Post-combustion Effect on Nickel and Cobalt Extractions from the Caron Process

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The conference is dedicated to the outstanding reviewers, authors, editors, and organizers of the conference to commemorate their inevitable contributions to advanced materials science and related fields. Without their participation, it would be impossible to hold 4th ICIMA 2021 successfully and ensure high quality of papers published in the conference proceedings.

Preface

We are very pleased to introduce the Proceedings of the 4th International Conference on Inventive Material Science Applications (ICIMA 2021). The 4th ICIMA event was held at PPG Institute of Technology, from May 14 to 15, 2021.

One of the significant and valuable aspects of this 4th conference edition is the way it brings together researchers, academicians, and engineers from various countries and initiates discussion on relevant issues, challenges, opportunities, and research findings. The primary focus of ICIMA 2021 is to provide an excellent platform for the conference participants to share and exchange novel and innovative ideas of original research and to build international association. The main intend of this conference is to create a smart and advanced research landscape for the areas of inventive material science.

As 282 number of submissions are received from different parts of the world, only 66 submissions were accepted as full papers for publication and presentation in ICIMA 2021. These papers provide brief illustration for current research on relevant topics, covering sustainable strategy, micro-/nano-materials, bio-materials, hybrid electronic materials, innovative electronic materials processing, computational material science, material characterization, fabrication, and synthesis technologies.

The success of the conference is due to the collective efforts of all the reviewers and advisory/review board members. We would like to express and record our gratitude and appreciation to the authors for their contributions. Many thanks to the reviewers, who helped us to maintain high-quality manuscripts included in the proceedings. We also express our sincere thanks to the members of the conference committees and organizing team for their hard work. We wish that all the authors and delegates find ICIMA 2021 proceedings interesting, exciting, and inspiring.

Coimbatore, India — Dr. V. Bindhu
Porto, Portugal — Dr. João Manuel R. S. Tavares
Cluj-Napoca, Romania — Dr. Ştefan Ţălu

Acknowledgements

The Organizing Committee of the 4th ICIMA 2021 would like to acknowledge all supporters and editors of this conference. The organizers are pleased to acknowledge the keynote speakers for their presentation on ICIMA 2021. Also we wish to acknowledge all the valuable services provided by the reviewers.

On behalf of the organizers, editors, and readers of this conference, we wish to thank the reviewers and conference technical and non-technical committee members for their time, hard work, and dedication to this conference. Without their continual services, the editors could not be able to maintain the high-quality standards of material science research. The organizers wish to acknowledge the technical program chairs for their valuable and continuous suggestion, discussion, and cooperation in organizing the conference. The organizers also wish to acknowledge the speakers and participants, who attended this conference although there is a pandemic situation across the globe.

We are pleased to acknowledge the great efforts invested by the committee in reviewing the papers submitted to the conference and organizing the sessions to enable the participants to gain maximum benefit. We would also like to express our gratitude to the unbelievable number of authors for contributing their innovative and novel research results to the conference.

Special thanks to Springer publications. We also hope to meet again in the upcoming conferences.

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About the Editors

Dr. V. Bindhu received the B.E. degree in Electronics and Communication Engineering from Bharathair University, Coimbatore in 2002 and M.E. Degree in Applied Electronics from Anna University, Chennai in 2007 and Ph.D. Degree from Anna University, Chennai in 2014. She has 10 years of teaching experience and 5 years of research experience. Currently, she is a professor at PPG Institute of Technology, Coimbatore. Her area of interest includes Signal processing, and VLSI Design.

Dr. João Manuel R. S. Tavares graduated in Mechanical Engineering at the Universidade do Porto, Portugal in 1992. He also earned his M.Sc. degree and Ph.D. degree in Electrical and Computer Engineering from the Universidade do Porto in 1995 and 2001, and attained his Habilitation in Mechanical Engineering in 2015. He is a senior researcher at the Instituto de Ciência e Inovação em Engenharia Mecânica e Engenharia Industrial (INEGI) and Full Professor at the Department of Mechanical Engineering (DEMec) of the Faculdade de Engenharia da Universidade do Porto (FEUP).

