Effect of particle size in the reduction of lateritic Ni ore in a Linder reactor

Efecto del tamaño de partícula en la reducción de mineral de Ni laterítico en un reactor de Linder

Efeito do tamanho de partículas na redução do minério de Ni laterítico em um reator de Linder

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Abstract

Colombia possesses Nickel ore in the highlands of the San Jorge region, south of the department of Cordoba. The material extracted from the area is processed by means of calcination and fusion in a Rotary Kiln and an Electrical Arc Furnace EAF respectively to obtain the Ferronickel. During the crushing, drying, and calcination of the mineral within the Rotary Kiln, fine ore particles that contain Fe and Ni are produced; these fine particles must be agglomerated and then returned to the furnace for calcination. Inside the furnace, the size of the material and the velocity of the gases, limits the efficiency of the process, where particles less than 2 mm do not calcine and is expelled from the furnace. The present work analyses the influence of particle size of a lateritic mineral of nickel, submitted to reduction in a Linder reactor, using two particle size distributions; a conventional sample for reduction test and another fine. The materials were dried and then reduced at a temperature of 950°C, with coal as a reductive agent. It was found that the loss of mass due to the particle size was not relevant and the decrease of particle size improves the concentration of the desired material, optimizing the recuperation of Fe and Ni during the reduction stage.

Keywords: lateritic mineral, rotary kiln, nickel, direct reduction, particle size, linder reactor.

Resumen

Colombia posee mineral de níquel en las tierras altas de la región de San Jorge, al sur del departamento de Córdoba. El material extraído del área se procesa mediante calcinación y fusión en un horno rotatorio y horno eléctrico de arco eléctrico, respectivamente, para obtener el ferroníquel. Durante la trituración, secado y calcinación del mineral dentro del horno rotatorio, se producen finas partículas de mineral que contienen Fe y Ni. Estas partículas finas deben ser aglomeradas y luego devueltas al horno para la calcinación. Dentro del horno, el tamaño del material y la velocidad de los gases limitan la eficiencia del proceso, donde partículas de menos de 2 mm no calcinan y son expulsadas del horno. El presente trabajo analiza la influencia del tamaño de partícula de un mineral laterítico de níquel, sometido a reducción en un reactor de Linder, utilizando dos distribuciones de tamaño de partícula; una muestra convencional para prueba de reducción y otra multa. Los materiales se secaron y luego se redujeron a una temperatura de 950 °C, con carbón como agente reductor. Se encontró que la pérdida de masa debida al tamaño de

partícula no era relevante y la disminución del tamaño de partícula mejora la concentración del material deseado, con lo cual se optimiza la recuperación de Fe y Ni durante la etapa de reducción.

Palabras clave: mineral laterítico, horno rotatorio, níquel, reducción directa, tamaño de partícula, reactor linder.

Resumo
A Colômbia possui minério de níquel nas terras altas da região de San Jorge, ao sul do departamento de Córdoba. O material extraído da área é processado por meio de calcinação e fusão em um Forno Rotativo e uma FEA de Forno a Arco Elétrico respectivamente para obter o Ferroníquel. Durante o esmagamento, secagem e calcinação do mineral dentro do forno rotativo, partículas finas de minério contendo Fe e Ni são produzidas; estas partículas finas devem ser aglomeradas e depois devolvidas ao forno para calcinação. Dentro do forno, o tamanho do material e a velocidade dos gases, limita a eficiência do processo, onde partículas menores que 2 mm não calcinam e são expelidas do forno. O presente trabalho analisa a influência do tamanho de partículas de um mineral laterítico de níquel, submetido a redução em um reator de Linder, utilizando duas distribuições de tamanho de partícula; uma amostra convencional para teste de redução e outra multa. Os materiais foram secos e depois reduzidos a uma temperatura de 950 ° C, com carvão como agente redutor. Verificou-se que a perda de massa devido ao tamanho das partículas não foi relevante e a diminuição do tamanho das partículas melhora a concentração do material desejado, otimizando a recuperação de Fe e Ni durante o estágio de redução.

Palavras-chave: mineral laterítico, forno rotativo, níquel, redução direta, tamanho de partículas, reator linder.

