ratio increased from 0.1 to 0.3, the dissolution ratio of Ni increased from 57.0% to 92.8%. Similarly, the dissolution ratio of Co increased from 48.1% to 94.3%. The dissolution ratio values of more than 90% obtained for both Ni and Co at a/o ratio of 0.3 were similar to the values reached both in laboratory and industrial scale studies performed under different experimental conditions (Carlson and Simons, 1960; Whittington and Muir, 2000; Georgiou and Papangelakis, 2009; Chalkley et al., 2010; Secen, 2011). With the increase of a/o ratio from 0.3 to 0.6, similar reductions were observed in the dissolution values of Ni and Co were observed. This may be caused by the phases (hematite, amorphous silica,

basic ferric sulphate) that precipitate in the solution during leaching and remove some part of the dissolved Ni and Co from the solution (Whittington and Johnson, 2005; Liu et al., 2010).

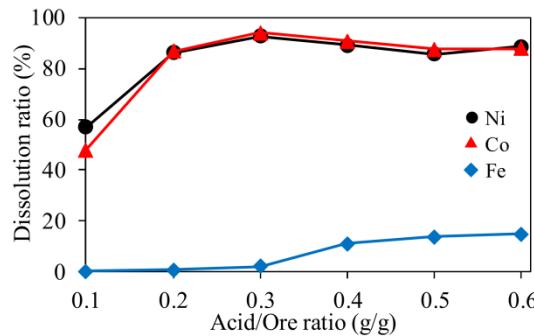


Fig. 2. The effect of acid/ore ratio on dissolution ratios of Ni, Co and Fe

The dissolution ratio of Fe increased slowly from 0.2% to 2.2% with increasing a/o ratio from 0.1 to 0.3 (Fig. 2). The increase of the sulphuric acid amount in the leach solution increased the amount of dissolved Fe, e.g. 14.7% Fe dissolved at a/o ratio of 0.6. Because the amount of Fe in the leach solution is one of the most important parameters that affect downstream processing, the ratio of dissolved Fe/Ni was also calculated (Fig. 3). It was found that the ratio of dissolved Fe/Ni increased slowly up to a/o ratio of 0.3 and further increase in a/o ratio increased greatly the ratio of dissolved Fe/Ni, which resulted in a contaminated pregnant leach solution and increased acid consumption.

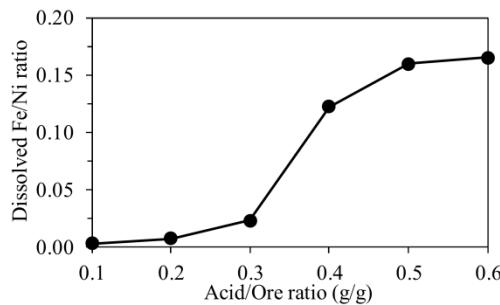


Fig. 3. Effects of acid/ore ratio on dissolved Fe/Ni ratios

In addition to Ni and Co, the dissolution ratios of Mg, Ca, Mn, Al and Cr were also determined (Fig. 4). Although having different dissolution values, Mg (mainly from smectite and dolomite) and Mn (mainly from asbolane) had similar leaching behaviours, increasing up to a/o ratio of 0.3, and they had nearly constant values above a/o values of 0.3. There were no XRD peaks indicating smectite, dolomite and asbolane in the pattern of leach residues obtained after leaching at a/o ratios of equal