João Tavares is co-editor of more than 60 books, co-author of more than 50 book chapters, 650 articles in international and national journals and conferences, and 3 international and 3 national patents. He has been a committee member of several international and national journals and conferences, is co-founder and co-editor of the book series "Lecture Notes in Computational Vision and Biomechanics" published by Springer, founder and Editor-in-Chief of the journal "Computer Methods in Biomechanics and Biomedical Engineering: Imaging & Visualization" published by Taylor & Francis, Editor-in-Chief of the journal "Computer Methods in Biomechanics and Biomedical Engineering" published by Taylor & Francis, and co-founder and co-chair of the international conference series: CompIMAGE, ECCOMAS VipIMAGE, ICCEBS and BioDental. Additionally, he has been (co-) supervisor of several M.Sc. and Ph.D. thesis and supervisor of several post-doc projects, and has participated in many scientific projects both as researcher and as scientific coordinator.

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Chapter 43
Post-combustion Effect on Nickel and Cobalt Extractions from the Caron Process

Hugo Javier Angulo Palma, Angel Legrá Legrá, Alisa Lamorú Urgellés, Edelmira Gálvez, and Jonathan Castillo

Abstract Lateritic ores are currently considered as the fundamental raw material for the extraction of Ni and Co through the Caron process. This directly affects the temperature control of the hearth 6 of reduction furnaces from the injection of the post-combustion air into the metallurgical process. To date, there is no consensus on the part of the researchers about the positive or negative effect that this variable generates in Ni and Co extractions; therefore, this research reports the results obtained by reducing a lateritic ore on a pilot plant scale, evaluating different temperature levels in the hearth 6, as the post-combustion air was fed. It was found that the injection of the post-combustion air in the reduction furnaces decreases the Ni extractions with respect to the Co extractions, the behavior is becoming more irregular by showing maximum and minimum values. The best result of the present study is obtained when working in an operational condition without the injection of post-combustion air with a temperature of 495 °C in hearth 6 of the reduction furnace.

Keywords Nickel and cobalt extractions · Reduction furnaces · Post-combustion · Caron process

1 Introduction

Nickel (Ni) and cobalt (Co) are very important metals in the production of special alloys [1]. The main sources of Ni are lateritic and sulfurous ores. Although 70% of

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the Ni in the world is contained in lateritic ores, only 40% of production comes from this source [2, 3]. There is an increasing interest and research on lateritic ores because they contain commercially viable levels of cobalt (Co), while the availability of high-grade Ni in sulfur ores gets decreased. In contrast to Ni, the Co in lateritic ores is potentially significant for the scarcity of cobalt and current demand levels. Currently, lateritic ores are considered as the main supplier of these metals, by concentrating Ni in more than 1.0% by weight. These deposits are produced by prolonged and deep weathering of Ni silica by including ultramafic rocks, usually in humid tropical or subtropical climates. Lateritic ores can be classified as hydrated silicate deposits, clay silicate deposits and oxide deposits; the latter being processed by different technologies, among which is the Caron process [4–6].

The decision to feed lateritic ores to the Caron process is known to depend on their composition, particularly on the relationship between Fe/Ni and SiO_2/MgO. When the Mg contents exceed 2%, where the use of hydrometallurgical process, before the Caron process, is not convenient due to the considerable increase in solvent consumption, since MgO is soluble in acid. In the case of using pyrometallurgical process, the SiO_2/MgO ratio in the lateritic ores must not exceed two, and the FeO must not exceed 25% [7].

The Caron process is a technology that combines the pyrometallurgical and hydrometallurgical process, and it is based on the leaching of previously reduced lateritic ores with ammoniacal ammonium carbonate solution; the reduction being one of the stages that most influences the final extractions [8–15].

In the Caron process, the reduction of lateritic ores has carried out in a multiple hearth furnace of the Herreshoff type. These metallurgical furnaces ensure that the ore is dried, heated and reduced properly by contacting the reducing gases that flow counter currently. In order to generate these gases, the furnace has the coupled combustion chambers and a quantity of oil have added directly to the mineral, which has the function of reducing additive that enriches the reducing atmosphere [16, 17].