Introduction

One of the current problems in the extractive metallurgic industries is the generation of fine particles during processing operations of its minerals. The fine particles require additional processes for agglomeration, and this requires additional energy and the addition of reagents that work as agglutinating agents. This improves the consistency of pellets, briquettes or extrudes obtained from fine particles, contributing with additional material with commercial value, nevertheless it implies; an increase in costs for the company [1].

To obtain ferronickel in the metal industry it is necessary to implement certain steps to optimize the extraction of the mineral (mining, transport, grinding, homogenization, drying, calcination, etc.) [2] which permit a higher material concentration and better characteristics. As a result, the material can be introduced to a Rotary Kiln to produce calcined and pre-reduced products.

Before the chemical transformation of the mineral, precise control of the particle size is required during crushing, so that the particles arrive to the drying stage with minimal loss of material. These particles can continue to the Rotary Kiln where the calcination stage is completed with a coarse mineral that can resist the abrasion of rotation and can be reduced to iron and nickel with the least amount of material losses, which could be dragged by the gases, or could sinter to agglomerate, and ultimately form crusts or ringing inside the furnace [3-5]. During the calcination stage, the handling of the particle sizes is important, because it is the Rotary Kiln that decides the final size of the calcined product and the quantity of the fine particles that can be generated according to the mineralogical quality of the processed materials [4].

Fine particles in the industry are inevitable and they must undergo further studies that allow its concentration for later processes or the manipulation of these as individual materials without a necessity for agglomeration. With this premise, a typical mineral is taken to obtain ferronickel in two distribution sizes; one adapted to the reduction test (mesh +3/8" -1") and the other a fine material (mesh +16 -8). In this way, it is possible to know the behaviour of the minerals concerning the mass loss, reduction capacity, particle size and chemical modification [4]. The investigation proceeds at a laboratory test level, to demonstrate that the sieve classification does not influence the loss of the element with high economic value.

Materials and methods

Sampling
The mineral used as the study object, corresponds to a nickel laterite. The chemical composition is presented in Table 1. The material was obtained from deposits situated in Montelibano, Cordoba

[6]. This laterite was previously crushed, followed by sieving / screening processes and determines the size fraction and sectioning process, until two different representative samples, fine and coarse, were obtained. The samples were weighed and separated using a -100 mesh for its respective quantitative chemical analysis using X-Ray Fluorescence XRF [7].

Table 1. Chemical composition of lateritic nickel ore, in weight percent.

Compound	Ni	Fe	SiO2	MgO	Al2O3	CaO	Co3O4	Cr2O3	MnO	C	S	SiO2/MgO
Coarse	0.99	9.3	48.02	24.86	1.79	0.42	0.03	0.6	0.2	0.2	0.03	1.93
Fine	1.02	9.4	48	24.72	1.8	0.43	0.03	0.6	0.2	0.22	0.02	1.94

Granulometry

For the separation of the two particle sizes, 20 Kg of material was taken from a mineral sack with a capacity of 1 ton of lateritic mineral from the mines of Montelíbano Cordoba. The sample was obtained by Jones rifles and coning. Following size reduction was performed in a jaw crusher (Blake type - patented in 1858 by E. W. Blake, which consists of a jaw or fixed jawbone and a mobile section which is supported by an upper pivot), it is possible to take a representative sample of coarse particle material (9,50 to 19,00 mm), (mesh +3/8" -1"). Of From this coarse mineral, a proportion is taken to the second grinding stage where the fine particle samples are produced (1,18 and 2,00 mm) using a mesh +16 -8. For the drying stage 1,5 kg of material was sample was taken from each both the coarse and fine sizes for the drying stage [3-4]. The size distribution of the particles of lateritic mineral were determined processed according to the Standard ASTM 11/95.

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Drying

The fine and coarse particle samples (1,5 kg of each) were weighed on a gravimetric scale with a precision of ± 0,1 g, and then dried in an electrical oven at 120 °C for a period of 12 hours [3]. The samples were deposited on aluminium trays with dimensions of 25x40 cm, which were resistant to corrosion and to changes of weight by the effects of temperature.

