

# Characterization and dissolution of low-grade ferruginous nickel lateritic ore by sulfuric acid

L. Panda, D.S. Rao, B.K. Mishra and B. Das\*

Research scholar, senior principal scientist, director and chief scientist, CSIR-Institute of Minerals and Materials Technology, Bhubaneswar, India

\* Corresponding author email: bdas@immt.res.in

## Abstract

*The dissolution behavior of lateritic nickel ore containing 1.2% Ni, 40.6% Fe, 9.5% SiO<sub>2</sub> and 9.59% MgO by sulfuric acid was investigated. The ore was characterized adequately by different techniques such as chemical analysis, scanning electron microscope (SEM), X-ray diffraction (XRD), field emission scanning electron microscope (FESEM), thermal analysis and Fourier transform infrared (FTIR) spectroscopic measurements. The characterization results did not reveal any distinct mineral phase for nickel; it was associated with mineral phases like goethite, hematite, chromite and serpentine in various proportions. The beneficiation studies by hydrocyclone, magnetic separation and flotation did not enrich the nickel value. Reduction roasting followed by magnetic separation indicated only 2% Ni with 28.9% yield. Consequently, the recovery of nickel values from the lateritic ore by acid leaching under different conditions was studied. The kinetics of leaching obey the first order rate equations. The recovery of nickel was found to be influenced by temperature. It was observed that at an acid concentration of 2M, leaching temperature 363 K, time 240 min and solid-to-liquid ratio of 1:10, it is possible to leach out more than 95% Ni.*

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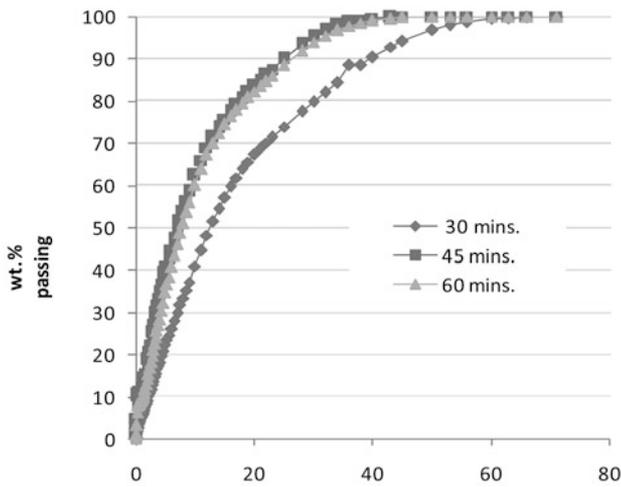
Key words: Nickel, Leaching, Sulfuric acid

## Introduction

The demand for nickel is ever increasing. Nickel is used to impart specific properties to steel, titanium and aluminium, and also is used in petrochemicals, catalysts, batteries, etc. It has been estimated that nickel oxide ores account for about 70% of the world nickel reserves. However, most nickel is sourced from sulfide ores. Nickel is extracted from sulfide ore by size reduction, followed by flotation to obtain a nickel-rich concentrate. The concentrate is then pyrometallurgically processed by smelting to produce a nickel-rich matte after discarding an iron-rich slag. Due to the increasing demand for nickel in stainless steel and other alloys, nickel is also extracted from many secondary sources. The recovery of nickel from oxide ores is now gaining momentum, and more exploitation of this ore is being attempted by advanced as well as environmentally feasible, cost-effective techniques. Dalvi et al. (2004) has reported that, due to the increase in demand, the world nickel production from laterites is expected to increase every year from the current level of production. In recent years, investigations of the use of low-grade ores, spent catalysts, clays, etc., have intensified to meet the market demand and also due to a shortage of high-grade nickel bearing ores (Al-Mansi and Abdel, 2002; Baba et al., 2009; Ismail et al., 2011).

It has been reported that conventional mineral processing techniques cannot be applied to beneficiate the nickel oxide ore of lateritic or limonitic type, as nickel is present in a finely disseminated state either with silicate or clay, or in the limonite or serpentine phase (Zubryckj et al., 1965; Whittington and Muir, 2000; Norgate and Jahanshahi, 2011). Most of the viable nickel laterites do not contain any specific nickel minerals; rather, they contain nickel in an adsorbed state associated with secondary oxides and silicate minerals (Burger, 1996).

The chromite overburden material of the Sukinda region of India contains 0.2-0.9% Ni and is present in the goethitic/limonitic phases. These materials are difficult to upgrade by conventional mineral beneficiation techniques (Kanungo, 1987; Ananda Rao et al., 1995; Suri and Gupta, 1995; Mohanty et al., 2000). The maximum nickel that could be upgraded by hydrocyclone is limited only to 1.1% Ni from a feed containing 0.5-0.9% of Ni (Das and Reddy, 1990). Pyrometallurgical routes involving reduction roasting, hydrometallurgical processes involving acid leaching or a combination of both have been applied to recover nickel from laterite ores. One such example is the Caron process, where the ore is reduced at 700° C in a reducing atmosphere to convert nickel into a metallic state, followed by selective leaching with ammonia-ammonium carbonate solution (Caron, 1950). Microwave radiation or a combined method such as segregation roasting of laterites followed by flotation or magnetic separation, as well as microbial aspects of mineral beneficiation, have also been



Particle size,  $\mu\text{m}$ , at different grinding times

**Figure 1** — Particle size distribution of three different ground samples carried out in a ball mill at different intervals of grinding time.

**Table 1** — Chemical analysis of the samples.

Constituents	%
Fe (T)	40.61
Fe <sub>2</sub> O <sub>3</sub>	58.07
CaO	4.19
SiO <sub>2</sub>	9.5
Al <sub>2</sub> O <sub>3</sub>	4.24
Ni	1.2
MgO	9.59
Cr	0.95
Co	0.16
LOI	12.1

attempted to recover nickel and other metal values (Iwasaki et al., 1966; Kawatra and Natarajan, 2001; Zhai et al., 2009). However, these methods are found to have their own merits and demerits due to several processing steps involved. Hence, direct leaching of nickel from laterites is found to be advantageous over other methods. In direct leaching, besides nickel, other elements such as cobalt, iron and magnesium generally associated with such ores can be recovered comprehensively.