Different researchers have studied the reduction of lateritic ores; all agree that the temperature profile and the concentration of the reducing atmosphere are the most important variables of the process, which is why the control of the temperature from the post-combustion air supply in hearth 4 and 6 is decisive in the metallurgical efficiency of the Herreshoff furnace [18–25].

Post-combustion has called secondary air injection in the upper part of the Herreshoff furnace with the aim of burning the reducing compounds (CO and H_2) in excess. The process is important for controlling the composition of the exhaust gases by preserving the mechanical integrity of the furnace and for energy recovery [26, 27].

In spite of the energy benefits of the post-combustion process, it is necessary to have a strict control of the air supply in hearths 4 and 6, as nickel and cobalt extractions may decrease due to changes in temperature and the reducing atmosphere [28].

There is no consensus by researchers regarding the positive [27] or negative [16] effect that the variation in the post-combustion airflow can generate Ni and Co extractions from laterites. The main investigations reported are focused on modeling the process with artificial neural networks [29–31], presenting as a limitation that they have not determined the influence of the temperature in the hearth six in the nickel and cobalt extractions in multiple hearth furnaces. To determine the influence of this variable, different temperature profiles were evaluated, which were controlled from the post-combustion supply in hearths 4 and 6 in a pilot plant-scale multiple hearth furnace, determining the nickel and cobalt extractions in each of the experiments to determine its effect on the efficiency of the metallurgical process.

2 Materials and Experimental Design

This research was carried out at the pilot plant, which simulates the Caron process, of the Centro de Investigaciones del Niquel: Capitan Alberto Fernandez Montes de Oca (CEDINIQ). The reduction process was carried out in a Herreshoff furnace (Fig. 1) composed of 17 hearths, listed from top to bottom from hearth 0 (H0) to 16 (H16), enclosed in a metal cylinder 11 m high and 2,51 m in diameter, coated internally by

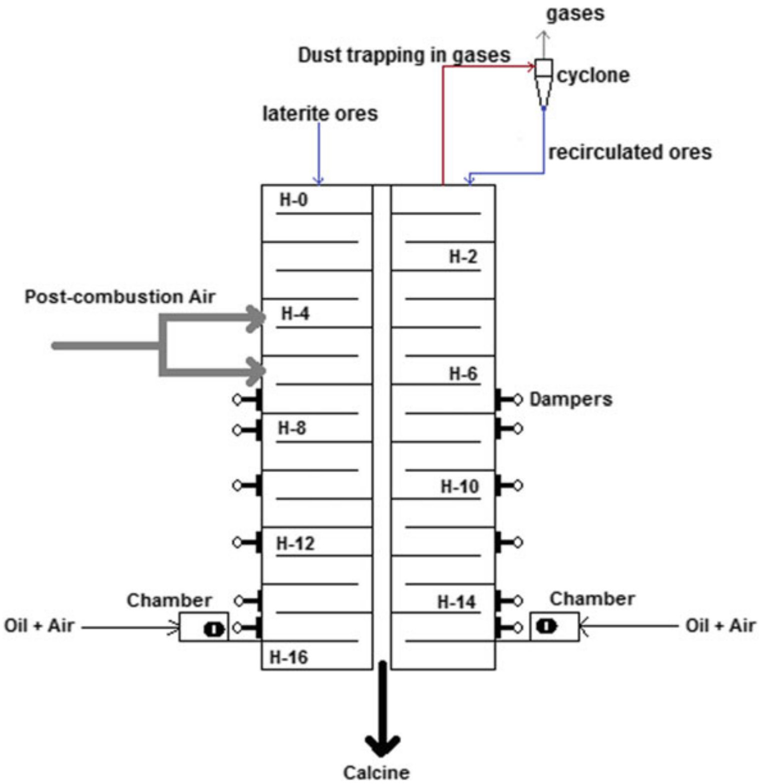

Fig. 1 Pilot plant-scale reduction furnace

a refractory material; the post-combustion air being fed by furnace hearth 4 (H4) and 6 (H6).

2.1 Physical Chemistry Characteristics of the Fed Lateritic Ore

Lateritic ore with a degree of homogenization greater than 89% was fed to the furnace at a rate of 625 kg/h, after being dried and ground until its humidity was less than 5% and presented a percentage of 86 to 88 for the fraction smaller than 75 µm. Table 1 shows its main characteristics.