or greater than 0.3 (see Figs. 6c, d and e). Although not investigated in detail, in this study, Co was observed to exhibit similar dissolution behaviour with Mn (and correspondingly asbolane ($\text{Mn}(\text{O},\text{OH})_2 \cdot (\text{Co},\text{Ni},\text{Mg},\text{Ca})_x(\text{OH})_{2x} \cdot n\text{H}_2\text{O}$)), as also observed in the related literature studies (Kuxmann and Landau, 1981; Whittington and Muir, 2000; Georgiou and Papangelakis, 2009; Nasuh, 2014). The leaching behaviour of Ca in the sample was probably related with anhydrite (CaSO_4) that formed during leaching. The dissolution ratio of Ca increased slowly with the increase of acid content of solution, and similar behaviour has been observed for anhydrite in the literature (Marshall and Jones, 1966; Azimi and Papangelakis, 2010). The dissolution ratio of aluminium increased up to a/o ratio of 0.3, and then started to decrease at greater a/o ratios probably due to the formation of different aluminium containing precipitates like alunites (e.g. hydronium alunite ($\text{H}_3\text{O}\text{Al}_3(\text{SO}_4)_2(\text{OH})_6$) or basic aluminium sulphate- AlOHSO_4 that may form at higher acidities (Georgiou and Papangelakis, 1998). The dissolution ratios of Cr were nearly constant and very low for all of the a/o ratios studied.

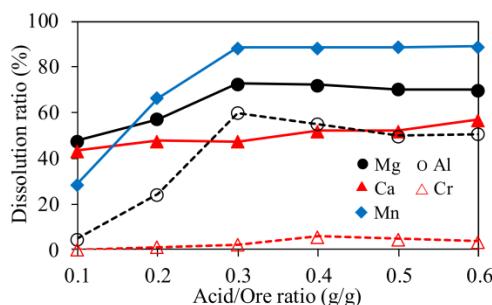


Fig. 4. The effect of acid/ore ratio on dissolution ratios of Mg, Ca, Mn, Al and Cr

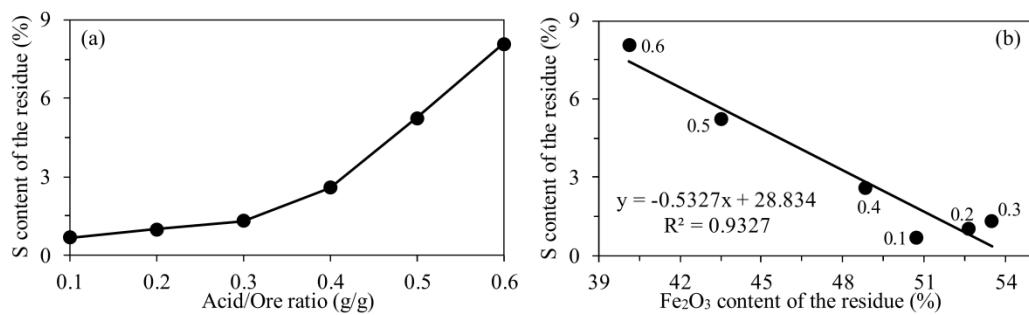


Fig. 5. The effect of acid/ore ratio on S content of leach residues (a).

Relationship between Fe_2O_3 and S contents of leach residues (b)

The sulphur contents and the XRD patterns of the leach residues were also determined after leaching at different a/o ratios to observe the possible precipitation of basic ferric sulphate (FeOHSO_4) phase, especially at higher a/o ratio values. Although

the initial S content of the lateritic ore sample was 0.03%, its amount in the residues increased slowly up to a/o ratio 0.3 and rapidly above 0.3, up to a/o ratio 0.6 (Fig. 5a), indicating the possible precipitation of FeOHSO_4 phase that theoretically contains 47.27% Fe_2O_3 and 18.98% S. The relationship between Fe_2O_3 and S contents of the leach residues (Fig. 5b) supported the formation of FeOHSO_4 phase at higher a/o ratios. When the amount of precipitated FeOHSO_4 increased, there should be a reverse relationship, i.e. Fe_2O_3 content of the residues should decrease and S content of the residues should increase.