Coal

The coal used is a carbon mineral that is collected by a company for the whole calcination process and originates from mines located in the Jagua de Ibirico within the department of Cesar [3]. This material was subjected to size reduction using crushing jaw to obtain a coal particle size of one half of the nickel ore samples, between 3 to 4 mm in diameter (sifter +7-5) to be added to the coarse lateritic material and 0,85 to 1,00 mm to the fine-particulate material (mesh +20-12) [8].

Linder reactor

The reduction test was undertaken in a laboratory reactor, which allows the use of different types of atmospherically conditions such as O2 for oxidation, H2, Co and CO2 for reduction and nitrogen in the neutral atmosphere. It is possible as well to use solid additives combined with the mineral to study the effects of the chemical changes that could occur. The reactor is constructed of stainless steel AISI 304 with a maximum capacity of 1Kg of solid material. It consists of a central cylinder with an outside diameter of 20cm, 50 cm in length and 9,5mm thick. There are lateral reductions that diminish the external diameter by 6cm. For the heating, two refractory blankets of concrax 1500 are used to isolate and protect direct contact of the electrical system and the cylinder. For the temperature control, the furnace possesses two

thermocouples of K type inserted in metal protection tubes of stainless steel AISI 308, one located on top of the refractory blanket and the other inside the camera of the reactor. The reactor is controlled from a panel where it is possible to modify the flow of gases, the working temperature and the speed revolutions of the reactor. It is possible to control the temperature up to 1200ºC. To perform changes to heating or cooling, the mechanism possesses a rotor with a variety of speeds that range from 0 to 50 rpm. For the control of the different types of gases, the apparatus has a series of electrical flow valves, with the possibility to modify the flow of gases from 0 to 30 litters per minute. The extraction of gas is done by means of a side connection to the cylinder using a flexible hose that allows the retention of liquids and the treatment of combustion gases [3-5].

Reduction tests

Each of the samples underwent a reduction process inside the Linder reactor. All the reduction processes were carried out under the following experimental conditions summarized in Table 2 [3-5,9]

Table 2. Reduction test parameters.

INITIAL QUANTITY ORE	810 g
INITIAL QUANTITY COAL	90 g
TOTAL WEIGHT	900 g
REDUCTION TEMPERATURE	950 C
HOLDING TIME AT MAX. TEMP.	1 h
ROTATIONAL SPEED	13rpm
FLOW OF N2	2,5 L/mn.
INITIAL TEMP. FOR FLOW OF N2	80 C
CLOSING OF THE FLOW OF THE GAS	40 C
REACTOR MATERIAL	SAE 304

A constant nitrogen flow (2. 5 L/min) [10] was injected at 80°C during heating and interrupted at 40°C during the cooling stage for each one of the samples. These temperatures are defined by the susceptibility of the material to oxidize above 80°C [11].

Table 1 shows that the mineral relationship between nickel ore and coal is 90\% and 10\% respectively, the mixture of 900 g was deposited inside Linder reactor furnace. A maximum temperature of 950°C was taken, considering bearing in mind that in a

Rotary Kiln at industrial level the nickel ore and coal mix reaches a maximum of 1000 °C, but during the discharge from the Rotary Kiln to the Electrical Arc Furnace (EAF), there is a loss of temperature to 950°C due to the contact with air. Finally, the holding time during the reduction test was of 1 hour for each sample, this in relation to the time handled inside the rotary kiln.

Results and discussion

Analysis of mineral
The analysis of minerals was carried out for each of the samples by the method of X-Ray Fluorescence XRF using a Philips RX-240 sequential spectrometer. The obtained chemical composition is summarized in Table 1 [7,14]. The Colombian lateritic mineral object of the study possess acidic characteristics due to the predominance of SiO_2 over MgO and the small quantity of CaO. The nickel content in the rock is very poor and is low in this type of laterites (0.3 to 1.3 per 100 of Ni), and the mineralogy is a product of intense weathering of the coffee saprolite (3.4 per hundred of Ni). However, the homogenization of nickel reduces the content of nickel at the expense of maintaining the percentages of Fe, SiO_2 and MgO [6], which guarantees a slag with physical and chemical characteristics suitable for the drainage and for the formation of ferronickel. In general, the compositional differences between both sizes are not significant. except for the valuable materials.