2.2 Mineralogical Characteristics of the Fed Lateritic Ore

The mineralogical characteristics were determined by X-ray diffraction (PANalytical X'PERT3 –Diffractometer with Gonio-type scan at [$^\circ 2\theta$]) and the Panalytical High-Score software by processing two competing samples. Figure 2 and Table 2 present the mineralogical characteristics of the lateritic ore fed to the reduction process. It observed that the samples correspond to iron minerals with a predominance of oxides and oxy-hydroxides goethite and maghemite, as well aluminum hydroxide. The low silicon and magnesium contents were found in the lizardite and quartz phases.

2.3 Temperature Profile of the Reduction Process

In the investigation, six experiments were performed (Fig. 3). In five of them, the influence of the effect of post-combustion on Ni and Co extractions was evaluated, feeding secondary air in the H4, until a temperature between 660 and 670 °C was achieved; and in the H6 until reaching the temperatures of 660, 720, 780, 810 and 850 °C. The seventh experiment was characterized by the elimination of the post-combustion air supply in hearths four and six of the reduction furnace. Temperature measurements inside the different hearths were made using K-type thermocouples.

2.4 Nickel and Cobalt Extractions

To determine nickel and cobalt extractions, the reduced mineral (for 75 min in each of the experiments) was leached with an ammonia carbonate solution with an NH_3 concentration of 80–85 g/L and CO_2 of 40–42 g/L for two hours with a liquid/solid

Table 1 Characteristics of lateritic ore fed to the reduction process

Chemical analysis of lateritic ore, % mass							% Granulometry, size in µm			
NiO	CoO	FeO	MgO	SiO_2	Al_2O_3	H_2O	150	75	45	−45
1.190	0.112	39.917	3.204	9.563	7.694	2.584	4.26	9.02	8.46	78.26
Limonite/Serpentine										

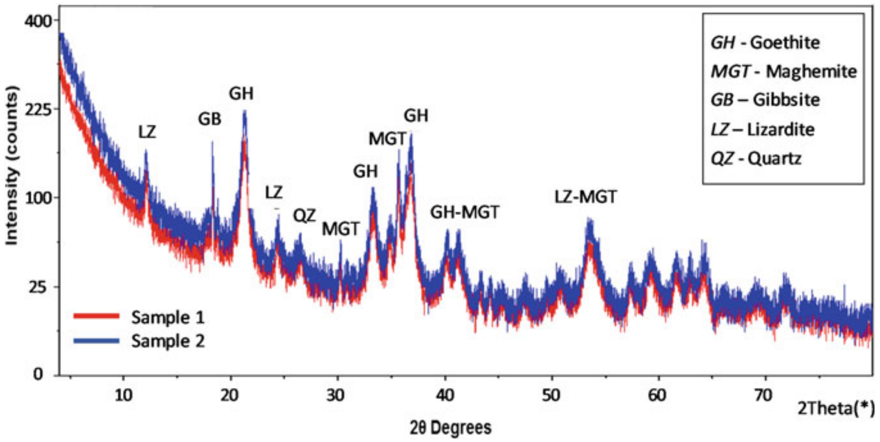

Fig. 2 X-ray diffraction diagram of samples

Table 2 Initial mineral composition

Mineral	Formula	Score
Goethite	$Fe^{+3}O(OH)$	40
Maghemite	$Fe_{21.16}\ O_{31.92}$	26
Lizardite-1\ITT\RG	$Mg_3Si_2(OH)_4O_5$	15
Gibbsite	$Al(OH)_3$	10
Quartz	SiO_2	8