Most of the investigations carried out in the past were concerned with high pressure and high temperature acid leaching to extract nickel values from oxide ore. These processes showed problems like high acid consumption and high dissolved iron content in leach solution (McDonald and Whittington, 2008; Buyukakinci and Topkaya, 2009). Current nickel extraction methods from lateritic ore are being carried out by hydrometallurgical processes like high pressure acid leach (HPAL) or heap leaching methods. The HPAL process is highly energy intensive, whereas the heap leaching process is cheaper but limited to certain types of ore. Both processes use low iron and high magnesia ore. In another method, the ferronickel process, ore containing high iron content (~50% Fe) is used to produce stainless steel by simply blending the limonite ore containing ~1% Ni with iron ore.

In the present investigation, a nickel lateritic ore was taken up with the primary objective to enrich the nickel content by an appropriate physical beneficiation technique. In the process of the investigation, hydrocyclone, magnetic separation and flotation techniques were applied, but a suitable concentrate could not be obtained due to the complex nature of the ore. Hence, an approach of acid leaching was attempted for this difficult-to-treat ore. In this method, sulfuric acid at different operating variables was employed to bring the maximum recovery of nickel into the solution and the characterization of the ore and leach residue by different techniques is investigated.

## Materials and methods

**Sample.** An Indonesian nickel overburden, part of the ongoing project work of our institute, was taken up during the course of the investigation. The ore was crushed and ground to different particle sizes. The sample was ground in a laboratory ball mill at different intervals of grinding time. The jaw-and-roll crushed product of -3 mm size was used for the grinding studies. At the end of 30, 45 and 60 minutes of grinding time, all of the products were subjected to particle size measurements. The purpose of making three different sizes was to understand the effect of particle size on the recovery of nickel. Similarly, a particle size of -150  $\mu\text{m}$  was prepared by ball mill grinding and used for the conventional wet, high-intensity magnetic separation studies, while -5+1 mm sized material was selected for reduction roasting studies.

**Particle size and chemical analysis.** The particle size of three different ball mill ground samples selected for leaching studies were analyzed by a laser particle size analyzer CILAS-1064 and shown in Fig. 1. More than 60% by weight of the particle population is below 45  $\mu\text{m}$ . The 80% passing size of the sample as determined from the size analysis curve of 30, 45 and 60 min of grinding was found to be 30, 18 and 17  $\mu\text{m}$ , respectively. Chemical analyses of the dried lateritic ores carried out by wet chemical and X-ray fluorescence (XRF) are shown in Table 1. It can be observed that the sample contains 1.2% Ni and 40.6% Fe. The loss on ignition (LOI) is 12.1%, indicating that the sample contains hydroxyl-bearing minerals such as limonites and goethites. The sample also contains a higher amount of magnesia (9.59% MgO). The nickel analysis of the leaching experiments was carried out by atomic absorption spectrophotometer techniques.

**Characterization studies.** The X-ray diffraction study was carried out by a Philips X-ray diffractometer using Cu-K $\alpha$  radiation. The purpose was to identify different mineral phases present in the sample. The samples were examined by Fourier transform infrared spectroscopy (FTIR) spectra (Shimadzu FTIR, IR Prestige-21), using KBr as the reference to identify the band position of the original and leached sample. The back scattered electron (BSE) image, morphology of the sample and leach residue were analyzed by Hitachi 3400N scanning electron microscope. The thermogravimetry (TG) and differential thermal analysis (DTA) studies were carried out using Netzsch Sta 449 C equipment that can heat to a maximum temperature of 900° C.

**Magnetic separation and reduction roasting studies.** The magnetic separation studies were carried out in a standard wet magnetic separation unit supplied by a Boxmag-Rapid separator (Birmingham, UK). Experiments were carried out by varying the magnetic intensity for possible enrichment of iron and nickel values. In another attempt, the sample was subjected

to reduction roasting followed by magnetic separation. The reduction roasting studies of the ore were carried out in a muffle furnace. In the reduction process, powdered charcoal of high quality was used as the reductant. The reduction was carried out at 950° C for 1 hr. The reduced mass was then cooled, ground in a ball mill to below 150 µm size and subjected to low intensity magnetic separation studies. The purpose of the study was to convert the goethite and hematite phases of the sample to magnetite, which can be easily separated by low-intensity magnetic separation techniques.

**Leaching studies.** Leaching experiments were performed on a 1-L glass vessel that was placed in a magnetic stirrer with temperature control. The glass vessel was fitted with a thermometer and a variable speed stirrer. As a typical experimental procedure, acid solution with predetermined concentrations was added to the leaching vessel containing dried sample and then heated to the desired temperature. The effect of leaching parameters such as acid concentration, time, temperature and particle size were studied to determine the optimum leaching conditions. The leachates were analyzed for nickel and iron using an atomic absorption spectrophotometer and wet chemical techniques. Initially, three different mineral acids, sulfuric acid, hydrochloric acid and nitric acid, were chosen as solvents for the dissolution of nickel.

## Results and discussions

**XRD analysis.** The XRD studies of the sample before and after leaching with sulfuric acid are shown in Fig. 2. The major minerals identified in the ore sample were amesite {Mg<sub>2</sub>Al(SiAl)O<sub>5</sub>(OH)<sub>4</sub>}, chrysotile {Mg<sub>3</sub>Si<sub>2</sub>O<sub>5</sub>(OH)<sub>4</sub>} and forsterite (Mg<sub>2</sub>SiO<sub>4</sub>). These are Al- and Mg-bearing silicate minerals in association with iron. However, a major change with regard to the X-ray peaks was observed in case of the leached residue. The change in XRD pattern observed in the case of leach residue is due to the removal of some elements like Mg, Fe and Ni in the process of leaching. Therefore, a new prominent peak of silica (quartz) is observed.

**SEM observations.** Stereomicroscopic microphotographs as shown in Fig. 3a indicated few black-colored grains. These are identified as chromite grains, while the other gray-colored particles are silicates. Figure 3b is the general view of the sample by scanning electron microscope (SEM), indicating wide size range for the particles. Figure 3c and 3d show the morphologies that are fibrous, elongated and nonporous in nature, indicating the presence of serpentine minerals. The BSE image, along with the elemental mapping, is shown in Fig. 4. It can be observed that the concentrations of Mg, Si and Al are greater

