

EFFECT OF ACID CONCENTRATION ON THE EXTRACTION OF NICKEL FROM LATERITE

TUNÇ PARLAK Tuğba, YILDIZ Kenan

Sakarya University, Engineering Faculty, Metallurgy and Materials Engineering, Sakarya, Turkey <u>ttunc@sakarya.edu.tr</u>

Abstract

Nickel is essential metal due to usage area as stainless steel, non-ferrous alloy production, electroplating and chemical industry. Degradation of nickel sulphides leads to extraction of nickel from laterites. High cost equipment of high pressure acid leaching and since heap leaching needs longtime, atmospheric acid leaching of laterites becomes popular. Parameters of atmospheric leaching are important in terms of acid consumption, content of pregnant solution and residue. Therefore in this study, lateritic nickel ore from Manisa-Çaldağ/Turkey was leached for determining acid concentration parameter, both sulfuric acid and nitric acid - sulfuric acid mixture with varying percentage by volume. XRD analysis of lateritic nickel ore was showed that the ore mainly consist of quartz and goethite. Pregnant leach solutions were analyzed with atomic absorption and leach residues were characterized by X-ray diffraction and scanning electron microscopy. Increment of acid concentration but iron content increased as well.

Keywords: Laterite, atmospheric acid leaching, nickel, extraction

1. INTRODUCTION

Because of specific feature of nickel, it has found many usage area in industry. Nickel is mainly used in stainless steel, non-ferrous alloys and electroplating. Nickel is preferred for production of airplane, ship and car parts which exposed to corrosion.

World nickel reserves consist of sulfide and lateritic types of ores. While lateritic type of reserves form 73 % of world's nickel resources, sulphide ores are preferred for nickel production [1]. Because of dependition of sulfide reserves and in respect to traditional pyrometallurgical methods, costs of nickel production increase, laterite becomes available source [2]. Laterite ores are homogenous mixture that comprises of hydrated iron oxides and hydrous magnesium silicates [3]. Today world nickel supply is covered predominantly by sulphide ores (60 % against 40 % by laterites). By taking into consideration that any additional nickel demand is expected to be mainly satisfied by mining of laterite deposits, the optimization of the metallurgical laterite processing methods constitutes a great challenge for the nickel industry and there is an increasing focus on the processing of the huge reserves of nickel-rich laterite ores due to declining global reserves of nickel sulphides [4, 5].

Extractive metallurgy of nickel is dependent on the type of ore body. Although recovery of nickel from sulfide ores is based on only pyrometallurgical methods, flow-sheets of nickel extraction from laterites are based on both pyrometallurgical and hydrometallurgical methods. In laterites, nickel is mainly present in goethite, serpentine, smectite and in manganese oxides together with cobalt. Since cobalt is associated with nickel, processes to extract nickel are also applicable to extract cobalt. Therefore hydrometallurgical treatments are based on extracting nickel and cobalt from iron, magnesium and manganese oxides, based on leaching procedures [6].

In this paper, the aim is enhance the extraction of nickel and cobalt from lateritic nickel ore by investigating the role of acid ratio, temperature and HNO₃ addition on the dissolution of related minerals in the structure.



2. MATERIALS AND METHODS

Provided lateritic nickel ore from Manisa-Çaldağ region in Turkey was initially subjected to crushing and sieving under 75 μ m for standardization of the particle size before the leaching tests. Chemical composition and the phases of the lateritic ore was determined by X-ray fluorescence (XRF) with Bruker AXS S8 Tiger and X-ray diffraction (XRD) with RigakuUltima X-ray diffractometer respectively. Liquid-to-solid ratio was kept constant at 20 (wt.%). Leaching was performed with 200 ml solution under atmospheric conditions in 500 ml glass flasks that were placed on a temperature controlled and magnetically stirred hot plate. String speed was kept constant at 300 rpm. At high temperatures, condenser was used to avoid evaporation. 25 °C, 50 °C, 75 °C and 95 °C as leaching temperature, the ore was added into the solution and leached for 240 min fordetermination of H₂SO₄ % (v/v). For leaching studies with addition of HNO₃, determined parameters - leach temperature and sulfuric acid concentration - were kept constant, and experiments were carried out at 95 °C for 90 min for v/v 1 %, 3 % and 5 % HNO₃ addition to the determined sulphuric acid concentration. At the end of leaching, slurry was immediately percolated. Ion concentration of the pregnant leach solutions were determined by atomic absorption with Thermoscientific Ice 3300 model AAS.Residues were washed with distilled water for three times, dried at 110 °C for 24 h and analyzed with XRD.

3. RESULTS AND DISCUSSION

Chemical composition and phase analysis of the laterite were shown at **Table 1** and **Figure 1** respectively. As seen from the **Table 1** and **Figure 1**, laterite is composed of silicon and iron in the form of quartz and goethite. Because of the low content of nickel and cobalt, any related phases of this elements could not be detected in XRD. This elements are available in the laterite structure assubstitution elements [7].