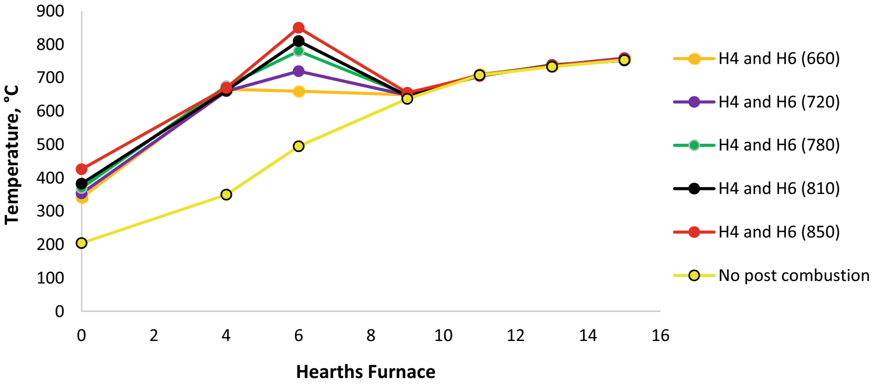

Fig. 3 Temperature profile in the reduction furnace

ratio (L/S) of 10/1. Ni and Co extractions were determined by Eq. 1, with an estimate of the error for Ni and Co of ±1.5 and 2.5, respectively.

$$\%\mathrm{Ext}_{\mathrm{Met}} = \left(1 - \frac{\mathrm{Met}_{\text{leached ore}} * \mathrm{Fe}_{\text{fed}}}{\mathrm{Met}_{\text{fed}} * \mathrm{Fe}_{\text{leached ore}}}\right) * 100 \qquad (1)$$

where:

- % Ext Met is the percentage of extractable metal under analysis, Ni or Co
- Met leached ore is the percentage of the Ni or Co in the ore after leaching
- Met fed is the percentage of Ni or Co in the ore fed to the reduction furnaces
- Fe fed is the percentage of iron in the ore fed to the reduction furnaces
- Fe leached ore is the percentage of iron in the ore after leaching

2.5 Reducing Atmosphere

The CO_2/CO ratio for each experiment ranged from 0.5 to 3.0 and evaluated from the combustion chambers to the chimney. All the experiments were carried out under the same reduction conditions in the reduction chambers and in the lower furnace hearths (from H9 to H16) with the objective of evaluating the effect of the temperature control of hearth 6 of the reduction furnace from injection of the post-combustion air. Table 3 shows the CO_2, O_2 and CO values in volumetric percentages in different areas of the furnace.

Table 3 Gaseous profile of the reduction furnace

Volumetric (%)									
Experiments	Combustion chamber			H10			Chimney		
	CO_2	O_2	CO	CO_2	O_2	CO	CO_2	O_2	CO
H4 and H6 (660)	6.9	0.0	13.0	9.5	0.0	8.8	10.5	0.4	5.6
H4 and H6 (720)	7.0	0.0	13.1	9.8	0.0	8.9	11.0	0.5	5.4
H4 and H6 (780)	6.6	0.0	13.6	8.9	0.0	8.8	11.3	0.7	5.3
H4 and H6 (810)	6.8	0.0	13.2	9.0	0.0	8.4	12.0	0.8	4.6
H4 and H6 (850)	6.5	0.0	13.5	9.5	0.0	8.6	12.5	1.0	4.1
No post-combustion	7.1	0.0	13.3	9.3	0.0	8.7	10.7	0.1	6.0

3 Results and Discussion

3.1 Effect of Post-Combustion Air on Ni Extractions

The Ni extractions are obtained during five days of continuous operation, in each of the experiments is shown in Fig. 4. They were obtained by using Eq. 1, and the methodology reported by Angulo et al. [16], taking a sample every six hours of the lateritic ore fed and reduced in the furnace.

It can be seen that the percentage of Ni extractions ranged in the range of 81.27–91.12 for the different temperature profiles evaluated. De Graaf [18] recognizes that when lateritic ores are processed with a predominance of limonitic minerals in reduction furnaces, extractions can reach up to 95%. Chang et al. [31] report similar extractions of Ni, at the Punta Gorda plant in Moa, which ranged from 76.56 to 88.07% in lateritic ores processed without homogenization.

The behavior presented by the average Ni extractions as a function of the temperature increase in H6 is presented in Fig. 5.