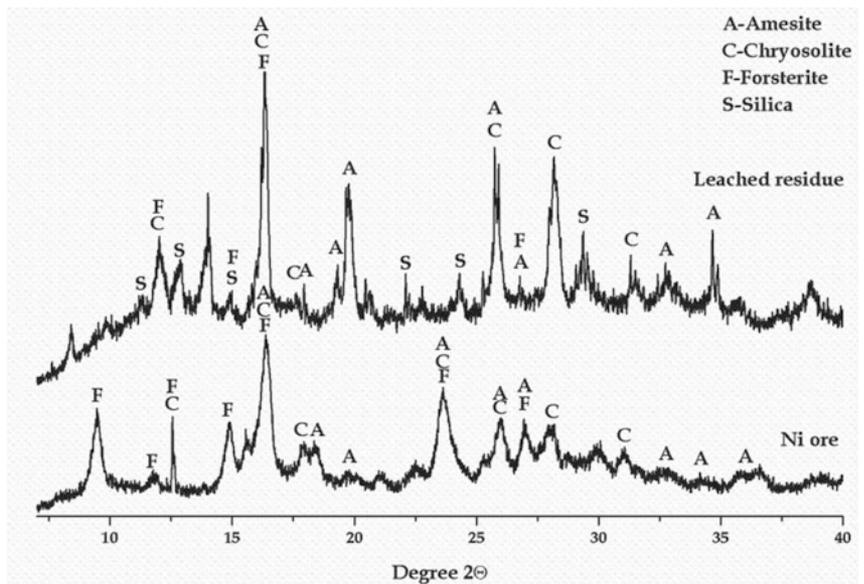


Figure 2 — X-ray diffraction of ore and leach residue.

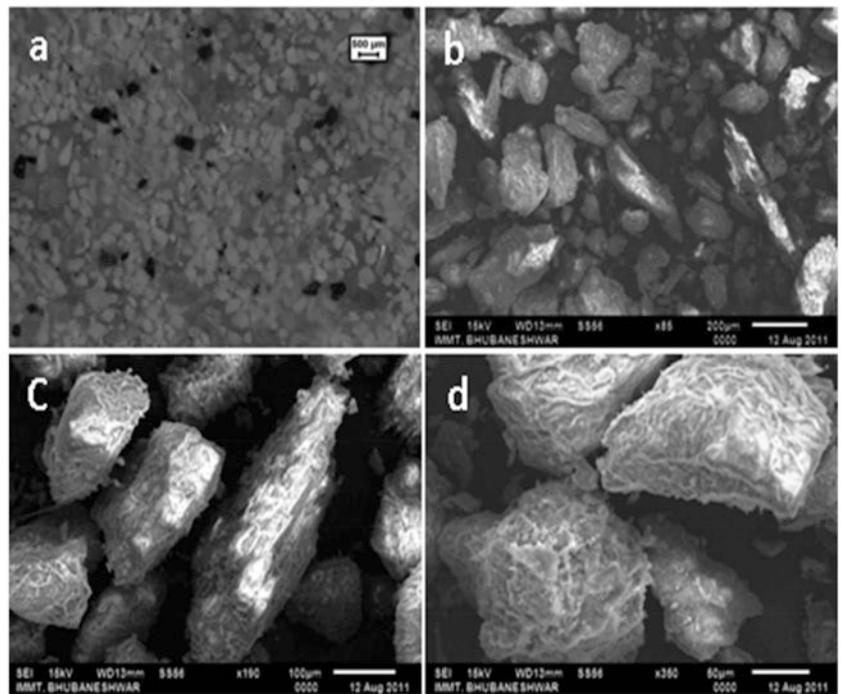
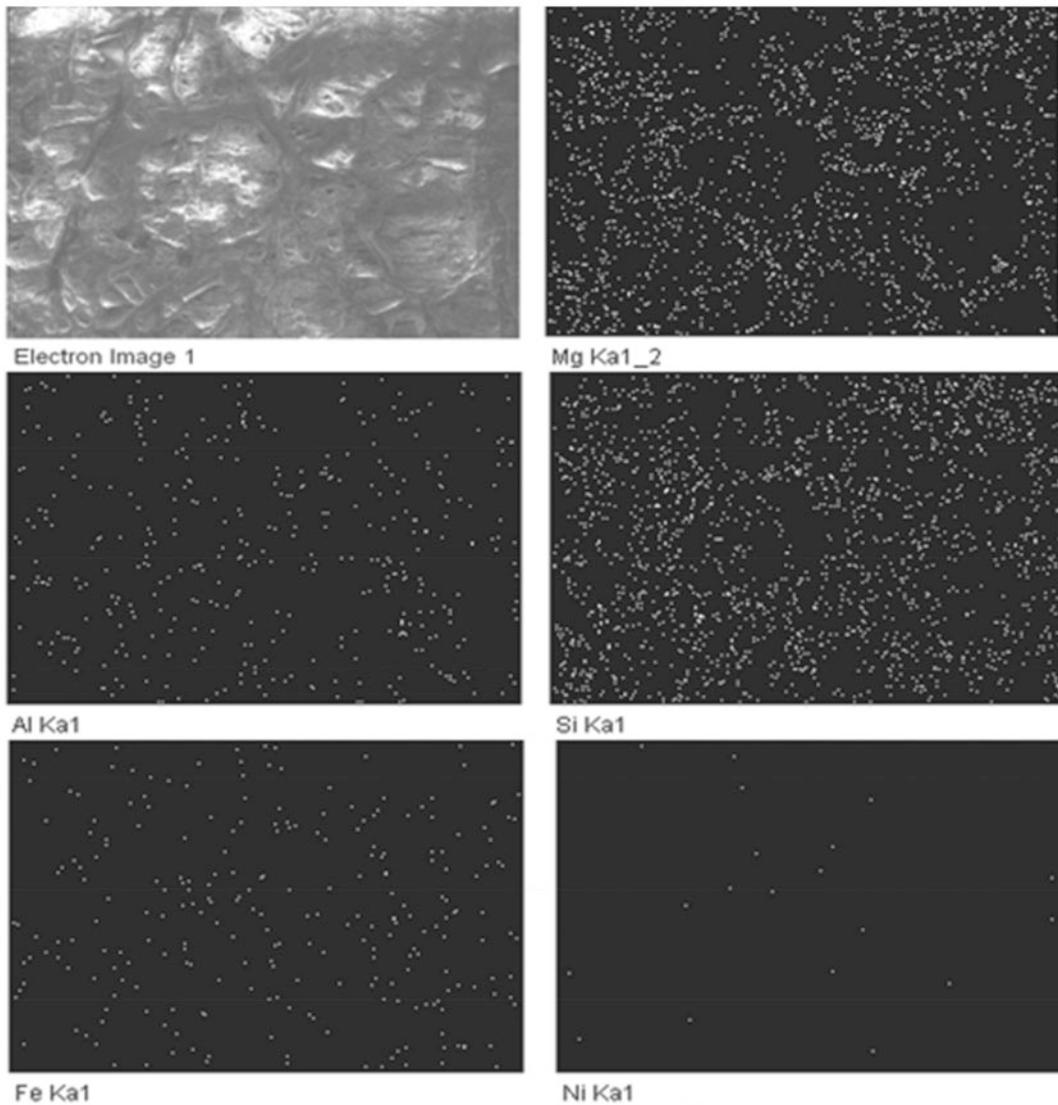