The XRD patterns of the leach residues were also determined (Fig. 6) to substantiate the precipitation of FeOHSO_4 phase. Although there is no peak related to FeOHSO_4 in the XRD pattern of residues obtained after leaching up to 0.3 a/o ratio (Fig. 6b and Fig. 6c), the patterns of residues obtained after leaching at a/o ratio of 0.5 (Fig. 6d) and 0.6 (Fig. 6e) have several peaks at $2\theta = 18.6^\circ$, 27.3° and 27.8° indicating the formation of FeOHSO_4 phase. As seen from Fig. 6, formation of basic ferric sulphate reduced the amount of hematite in the leach residues. Visually, the residues obtained after leaching at 0.3 a/o ratio had reddish colour, indicating higher hematite (Fe_2O_3) content, but the ones obtained after leaching at a/o ratio 0.5 had brownish-yellowish colour, indicating the precipitation of FeOHSO_4 (Long, 2000; Nasuh, 2014). The basal peak of smectite at lower angles disappeared after leaching at 0.3 and higher a/o ratios.

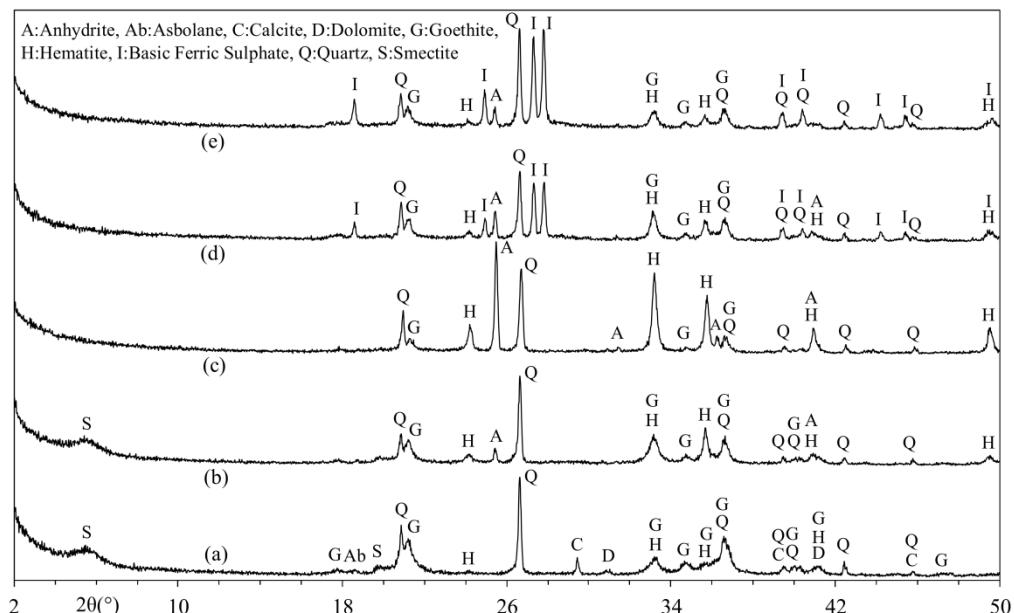


Fig. 6. XRD patterns of ore sample (a) and leach residues obtained after leaching at acid/ore ratios of 0.1 (b), 0.3 (c), 0.5 (d) and 0.6 (e)

Effects of leaching temperature

Among the most important parameters that affect leaching, the effects of leaching temperature were studied at $T = 200^\circ$, 210° , 220° , 230° and 240° °C. During these experiments, the a/o ratio and the leaching time was kept constant at 0.3 and $t = 60$ minutes, respectively. The changes in the dissolution ratios of Ni, Co and Fe, and the changes in the dissolved Fe/Ni ratio following high pressure leaching of the lateritic nickel ore sample at different leaching temperatures are given in Fig. 7 and Fig. 8, respectively. Figure 7 shows that the increase in leaching temperature from 200° °C to 240° °C increased the dissolution ratios of Ni and Co from 65.0% and 77.8% to 92.8% and 94.3%, respectively. The increase of leaching temperature decreased the dissolution ratio of Fe, resulting in lower dissolved Fe/Ni ratios (Fig. 8), because of the increased hydrolysis of iron in the leach solution and the lower hematite dissolution (Umetsu et al., 1978; Georgiou and Papangelakis, 1998; Whittington and Muir, 2000).