Judging by its behavior, it can be concluded that the temperature increase in H6, due to the feeding of the post-combustion air in the reduction furnace, exerts a negative influence on the Ni extractions that are achieved in the pyrometallurgical process, decreasing it by 1.3% for every 71 °C that increases. The maximum values of Ni extractions were achieved by processing the lateritic ores in the reduction furnace without introducing the post-combustion air. This behavior is logical and is justified because when the temperature of the hearth 6 increases, the nickel more easily changes place with magnesium in the silicates [32–35], which increases the non-leached phases of Ni in the form of olvines and complex spinels Mg, Fe, Al and Si that hinder the reduction process regardless of the reducing agent used [36].

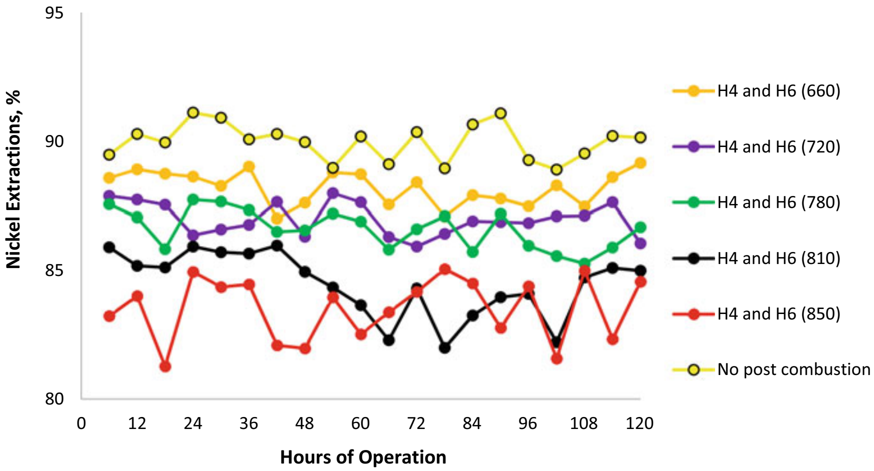

Fig. 4 Behavior of Ni extractions in each experiment

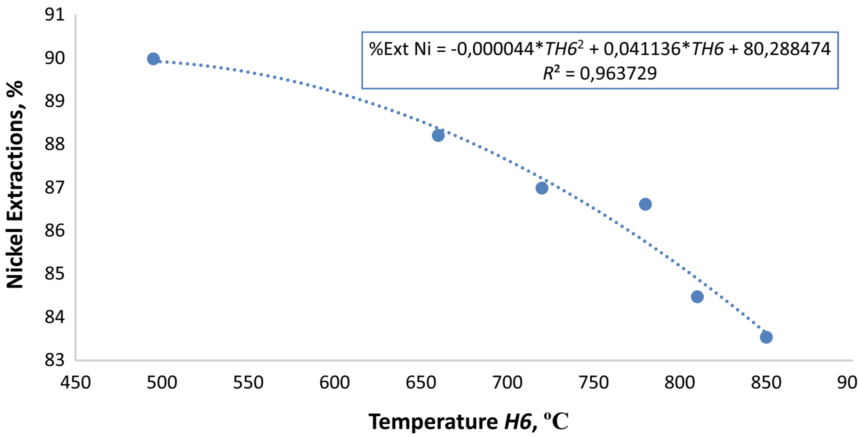

Fig. 5 Relationship between Ni extractions and temperature in H6

The mathematical model that describes this relationship corresponds to a polynomial of order two with a coefficient of determination greater than 0.96 and an error of estimation less than 0.5%.

3.2 Effect of Post-combustion Air on Co Extraction

Ni and Co extractions were determined using the same methodology. Co extractions (Fig. 6) ranged from 59 to 75%, during the five days of operation of each experiment.