Drying
Based on the results obtained during the drying process of the samples, the percentage of moisture was calculated for each sample, using the following formula:

$$\%MOISTURE = (pi\text{-}pf) / (pi) \qquad (1)$$

Were,
pi is the initial weight of the sample and,
pf is the weight of the sample after the drying stage.

Table 3. Results of the moisture test for lateritic nickel ore.

Material	Initial Weight	Final Weight	%Moisture
Coarse particle (g)	1500	1217.3	55.113
Fine Particle (g)	1500	1395.8	69.447

The differences in moisture between the coarse-particle and the fine particle mineral is around 1.4%, which and this can be explained by the specific surface area, being higher in the fine-particulate material [3-4]. By increasing the surface area of the mineral and it is possible to achieve a higher efficiency during mineral drying.

Curves of heating and cooling
The heating and cooling curves were obtained by recording temperature changes at ten-minute intervals [15]. As the particle size reduces, also the time required to reach the desired temperature, diminishes. This effect influences the total dwell time inside the furnace, reducing the consumption of electricity and of nitrogen during the reduction test [4-5].
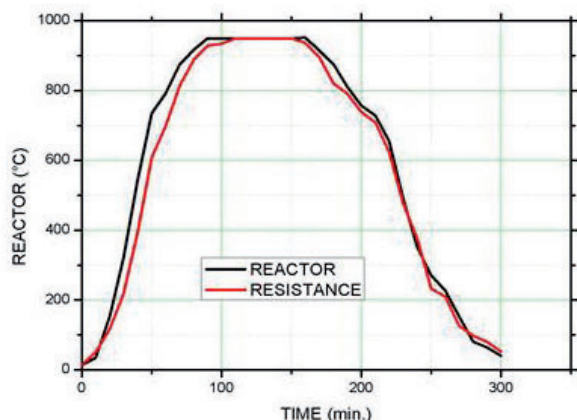

Figure 1. Heating and cooling curve of fine particle sample.
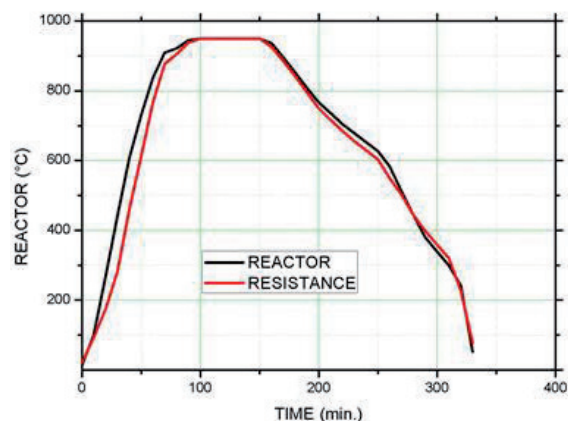

Figure 2. Heating and cooling curve of coarse particle sample.

Figures 1 and 2 demonstrate that the changes in particle size of the material influence significantly

the energy consumption, time and materials used during the processes of oxide-reduction. Therefore, as the particle reduces in size, the yield of the process increases in terms of cost reduction and thermal efficiency.

Reduce material
The products obtained from the reactor of the Linder furnace were weighed on analytical precision scales with an error margin of ± 0,1g. Following this, the remaining samples were kept in hermetic bags to avoid contamination or oxidation with external agents found within the immediate environment. The data of the materials were then loaded on to the reactor and the results are summarized in the following table:

Table 4. Relationship of sample weights before and after the reduction test.

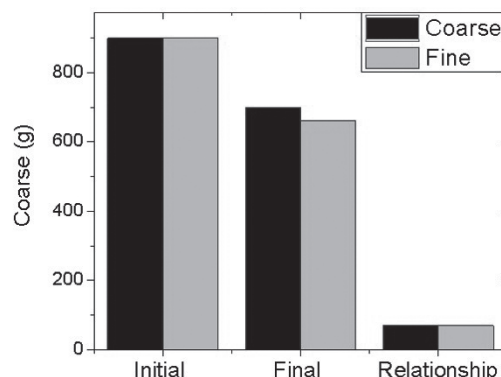| Material	Initial	Final	Relation
Coarse sample (g)	900g	699.1g	735
Fine sample (g)	900g	661.2g	69.447

Figure 3. Relationship of sample weights before and after the reduction test.