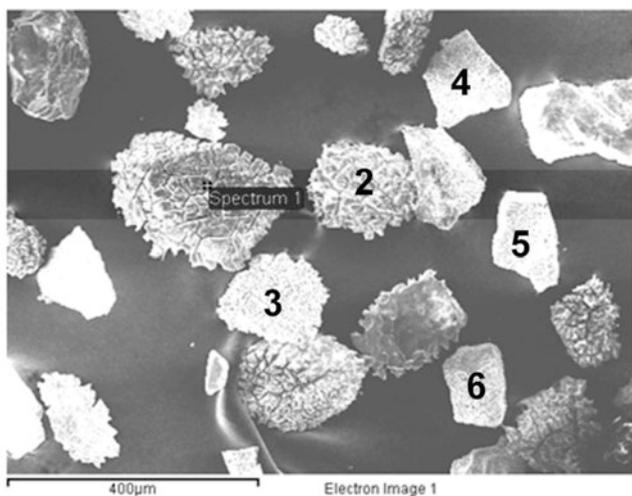
Figure 3 — (a): Stereomicroscopic image; (b, c, d): SEM microphotographs of lateritic ore.

Table 2 — SEM-EDX analysis of different phases present in the sample.

Constituents	Monticellite			Forsterite		
	Point 1	Point 2	Point 3	Point 1	Point 2	Point 3
MgO	27.82	27.3	30.82	27.06	33.28	35.76
SiO <sub>2</sub>	63.85	63.79	60.48	59.57	66.72	64.24
CaO	8.32	8.91	8.7	8.00	ND	0
FeO	ND	ND	ND	ND	ND	ND
Al <sub>2</sub> O <sub>3</sub>	ND	ND	ND	5.37	ND	ND
Total	99.99	100	100	100	100	100



**Figure 4** — SEM image of lateritic ore.

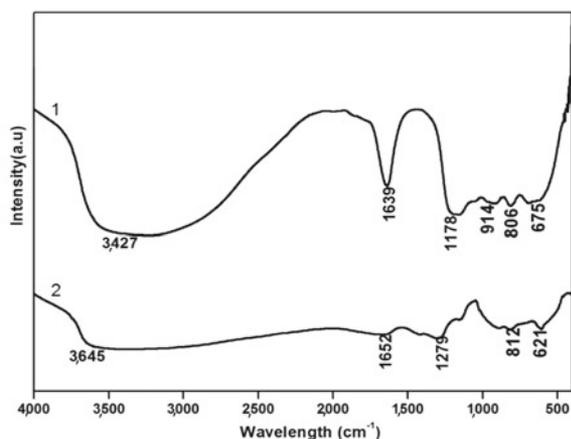


**Figure 5** — BSE image of the chromite grains present in lateritic ore.

than that of Ni. Nickel is embedded in the sample in the form of minute particles, and is not present uniformly. In fact, it might be associated in very trace levels in an adsorbed state within

these phases. The point analysis by SEM/ energy dispersive X-ray (SEM-EDX) results indicated that the sample consists of monticellite and forsterite as the two silicate phases. It is also indicated that the silicate phases contain high values of silica, magnesia and calcium but do not show any value for nickel, iron and aluminum (Table 2). The leach residues of the grains were also studied under SEM, which revealed that the grains have developed some fractures and cracks. The SEM image of the ore revealed the elliptic morphology, while the leach residue shows high cracking and porous structures that indicate modifications of the surface area of the ore particles due to phase transformation.

**FESEM study.** Quantitative analysis of a few selected black particles identified under the stereomicroscope was carried out with the help of field emission scanning electromicroscopy (FESEM). The BSE image of the same is shown in Fig. 5. The grains were observed to be chromite. Analysis of a few points by FESEM indicated that they exhibit a wide range of  $\text{Cr}_2\text{O}_3$  values (42.17% to 85.98%). It is observed that in some grains, nickel (0.13%-0.42%) is also associated with these chromites. The MgO content varies from 8.61%-13.08% and  $\text{Al}_2\text{O}_3$  from 11.25%-31.01%. Observing these grains as chromite with a high level of association with MgO and  $\text{Al}_2\text{O}_3$  suggests that

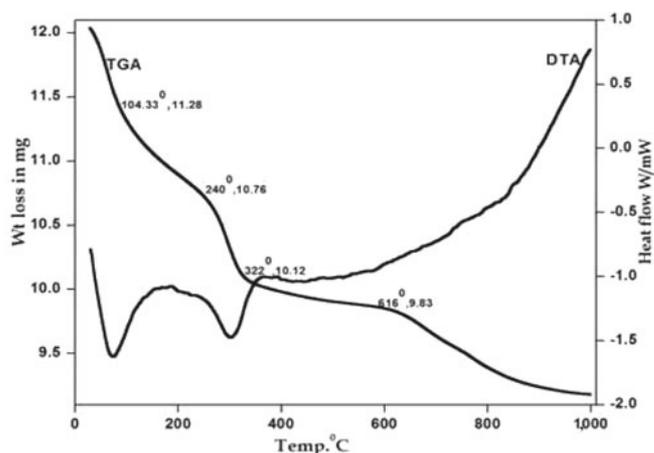


**Figure 6** — FTIR spectra of (1) lateritic ore and (2) leach residue.

these particles were nonliberated.

**FTIR study.** Figure 6 shows the FTIR spectra of laterite ore (1) and the leach residue (2). A broad peak is detected in the spectrum of feed sample at around  $3,427\text{ cm}^{-1}$  due to the stretching vibrational band of the OH group (Langer and Letteno, 1980). However, the peak intensity becomes weak after treatment with sulfuric acid. The sharp band centered around  $1,639\text{ cm}^{-1}$  is due to the banding mode of H–O–H vibration of water molecules (Motlagh et al., 2011). This peak is virtually absent in the leach residue sample due to the transformation of this group in the presence of heat. The other sharp absorption band appeared at around  $1,178\text{ cm}^{-1}$ , which may be due to C–H bending vibration. This band is also missing in the case of the residual sample. It was observed that the absorption peak appears at around  $675\text{ cm}^{-1}$  in the spectra of the ore belonging to the banding mode of HO-Mg, and is shifted to  $621\text{ cm}^{-1}$  in the case of the leach residue (Zhang et al., 1997). The peaks decrease gradually after leaching the sample, which suggests that the binding state of the OH base in the structure of serpentine is altered due to disordering of the local structure around metal ions. The bands at  $806\text{ cm}^{-1}$  and  $914\text{ cm}^{-1}$  are due to the bands between H and O (Prasad et al., 2006).