Components (wt.%)									
Fe ₂ O ₃	SiO ₂	NiO	C03O4	MgO	Al ₂ O ₃	Cr ₂ O ₃	CaO	MnO	Total trace elements
51.78	31.54	2.15	0.19	5.18	5.18	1.28	0.83	0.52	1.35

 Table 2 Chemical composition of laterite



Figure 1 XRD analysis of laterite (1: Quartz-SiO₂, 2: Goethite-FeO(OH))

Extraction results of nickel, cobalt and iron depending on various acid concentration were given in **Figure 2a - 2c** respectively. When leach temperature was kept constant, extraction values of metals increased slightly with increasing acid ratio whereasincreased temperature at a certain acid ratio caused significant increase in



extraction values. For 5 % H₂SO₄ concentration, nickel extraction of 25 °C, 50 °C, 75 °C, 95 °C was determined as 7.4 %, 22.0 %, 52.3 % and 68.5 % respectively. Increment of acid concentration to10 %, 15 % and 20 % at 95 °C caused to increase nickel extraction ratios to 79.4 %, 82.4 % and 83.6 % respectively. After leaching at 95 °C for 240 min, cobalt extractions were determined as 33.5 %, 42.3 %, 46.2 %, and 50.7 %, for iron 60.4 %, 75.8 %, 80.9 %, 82.4 % at H₂SO₄ concentrations of 5 %, 10 %, 15 % and 20 % respectively. Because of the slight increase of nickel after 10 % H₂SO₄ concentration and avoid the excess dissolution of iron, acid concentration was kept constant at this ratio.



v/v H₂SO₄ (%) **Figure 2** Extraction values of (a) nickel, (b) cobalt and (c) iron depending on temperature and acid concentration

XRD analysis of leach residues and laterite were shown in **Figure 3** comparatively. At the right side of the figure, between 20° - 30° was shown separately for clear view. With increment of acid concentration, goethite intensity decreased and quartz intensity increased. Also amorphizationin quartz structure were detected from the given graph at the right side.

After fixing leach temperature and acid ratio at 95 °C and 10 % H_2SO_4 respectively, effect of nitric acid addition was investigated and for comparison, additive-free leaching study was carried out with 10 % H_2SO_4 at 95 °C for 90 min. XRD analysis of related leach residues and obtained extraction valueswere shown in **Figure 4a**), **4b**) respectively.XRD analysis was given between 20° - 30° as only comprises of intensive peaks of quartz and goethite. Significant amorphization of quartz structure like as the previous one was not detected.Conspicuous point at the results is only cobalt extraction. Nickel and iron behaved similar to addition of HNO₃ and increase is negligible. Extraction of nickel was determined as, 69.2 %, 69.7 %, 70.6 % and 73.7 %



for additive-free, 1 %, 3 % and 5 % respectively. For cobalt, 30.1 %, 42.5 %, 44.2 %, 44.2 % and for iron 67.9 %, 68.5 %, 68.8 %, 69.8 % extraction were determined for additive-free, 1 %, 3 % and 5 % respectively.



Figure 3 XRD analysis of related leach residues and laterite (1: Quartz-SiO₂, 2: Goethite-FeO(OH))

In **Figure 5**, SEM-mapping analysis of leach residues of additive-free 10 % H_2SO_4 (v/v) studies carried out at room temperature and 95 °C for 240 min were given. As observed from the mapping, room temperature wasn't enough for dissolution of metals. But increased temperature changed this trend. Also from the graphs, it is understood that nickel and cobalt are found in iron area that means they occupy same structure.



Figure 4 (a) XRD (1: Quartz-SiO₂, 2: Goethite-FeO(OH)) and (b) extraction values of metals

Luo et al. studied atmospheric pressure acid leaching of saproliticlaterite that rich in lizardite. They conclude that nickel was found as substitution in lizardite. They found that with increasing temperature, nickel extraction increased as well. In their study, it is indicated that for higher nickel extraction, particle should dissolved entirely for releasing substitution nickel [8]. Girgin et al. examined Adatepe-Eskişehir lateritic ore with acid concentration and temperature and found that iron and nickel concentration in the solution were raised with increased ratio up to 60 % H₂SO₄ at 95 °C but after this point because of formation of ferric sulfate type compounds that contain of nickel and silicon caused nickel loss [1]. Liu et al. found that entirely dissolution of goethite can be



achieved with 2.5 M H_2SO_4 solution at 80 °C. They also stated that minerals in the ore show different dissolution behaviours depending on M-O bond strength and situation of metal cation as substitution [9]. McDonald and Whittington stated that different crystallographic types of goethite may show different dissolution ratios that cause from single-double-triple bond coordination [10].