The relationship between the reduced fine and coarse lateritic samples present a significant difference in material quantity with the pre-reduced samples, where the coarse lateritic minerals retains a major part of its weight in relation to the fine particles, with a difference of 4.211 percent of material or 37,9 grams for a sample of 900 grams. The previous data means that despite being provided with a flow of nitrogen capable of eliminating the oxygen within the reactor, it is also capable of dragging the fine material and additionally the carbon that is used together with the fine material with particle sizes less than 0,85 to 1,00 mm (sieve +20-12) [8].

Composition
The analysis was carried out using X-Ray Fluorescence XRF by a Philips RX-240 sequential spectrometer and the reduced material was applied to a 100 mesh for evaluation. The results from the test are shown in the following table [7].

Table 5. Chemical characterization of the reduced lateritic mineral, in weight percent.

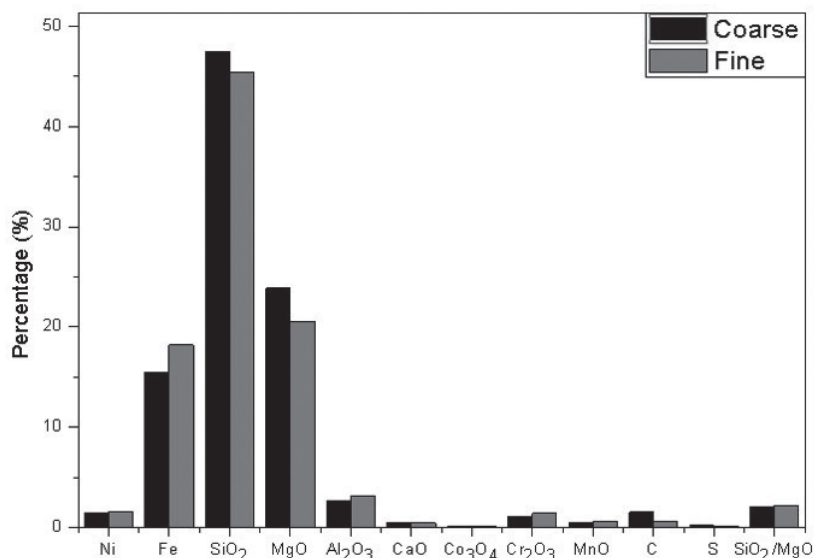| Compound	Ni	Fe	SiO$_2$	MgO	Al2O$_3$	CaO	Co$_3$O$_4$	Cr$_2$O$_3$	MnO	C	S	SiO$_2$/MgO
Coarse	1.47	15.5	47.48	23.86	2.57	0.5	0.07	1.12	0.41	1.5	0.17	1.99
Fine	1.5	18.2	45.37	20.54	3.11	0.44	0.09	1.42	0.53	0.55	0.12	2.21

Figure 4. Chemical characteristics of reduced lateritic mineral.

The characterization shows significant variations in components such as SiO$_2$, C and MgO which are found in large quantities within the fine-particle material, whilst whereas the elements Fe and Ni in low proportions increase their percentage in the mineral with smaller sizes. This demonstrates that as the particle decreases in size, the grade of liberation increases, thus eliminating easily the non-functional parts of the mineral of value; therefore, as the particle size diminishes the value mineral tends to increase its percentage in the sample [2].

The comparison of the composition of the samples before and after the reduction shows that the reduction in the size of the mineral increases significantly the elements of value such as Fe and Al$_2$O$_3$, whereas the content of SiO$_2$ and MgO are lower in the fine material after reduction.
The following table compares the gains and losses in weight of the material between the initial percentage and those recorded after the baking process or pre-reduction.

Table 6. Gains of material after reduction, in weight percent.