**Thermal analysis.** Figure 7 shows the TG and the corresponding differential thermal analysis DTA curves. The original mass of  $12.03\text{ mg}$  was used for the study. There are four mass loss steps observed in the TG curve. The first mass loss step begins at  $104^\circ\text{C}$  with a significant endothermic peak in the DTA curve. This step represents the vaporization of free water. The second mass loss step initiates at  $240^\circ\text{C}$  and is maximized at  $260^\circ\text{C}$ , also accompanied by an endothermic peak in the DTA curve. The water loss in lateritic ore can be classified as free water, crystal water and a hydroxyl group. During the process of heating, the removal of crystal water occurs at  $200\text{--}4,800^\circ\text{C}$  (Rizov, 2012). Hence, the mass loss shown at  $240\text{--}2,600^\circ\text{C}$  is due to the loss of crystal water. The third mass loss step is found at  $3,220^\circ\text{C}$ , which may be due to dehydroxylation of goethites. It has been reported that the dehydroxylation temperature for highly crystalline goethite is  $385^\circ\text{C}$ . It is lower for fine-grained and poorly crystalline goethite (Habashi et al., 1969). The fourth mass loss step starts at  $616^\circ\text{C}$  and



**Figure 7** — TG -DTA curves of lateritic ore.

has a maximum mass loss rate at  $667^\circ\text{C}$ , which is attributed to the decomposition of serpentines. The total mass loss in the TGA temperature range of  $30\text{ to }900^\circ\text{C}$  is around  $13.38\%$  of the total mass.

**Magnetic separation studies.** Initially, investigations were carried out to recover the nickel values by physical beneficiation techniques. Hydrocyclone and flotation studies failed to enrich the nickel value. The maximum Ni value that could be enriched by different design and operating variables using a  $125\text{-mm-diameter}$  hydrocyclone was limited to  $1.3\%$ . Similarly, the direct and reverse flotation studies carried out using oleic acid and dodecylmine as collectors could not enrich the nickel value to more than  $1.35\%$  Ni. Since the ore contains around  $40\%$  Fe, magnetic separation studies using a high intensity separation technique were then attempted.

The magnetic separation study of the lateritic ore was carried out with the assumption that nickel values can be enhanced with an increase in iron values. The result of the wet, high-intensity magnetic separation studies of the sample is shown in Table 3. It can be seen that the weight of the magnetic material increases slightly with an increase in magnetic intensity, but the nickel grade remains virtually the same in all magnetic products. Thus, the preliminary results indicated that nickel cannot be upgraded by a simple high intensity magnetic separation technique. The maximum Ni value that could be enriched by applying around  $15,000\text{ gauss}$  is around  $1.51\%$ .

**Table 3** — Wet, high-intensity magnetic separation of the sample particle size  $-150\text{ }\mu\text{m}$ .

Mag. Int. Gauss	Products	Weight, %	Fe, %	Ni, %
7,700	Magnetic	8.2	39.3	1.16
	Washing	6.6	39.9	1.27
	N-Magnetic	85.2	40.1	1.21
10,065	Magnetic	11.3	38.1	1.17
	Washing	7.5	38.3	1.25
	N-Magnetic	81.1	40.4	1.35
13,000	Magnetic	11.4	38.1	1.19
	Washing	10.0	39.4	1.10
	N-Magnetic	78.6	40.1	1.47
15,000	Magnetic	11.7	34.1	1.22
	Washing	9.0	39.1	1.24
	N-Magnetic	79.3	39.4	1.51

**Table 4** — Magnetic separation studies of reduced sample. Particle size 150  $\mu\text{m}$ , magnetic intensity  $\sim 2,000$  gauss.

Reductant, %	Products	Weight, %	Fe, %	Ni, %
2.5	Magnet	8.9	46.63	2.54
	N-Magnetic	91.1	39.65	1.47
5	Magnet	28.9	60.7	2.0
	N-Magnetic	71.1	46.6	1.47
7.5	Magnet	42.5	58.2	1.92
	N-Magnetic	57.5	43.2	1.40
10	Magnetic	48.1	60.3	1.87
	N-Magnetic	51.9	36.7	1.3
12.5	Magnetic	57.1	55.6	1.66
	N-Magnetic	42.9	28.9	1.3

Among the three acids,  $\text{H}_2\text{SO}_4$  was found to be more effective with nickel compared to  $\text{HCl}$  and  $\text{HNO}_3$ . It was observed that at the end of four hours of leaching with 10M  $\text{H}_2\text{SO}_4$ , Ni extraction is about 50% at room temperature, whereas in the case of  $\text{HCl}$  and  $\text{HNO}_3$ , it is 58% and 25%, respectively. However, the dissolution of iron values in the three acids is 34%, 60% and 12%, respectively. It was observed that in the case of  $\text{HCl}$ , nickel and iron values are extracted simultaneously, whereas in the case of sulfuric and nitric acid, only nickel is extracted, with less iron. As the extraction of nickel by sulfuric acid is more as comparable to nitric acid, therefore, it was used for the subsequent leaching studies.