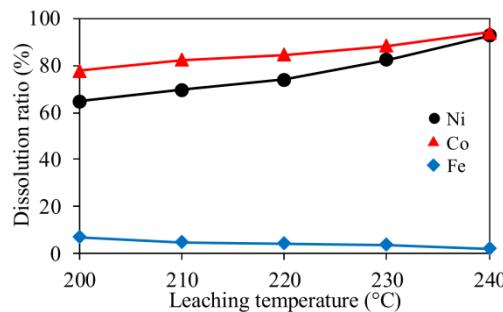


Fig. 7. The effect of leaching temperature on dissolution ratios of Ni, Co and Fe

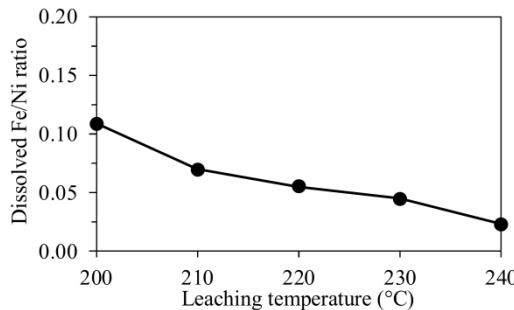


Fig. 8. The effect of leaching temperature on dissolved Fe/Ni ratio

The changes in the dissolution ratios of Mg, Ca, Mn, Al and Cr were given in Fig. 9. As can be seen, the increase of temperature from 200° °C to 230° °C increased Mg, Mn and Al dissolution ratios and decreased Cr dissolution ratio. Between 230° °C and 240° °C, all of the dissolution ratios of the mentioned cations stabilized and stayed at nearly constant values.

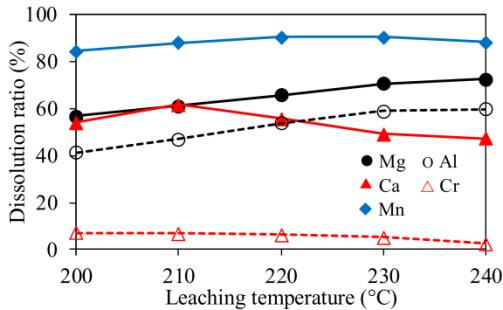


Fig. 9. The effect of leaching temperature on dissolution ratios of Mg, Ca, Mn, Al and Cr

For different a/o ratio experiment series, no significant differences in the S contents of the leach residues were observed, that changed between 1.04 and 1.31% S. Such low values of S contents of the leach residues suggested that no observable FeOHSO₄ formation occurred with the increase in the leaching temperature. This was also substantiated by the XRD patterns of the leach residues obtained after leaching at 200 °C and 240 °C (Fig. 10) that no peaks belonging to FeOHSO₄ phase were observed. Other than FeOHSO₄ phase, the formation of Fe₂O₃ phase with the increase in the leaching temperature could easily be seen when the XRD patterns in Fig. 10b, 10c and 10d compared, where the intensities of peaks at $2\theta = 24.2^\circ$, 33.2° and 35.7° belonging to hematite were increased.