Compound	Ni	Fe	SiO$_2$	MgO	Al$_2$O$_3$	CaO	Co$_3$O$_4$	Cr$_2$O$_3$	MnO	LOI	C	SiO$_2$/MgO
Coarse	0.48	6.2	-0.54	-1	0.78	0.08	0.04	0.54	0.21	-9	1.3	0.06
Fine	0.48	8.8	-2.63	-4.2	1.31	0.01	0.06	0.82	0.33	-9	0.3	0.27

The strongest differences of the components between the of nickel laterites and those of the baked samples are carbon and alumina. The increase in Fe and Ni is due to a large specific density (see Table 7) and the influence of sulphides within the material and the carbon, since the presence of this element promotes to a great extent the growth of ferronickel particles in the metallic phase [12]. Components like SiO_2 and MgO diminished their percentages during the baking stage.

Table 7. Densities of components present within laterite mineral.

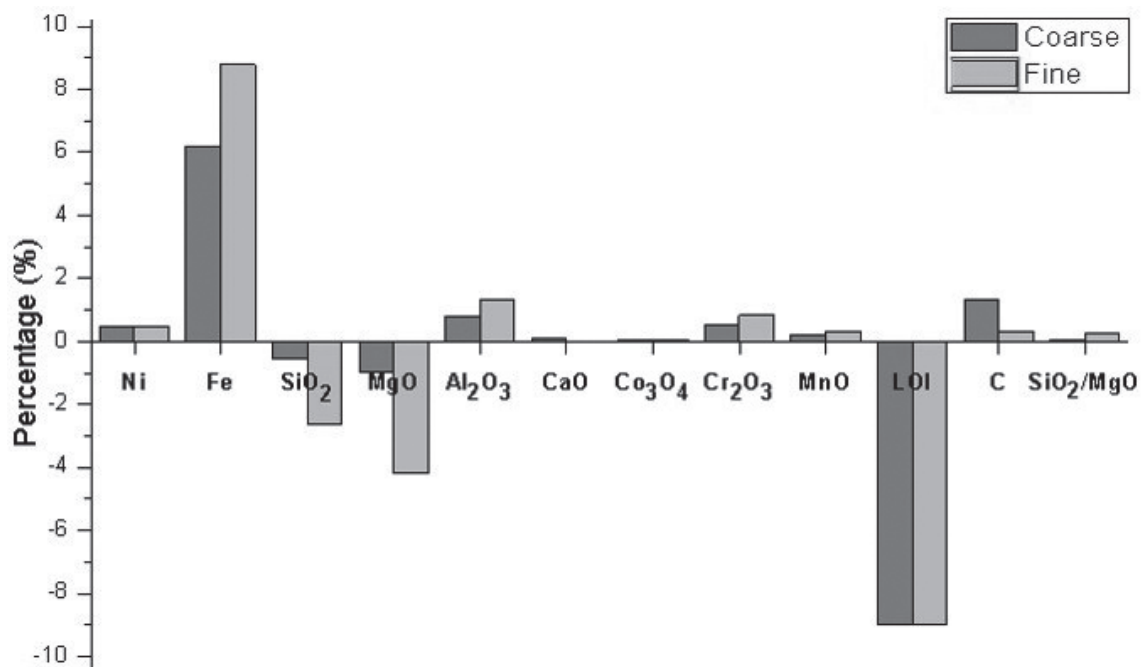| Compound	Ni	Fe	SiO_2	MgO	Al_2O_3	CaO	Co_3O_4	Cr_2O_3	MnO	C	S
Density (g/cm3)	6.67	5.59	2.65	3.58	3.95	3.35	6.33	4.8	5	2.26	2.07

Figure 5. Gains in material after reduction.

Taking into consideration the density, analysing the conditions to which the materials were submitted to during the reduction, and considering the physical properties of the material [2], it can be deduced that the gain or loss of certain elements is a result of several conditions, chemical composition and hardness and stability of the oxide before atmospherically reduction.

Conclusions

Samples of nickel ore subjected to drying and reduction processes in a Linder reactor, can reduces by the contact between the reducing gas and lateritic mineral without significant losses in mass.