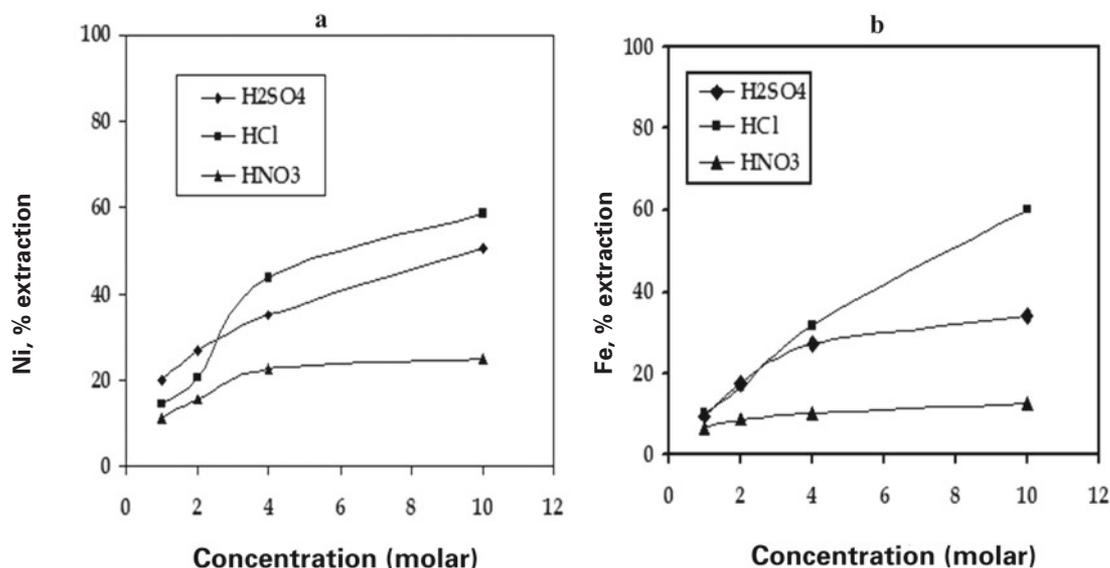
This has been obtained in the nonmagnetic fraction, indicating that the very finely divided goethite and limonite present in lateritic ore could not be captured at this magnetic intensity. This also indirectly reflects that Ni values are associated with the iron-bearing minerals.

The results of reduction roasting followed by magnetic separation as presented in Table 4 indicate that, even at this stage, the iron particles could not be liberated, and the value of nickel grade was limited to 2.0%, with 28.9% yield. The effect of reductant could indicate that the weight of the magnetic product increases but the nickel value could not be enhanced to an appreciable grade. Hence, further study by chemical beneficiation using mineral acids was attempted to recover the nickel values present in lateritic ore.

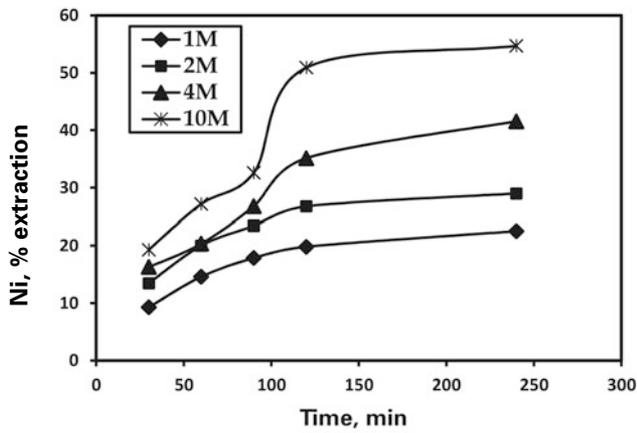
**Dissolution studies.** *Effect of acid concentration.* The dissolution studies of nickel from the lateritic ore were first carried out with three different mineral acids such as sulfuric acid, hydrochloric acid and nitric acid. The concentrations of acids were varied from 1M to 10 M. All experiments were carried out at a solid: liquid ratio of 1:10. The sample from 30 minutes grinding time ( $d_{80} \sim 30 \mu\text{m}$ ) was used for the dissolution study. The results of these studies are shown in Fig. 8. The results indicated that in all cases, extraction of nickel into the solution increases with the increase in acid concentration.

*Effect of leaching time.* The effect of leaching time by sulfuric acid on the dissolution of nickel was studied at 303K temperature and at a solid-to-liquid ratio of 1:10. Particle size of 30 min grinding time ( $d_{80} \sim 30 \mu\text{m}$ ), acid concentration in the range of 1M to 10M with leaching time of 30 to 240 min was studied. The results of these studies are presented in Fig. 9. The maximum extraction values reached at different acid concentrations were 53.6%, 42.9%, 28.7% and 20% at 10, 4, 2 and 1M acid solutions, respectively. The extraction of nickel increased up to 120 min. Thereafter, the values remained almost constant. It is assumed that iron, magnesium and aluminum, as the major constituents of laterites, transform during leaching to their respective sulfates. A considerable amount of iron during hydrolysis converts to  $\text{Fe}_2\text{O}_3$ , and aluminum cations also hydrolyze, which leads to the formation of solid products like  $(\text{H}_3\text{O})\text{Al}_3(\text{SO}_4)_2(\text{OH})_6$  (Georgiou and Papangelakis, 1998) similar to the type of complex compound,  $\text{H}_2\text{Fe}_3(\text{SO}_4)_2(\text{OH})_7$ , formed during the leaching of Serbian nickel lateritic ore under atmospheric pressure (Stopic et al., 2003).

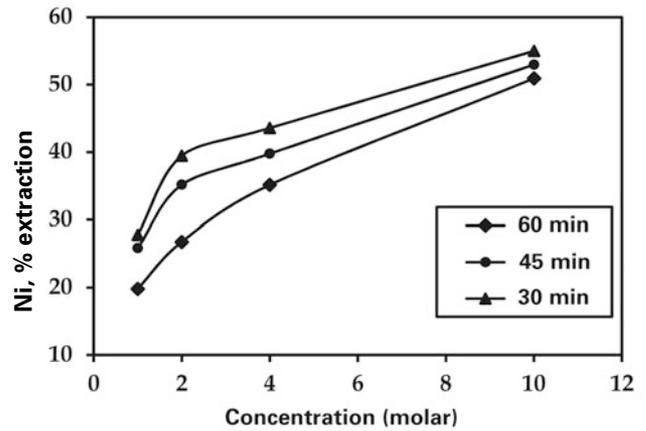
*Effect of leaching temperature.* The effect of leaching temperature was examined in the range of 303 to 363 K. During the experiments, leaching time of 120 min, acid concentration of 2M, particle size of  $d_{80} \sim 30 \mu\text{m}$ , and solid-to-liquid ratio of 1:10 was maintained. The results obtained are shown in Fig. 10. The dissolution values of nickel at 303 and 323 K were only 25% and 50%, respectively. It was also observed that iron dissolution to the extent of 16% and 23% could be achieved at



**Figure 8** — Nickel and iron dissolution at different molar concentrations of acids at 303 K, 240 min, solid: liquid ratio of 1:10.



**Figure 9** — Effect of leaching time on Ni dissolution at different  $H_2SO_4$  concentrations, at 303 K, solid: liquid ratio of 1:10 and particle size  $d_{80} \sim 30 \mu m$ .