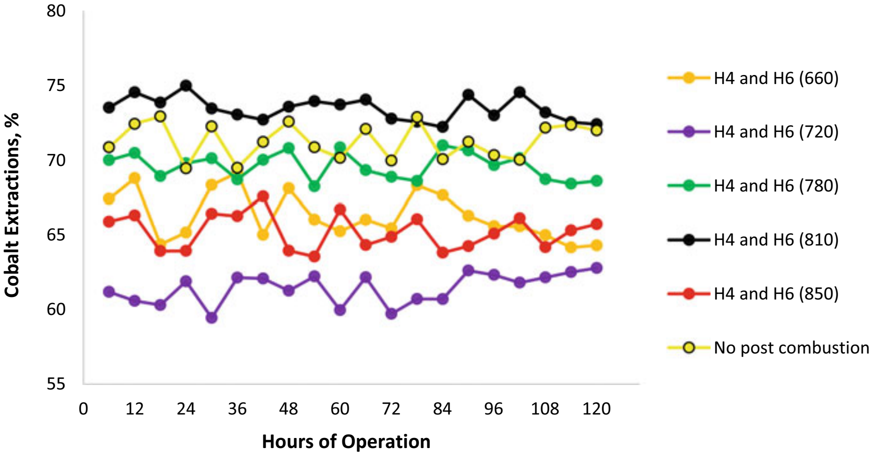

Fig. 6 Behavior of Co extractions in each experiment

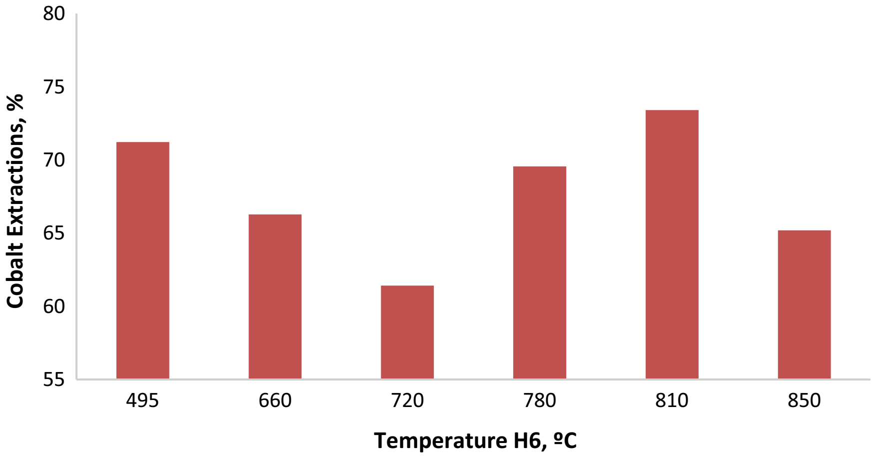

Fig. 7 Relationship between Ni extractions and temperature in H6

Kawahara, Toguri and Bergman [20] and Chang [35] report Co extractions between 40 and 75% depending on the Fe content and the mineral mineralogy, as well as the reduction temperature [37, 38].

The maximum values of Co extractions were reached by feeding the post-combustion air into H6 until a temperature of 810 °C was reached, followed by the experiment in which post-combustion air is not fed.

The effect of average Co extractions on the increase in H6 temperature is observed in Fig. 7, where irregularities are shown with respect to the behavior presented by Ni extractions. Two zones are observed in Co extractions by varying the temperature of H6 range from 495 to 850 °C.

The first zone, in the temperature range of 495–720 °C (Fig. 7), tends to show a behavior similar to that achieved by Ni extractions, characterized by a decrease of 2.1% for every 45 °C of temperature increased by the H6. The second zone, from 720 to 850 °C, was characterized by increasing Co extractions as household temperature increased to 810 °C, followed by a sharp decrease in extractions as temperature reached the highest level evaluated.

At present, the phenomena that cause the decrease in Co extractions in multiple hearth furnaces are unknown. The most correct hypothesis that allows it to be explained is the fact that at hearth 6 temperatures of 810 and 495 °C, the process of exchange of positions between Co^{2+} and Fe^{2+} decreases, forming less solid solutions that lead to a better reductibility.

4 Conclusion

The supply of the post-combustion air in the reduction furnaces of the Caron process generates variations in the Ni and Co. extractions. In the case of Ni, the increase in the temperature of the hearth 6 causes a decrease in the extractions, while the Co extractions show an irregular behavior with the presence of maximum and minimum. The best result of the study was achieved when working in an operational condition without injection of the post-combustion air with a temperature of 495 °C in hearth 6 of the reduction furnace by keeping the other variables of the pyrometallurgical process constant.

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