The coarse-particle mineral that undergoes a reduction within a Linder reactor, degrades more than the fine-particle mineral under the same test conditions.

The iron and nickel gain differences during the reduction test in Linder reactor of mineral with different particle size is not significantly different.

Among all the compounds, only SiO_2 and MgO showed lower contents after reduction, resulting in a higher loss in the fine-particle mineral than in the coarse mineral. The components that were most enriched were iron and nickel.

The decrease in particle sizes of the nickel ore, favours significantly the processes of drying and calcination, as the material increases its superficial area, moisture, volatile matter and the organic matter can be eliminated more easily.

The alumina is the more stable compound in a nickel mineral submitted to reduction, primarily because of its high melting point and for its low reactivity with reducing atmosphere.

Acknowledgments

References

[1] Corredor W, Quintero H. Inuence of the grain size on nickel ore pellet quality and performance in the calcination process (graduation project). Tunja, Colombia: Universidad Pedagógica y Tecnológica de Colombia; 1992.

[2] Wills BA. Mineral processing technology. Processing of ore and mineral recovery. Mexico: Limusa Editorial; 1994.

[3] Medina FA. Capacity assessment pelletizing iron ore _nes from Topaip and Tibirita and reducibility test response in the furnace Linder (graduation project). Tunja, Colombia: Universidad Pedagógica y Tecnológica de Colombia; 2002.

[4] Guatibonza R, Forero Pinilla A, Pérez Villamil FR. Design, construction and assembly of a reactor type Linder, for testing laboratories reducibility Paz Del Rio S.A. (master thesis). Tunja, Colombia: Universidad Pedagógica y Tecnológica de Colombia; 2005.

[5] Soto Gutierrez JF. Study of lateritic ore reducibility from Cerro Matoso S.A. (graduation project). Tunja, Colombia: Universidad Pedagógica y Tecnológica de Colombia; 2002.

[6] Mejía-A VM, Durango JR. Geology of the Cerro Matoso nickel laterites of S.A. Cordoba, Colombia; 1983.

[7] Hernández Y, Cogollo R, Almanza O. Characterization of clay nickel samples ore from the Cerro Matoso (Crdoba - Colombia). Colombian physics journal. 2006;38:285-8.

[8] Fernández and Miranda. Mechanical preparation of ores and coals. Spain: Dossat editorial S.A; 2015.

[9] Zhu DQ, Cui Y, Vining K, Hapugoda S, Douglas J, Pan J, Zheng GL. Upgrading low nickel content laterite ores using selective reduction followed by magnetic separation. International Journal of Mineral Processing. 2012;106-109:1-7.

[10] Huang Z, Yi L, Jiang T. Mechanisms of strength decrease in the initial reduction of iron ore oxide pellets. Powder Technology. 2012;221:284-6.

[11] Li J, Li X, Hu Q, Wang Z, Zhou Y, Zheng J, Liu W, Li L. Effect of pre-roasting on leaching of laterite. Hydrometallurgy. 2009;99:85-7.

[12] Jiang M, Sun T, Liu Z, Kou J, Liu N, Zhang S. Mechanism of sodium sulfate in promoting selective reduction of nickel laterite ore during reduction roasting process. International Journal of Mineral Processing. 2013;123:32-5.

[13] Valix M, Cheung WH. Study of phase transformation of laterite ores at high temperature. Minerals Engineering. 2002;15:608-12.

[14] Africano Higuera D, Pineda Triana Y. Optimization of quantitative analytical method XRF team MiniPal 2 for characterization of nickel laterite (graduation project). Tunja, Colombia: Universidad Pedagógica y Tecnológica de Colombia; 2011.

[15] Lin HY, Chen YW, Li C. The mechanism of reduction of iron oxide by hydrogen. Thermochimica Acta, 2003;400(1-2):61-7.

[16] Unidad de Planeación Minero Energética – UPME, Mines and Energy Ministry. Mining and Energy Planning Unit, Colombian nickel. Bogota, Colombia; 2009.

[17] Li B, Wang H, Wei Y. The reduction of nickel from low-grade nickel laterite ore using a solid-state deoxidization method. Minerals Engineering. 2012;24(14):1556-62.