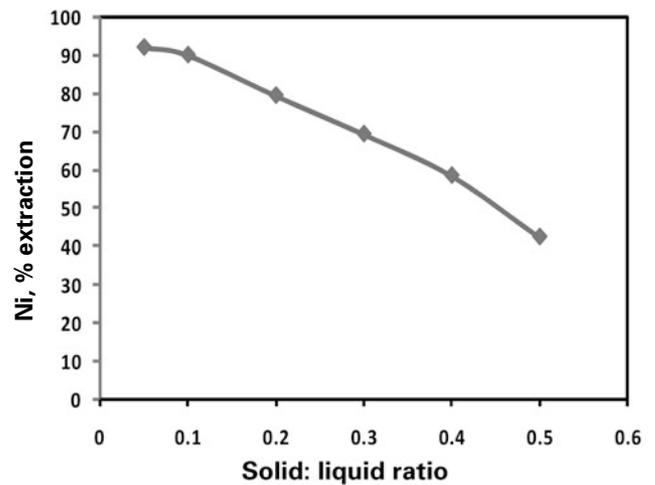


**Figure 11** — Effect of sulfuric acid concentration upon nickel extractions at different grinding times, at 303 K and 120 min leaching time.

these two temperatures. The dissolution tendency of nickel and iron increases with an increase in temperature. It was possible to extract more than 95% nickel at 363 K, whereas only <35% was possible for iron. The results indicate that an increase in leaching temperature has a positive effect on extractions of nickel and iron.

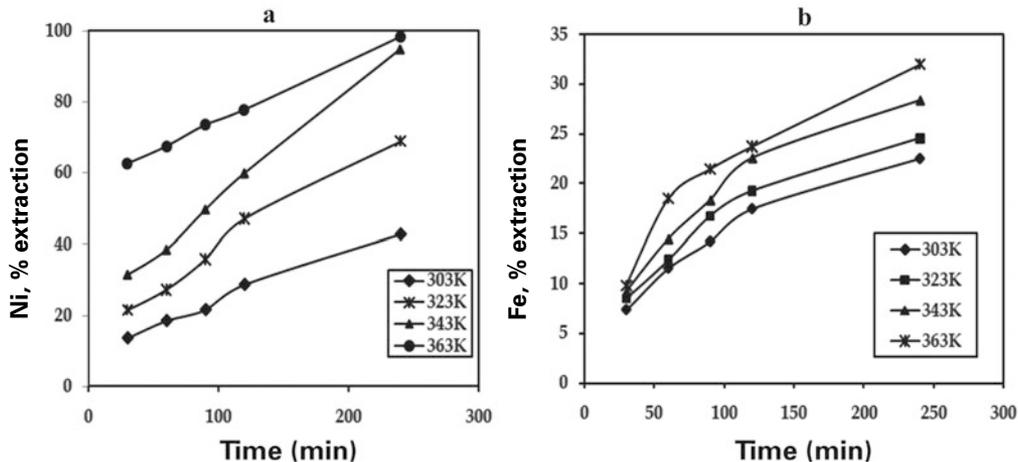
*Effect of particle size.* Particle size is a dominating factor influencing the leaching effect. A series of experiments were carried out by varying the concentration of sulfuric acid from 1M to 10M acid solution at three different particle sizes obtained by ball mill grinding. Here, the effects of particle size of the sample on extraction of nickel and iron were investigated. The leaching study was carried out at 303 K temperature and the solid-to-liquid ratio of 1:10 and leaching time 120 min were kept constant. The results of the experiments are shown in Fig. 11. It shows that the particle size has a significant effect on the dissolution of nickel. The efficiency of nickel extraction is greater in the case of 60 min grinding compared to 45 and 30 min grinding. It was possible to extract 45% to 55% nickel at normal temperature and 120 min of leaching time. The fine particles generated at the higher grinding time supply a larger surface area for the dissolution of the nickel, which is promoted by a thinner diffusion layer enabled by fine particles generated by grinding.

*Effect of ore-to-sulfuric acid ratio.* The effect of acid-to-

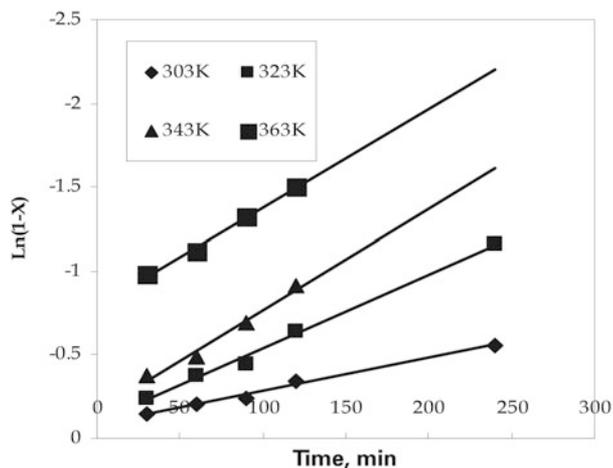


**Figure 12** — Dissolution of nickel at different solid:liquid ratios (temp. 363K, time 120 mins, acid conc. 2M).

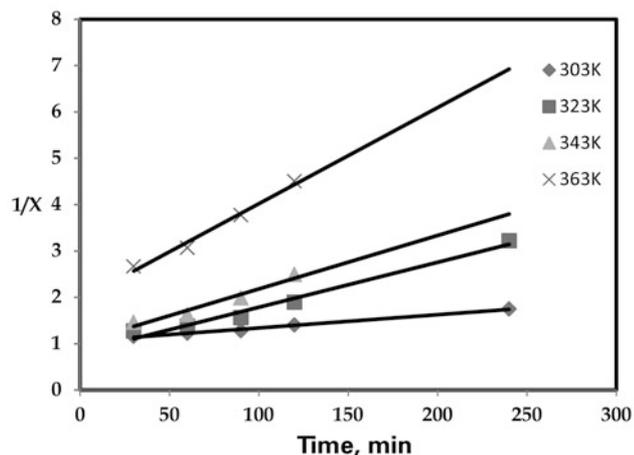
ore ratio on nickel dissolution at 363 K, 120 min leaching and 2M acid solution is shown in Fig. 12. It can be seen that the leaching efficiencies of nickel decrease as the solid-to-liquid ratio increases from 0.05 to 0.5, which is due to the decrease