Figure 5 SEM-mappings of leach residues

Büyükakıncı and Topkaya [11] studied extraction of nickel and cobalt with sulfuric acid from limonitic and nontronit lateritic type laterites and stated that acid concentration and leach temperature have important role on extraction of these metals and detected that highest extractions were obtained in experiments carried out at 95°C identically. Stopić et al. stated that nickel extraction up to 78 % from Serbian lateritic ore by sulfuric acid under atmospheric pressure at 90 °C was achieved [12].

4. CONCLUSION

In this study, the effects of acid ratio and temperaturewere investigated. The laterite ore from Çaldağ/Turkey, consists of quartz and goethite and nickel is found in goethite structure as substitution. Increased acid ratios at a certain temperature cause slight raise in metal concentration in the solution. But increasing temperature is more effective. For leaching studies carried out at 95 °C with 10 % $H_2SO_4(v/v)$ for 240 min, nickel, cobalt and iron extraction were determined as 79.4 %, 42.3 % and 75.8 % respectively. Other part of study additive-free and various HNO₃ additive at 95 °C for 90 min showed that ratio of addition may be insufficient. Extraction values of 5 % HNO₃ addition determined as 73.7 %, 44.2 % and 69.8 % for nickel, cobalt and iron respectively. But additive-free leaching test show approximation result as 69.2 %, 30.1 %, 67.9 %. Significant change only occurred in extraction of cobalt. When dissolution of nickel and iron compared, they show similar value that support substitution nickel in goethite structure.



ACKNOWLEDGEMENTS

This research was performed within the framework of project 2013-50-02-001. We thank the Commission for Scientific Research Projects of Sakarya University for funding this project.

REFERENCES

- [1] GİRGİN, İ., OBUT, A., ÜÇYILDIZ, A. Dissolution behaviour of a Turkish lateritic nickel ore. *Minerals Engineering*, 2011, vol. 24, no. 7, pp. 603-609.
- [2] MILIVOJEVIC,M., STOPIC, S., FRIEDRICH, B., STOJANOVIC, B., DRNDAREVIC, D.Computer modeling of highpressure leaching process of nickel laterite by design of experiments and neural networks. *International Journal of Minerals, Metallurgy, and Materials*, 2012, vol. 19, no. 7, pp. 584-594.
- [3] CRUNDWELL, F. K., MOATS M. S., RAMACHANDRAN, V., ROBINSON T. G., DAVENPORT W. Extractive metallurgy of nickel, cobalt and platinum group metals. Amsterdam, The Netherlands: Elsevier, 2011. 39 p.
- [4] ZEVGOLIS, E.N., ZOGRAFIDIS, C., PERRAKI, T., DEVLIN, E. Phase transformations of nickeliferous laterites during preheating and reduction with carbon monoxide. *Journal of Thermal Analysis and Calorimetry*, 2010, vol. 100, no. 1, pp. 133-139.
- [5] KING, M.G. Nickel laterite technology Finally a new dawn? *The Journal of the Minerals, Metals and Materials Society*, 2005, vol. 57, no. 7, pp. 35-39.
- [6] BÜYÜKAKINCI, T. Extraction of nickel from lateritic ores. MSc Thesis, METU, 2008. pp. 12-15.
- [7] SWAMY, Y. V., KAR, B.B., MOHANTY, J. K. Physico-chemical characterization and sulphatization roasting of lowgrade nickeliferous laterites. *Hydrometallurgy*, 2003, vol. 69, no. 1-3, pp. 89-98.
- [8] LUO, W., FENG, Q., OU, L., ZHANG, G., LU, Y. Fast dissolution of nickel from a lizardite-rich saprolitic laterite by sulphuric acid at atmospheric pressure. *Hydrometallurgy*, 2009, vol. 96, no. 1-2, pp. 171-175.
- [9] LIU, K., CHEN, Q., HU, H. Comparative leaching of minerals by sulphuricacid in a Chinese ferruginous nickel laterite ore. *Hydrometallurgy*, 2009, vol. 98, no. 3-4, pp. 281-286.
- [10] MCDONALD, R. G., WHITTINGTON, B. I. Atmospheric acid leaching of nickel laterites review: Part I. Sulphuricacid technologies. *Hydrometallurgy*, 2008, vol. 91, no. 1-4, pp. 35-55.
- [11] BÜYÜKAKINCI, E., TOPKAYA, Y.A.Extraction of nickel from lateritic ores at atmospheric pressure with agitation leaching. *Hydrometallurgy*, 2009, vol. 97, no. 1 2, pp. 33-38.
- [12] STOPIC, S., FRIEDRICH, B., FUCH, R. Kinetics of sulphuric acid leaching of the Serbian nickel laterite ore under atmospheric pressure. *Metalurgija Journal of Metallurgy*, 2002, vol. 8, no. 3, pp. 235–244.