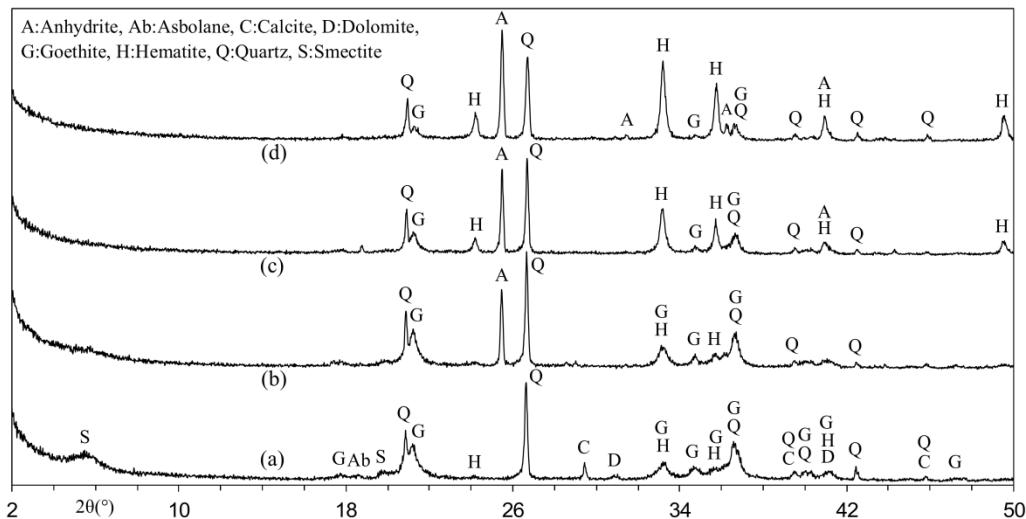


Fig. 10. XRD patterns of (a) ore sample and leach residues obtained after leaching at temperatures of (b) 200 °C, (c) 230 °C and (d) 240 °C

Effects of leaching time

When compared to the effects of acid/ore ratio and leaching temperature, the effect of leaching time on high-pressure acid leaching of lateritic nickel ores was smaller. In this study, the effects of leaching time were studied at $t = 30, 60, 90$ and 120 minutes. During these experiments, the a/o ratio and the leaching temperature was kept constant at 0.3 and $T = 240$ °C, respectively. The changes in the dissolution ratios of Ni, Co and Fe, and the changes in the dissolved Fe/Ni ratio following high-pressure leaching of the lateritic nickel ore sample at different leaching times are given in Fig. 11 and Fig. 12, respectively. Figure 11 shows that the leaching behaviours of Ni and Co were very similar and their dissolution ratios were 86.4% and 89.7% after 30 min, 92.8% and 94.3% after 60 min, 95.1% and 95.4% after 90 min, and 96.6% and 95.5% after 120 minutes of leaching. With the increase of leaching time, the amount of iron hydrolysed increased (Fig. 14; see also Whittington and Muir, 2000). So, the dissolution ratio of iron (Fig. 11) and correspondingly the dissolved Fe/Ni ratio (Fig. 12) decreased.

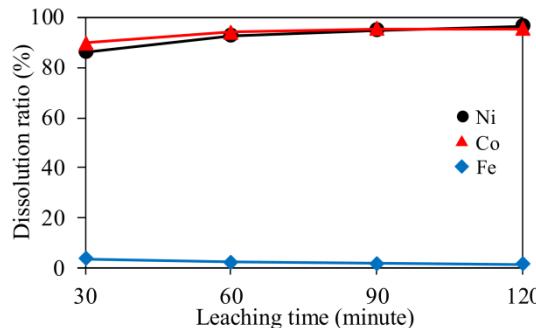


Fig. 11. The effect of leaching time on dissolution ratios of Ni, Co and Fe

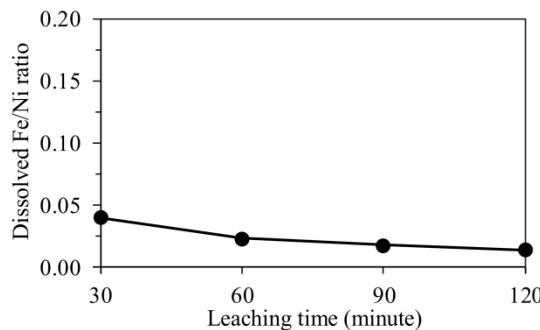


Fig. 12. The effect of leaching time on dissolved Fe/Ni ratio

The effects of time on the leaching behaviours of Mg, Ca, Mn, Al and Cr are given in Fig. 13. As can be seen, the dissolution ratio values of Mg, Mn, Al and Cr stayed nearly at constant values in the range of the leaching times studied. As in temperature effect experimental series, there were no significant differences in the S contents of

the leach residues, which changed between 1.11% and 1.31% S. Such low values of S contents of the leach residues suggested that no observable FeOHSO_4 formation was occurred with increased leaching time. This was also substantiated by the XRD patterns of the leach residues obtained after leaching for 30, 60 and 120 minutes (Fig. 14), in which no peaks indicating FeOHSO_4 phase were observed. When the XRD patterns in Fig. 14 were compared, the increased formation of hematite phase in the leaching solution with the increase in leaching times could easily be seen by the increasing peak intensities of hematite.