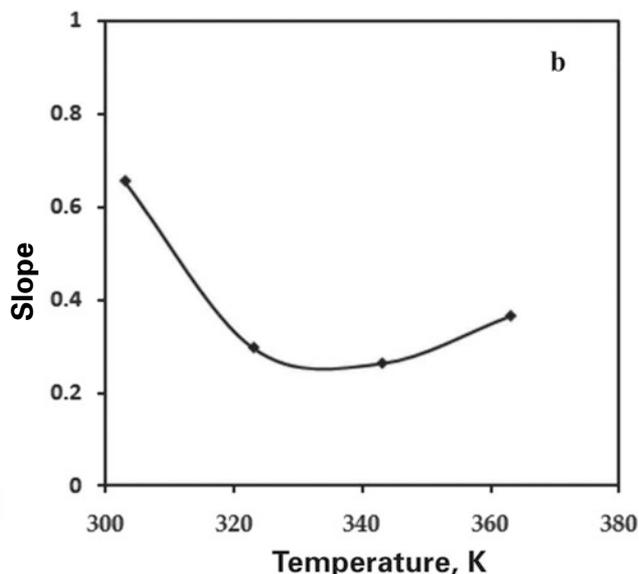
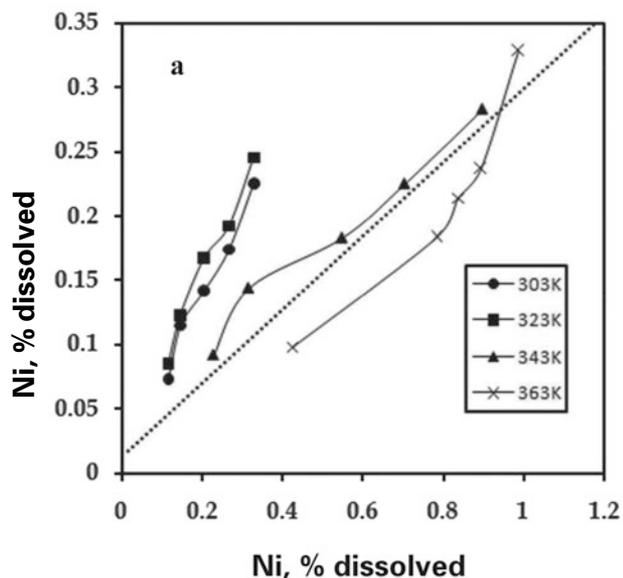
**Figure 10** — (a) Nickel and (b) iron dissolved at different temperatures and times, acid concentration of 2M, solid: liquid ratio of 1:10, particle size,  $d_{80} \sim 30 \mu m$ .



**Figure 13** — First order kinetics for the dissolution of nickel ore.



**Figure 14** — Second order kinetics for the dissolution of nickel ore.



**Figure 15** — (a) Congruency of dissolution graph for Ni as % of total Ni versus Fe. (b) Slope of dissolution congruency graphs for various temperatures of heating. The line represents slope  $\neq 1$  (i.e., in congruent dissolution).

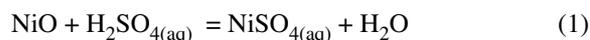
**Table 5** — Complete chemical analysis of the leach residue.

Constituents	%
Fe(T)	34.2
Fe <sub>2</sub> O <sub>3</sub>	48.92
CaO	3.8
SiO <sub>2</sub>	18.8
Al <sub>2</sub> O <sub>3</sub>	9.5
MgO	8.5
Cr	0.8
Co	0.02
Ni	0.05
LOI	9.1

of H<sup>+</sup> concentration in the leaching solution to the available solids. It has been noted that with high solid-to-liquid ratio, the slurry becomes viscous and hampers ion mobility, thereby decreasing the leaching efficiency.

The different studies carried out with sulfuric acid have indicated that it is possible to extract more than 95% nickel by adjusting different leaching parameters. The removal of nickel is clearly understood by analyzing the leach residue, which is shown in Table 5. It can be observed that, due to the removal of nickel and partly the removal of iron and magnesium from the ore, the percentage of other constituents such as silica and alumina increased.

*Kinetics of nickel extraction.* The leaching of nickel by sulfuric acid occurs according to the following reaction.



In order to study the kinetics of nickel extraction from the lateritic nickel ore, the data thus obtained in Fig. 10 are plotted in Figs. 13 and 14. The first order kinetics are studied by plotting  $\ln(1-X)$  versus  $t$ , where  $X$  is the fraction of the nickel recovered at a particular time  $t$ . Similarly, the second order kinetics are studied by plotting  $1/X$  versus  $t$ . The regression values of the equations are shown in Table 6. It is observed

**Table 6** — The regression coefficients for kinetics equations.

Kinetics	303 K	323 K	343 K	363 K
1st order $t$ vs. $\ln(1 - X)$	0.9954	0.9971	0.9938	0.9938
2nd order $t$ vs. $1/x$	0.9925	0.9767	0.9538	0.9842

that the first order kinetics better represent the leaching rate compared to the second order kinetics. The value of the rate constant  $K$  varies between 0.002 to 0.005  $\text{min}^{-1}$ .

**Congruency of dissolution.** An examination of the congruency of metal dissolution can provide information about the distribution of metal substituent within the phases (Perrier et al., 2006; Liu et al., 2009). In the present investigation, the congruency of nickel dissolution with respect to iron was investigated using the experimental data. If Fe and the associated metal Ni dissolve at identical rates, it can be assumed that Ni is uniformly distributed throughout the iron oxide phases present in lateritic ore. The congruency of dissolution graph for Ni versus Fe at different temperatures is shown in Fig. 15. The slope with respect to each temperature was determined. It can be observed that the slope of the congruency of dissolution graph (Ni versus Fe) decreased from 0.7 (near congruent) to 0.2 (noncongruent) with an increase in heating temperature up to 363 K. Hence, it can be concluded that nickel is not uniformly distributed in iron phases, and they are not associated in single mineral phases.

## Conclusions

Detailed characterization studies of lateritic nickel ore were carried out using various analytical techniques. The laterite ore was found to consist of phases like amesite, chrysotile and forsterite. The other major phases were found to contain iron-rich phases like limonite, goethite, chromite and hematite. Upon leaching, disintegration and degradation of the majority of the mineral phases were evident. It was observed that the majority of nickel and some components of iron magnesium and aluminum were leached out. As a result, distinct differences in phase changes between XRD, SEM and FESEM were observed. The acid leaching by sulfuric acid could extract more than 95% Ni and less than 16% iron from the laterite ore. The leaching efficiency is mainly influenced by the sulfuric acid concentration, leaching temperature and liquid-to-solid ratio. The optimum conditions of leaching are determined as follows: acid concentration of 2M, leaching temperature of 363 K, leaching time of 240 min and solid-to-liquid ratio of 1:10. The rate of reaction follows first order kinetics. The association of nickel could not be detected in any single mineral phase, as evidenced from the congruency dissolution study of Fe and Ni.

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