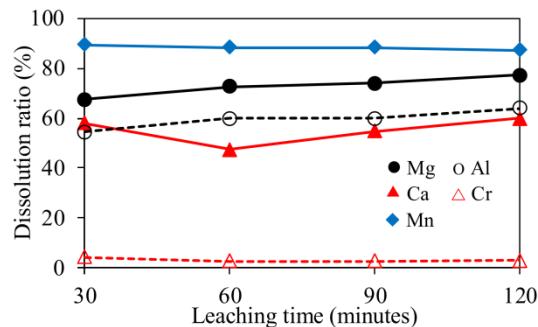


Fig. 13. The effect of leaching time on dissolution ratios of Mg, Ca, Mn, Al and Cr

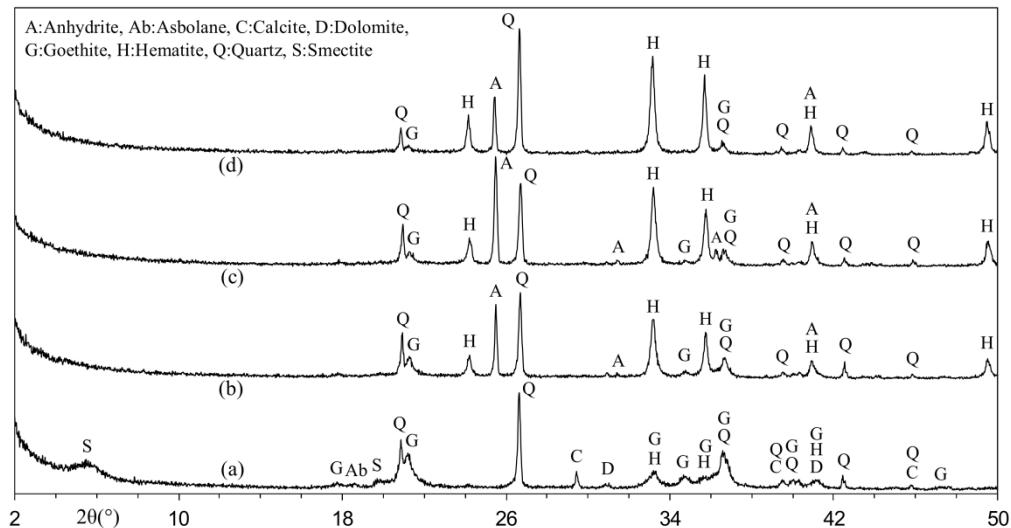


Fig. 14. XRD patterns of (a) ore sample and leach residues obtained after leaching for (b) 30, (c) 60 and (d) 120 minutes

Conclusions

In this work, high pressure sulphuric acid leaching behaviour of a local lateritic nickel ore sample having 1.37% Ni and 0.076% Co were investigated by using chemical and X-ray diffraction analyses. Among the investigated parameters, the acid/ore ratio and the leaching temperature showed the highest impact on high pressure leaching of the ore sample. The leach residues obtained after leaching at acid/ore ratio of 0.3 contained hematite, quartz and anhydrite, but the use of larger amounts of acid (e.g. 0.6) resulted in the formation of basic ferric sulphate phase, confirmed by sulphur and X-ray diffraction analyses of the leach residues. The increase of leaching temperature and leaching time were found to promote the precipitation of hematite rather than the precipitation of basic ferric sulphate. With the increase of leaching time, the amounts of dissolved nickel and cobalt increased. The dissolution ratio values of 92.8% and 94.3% and 95.1% and 95.4% for nickel and cobalt were obtained after 60 and 90 minutes of leaching, respectively, at acid/ore ratio of 0.3 and leaching temperature of 240 °C. Lowest dissolved Fe/Ni ratios were less than 0.025 obtained using high temperature and long leaching time. The dissolution ratios of Mg, Ca, Mn, Al and Cr were between 40% and 80%.

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