

Kinetic behaviour of the roasting/selective reduction process with the use of a mixture of bituminous coal and fuel oil as the additive

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Abstract

Lateritic ore is currently the main raw material that allows extraction of Ni by the Caron process. To date, the kinetic behaviour of the roasting/selective reduction process of lateritic nickeliferous ores on a pilot scale is largely unknown. In the present study, the kinetic behaviour, the controlling stage and the mechanisms that describe this process with the use of a mixture of 2 wt.% bituminous coal and 1.25 wt.% fuel oil as a reducing additive were determined during the evaluation of the high and low thermal profiles, respectively. The phases of the reduced/leached minerals and the feed ore were analysed by X-ray powder diffraction. It was observed that the mixture used as a reducing additive guarantees an adequate transformation in both thermal profiles; the relationship between the residual Ni and residence time is described by a first-order reaction with the determination coefficients greater than 0.949. Although the influence of post-combustion air is not analysed, the controlling stage was diffusion through the ash layer with an activation energy of 14.4060 kJ mol⁻¹. Thus, the most precise combination to describe the process is diffusion through the ash layer and the growth of the nuclei.

Keywords: Reaction kinetics and mechanisms; furnace operation; pyrometallurgy; lateritic nickeliferous ore; Caron process.

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1. INTRODUCTION

Nickel is considered a strategic and critical chemical element used in the production of nickel-cadmium and nickel-metal hydride batteries, as well as in the production of special steels and aerospace alloys [1]. With the explosion in the production of electric vehicles, the global demand for this metal is forecast to increase around 10 times from 2018 to 2025 [2]. Main reserves of this metal are found in sulphide and lateritic ores. As the supply of Ni sulphide ores gradually depletes, its extraction from laterite ores generates significant attention [3]. Laterites contain 72.2 % of the world's Ni reserves, with an increase in annual production from 2004 to the present from 42 to 69 % [4]. These deposits are produced by prolonged and deep weathering of Ni-silica-bearing ultramafic rocks, generally in humid tropical or subtropical climates, where the minerals will be classified into hydrated silicates, clay silicates and oxide deposits. One of the main technologies that is capable of extracting Ni from lateritic ores is the Caron process [5]. This technology combines pyrometallurgical and hydrometallurgical processes [6], and is based on leaching of previously reduced lateritic minerals with solutions of ammonium-ammoniacal carbonate ((NH₄)₂CO₃), reduction being one of the stages that most influences the extractions [7-9] and final costs [10,11].

Reduction of lateritic ore is carried out in Herreschoff multiple hearth furnaces. The furnaces are oil-fired and are operated at air deficiency to produce the required ratio of carbon monoxide to carbon dioxide in each hearth. Temperatures in the reduction zone are maintained at 700 to 800 °C [12].

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In general, different investigations recognize that the characteristics of the lateritic ore [13-15], the furnace operation [16-20] and the use of reducing additives [7,21-24] constitute the most important variables to control during the roasting/selective reduction process.

In previous research studies [25,26], specifically the ones focusing on the use of reducing additives in multiple hearth furnaces, it has been shown that the use of the mixture of bituminous coal and fuel oil at the concentrations of 2.0 wt.% and 1.25 wt.%, respectively (denoted here as BC2.0-FO1.25) benefits the process technically and economically.

Since the introduction of the reducing additive BC2.0-FO1.25 on commercial scales currently constitutes one of the main technological modifications that can benefit the Caron process, it is necessary to determine the kinetics and the controlling mechanism during the selective roasting/reduction of NiO, which is also justification of the present study.

2. METHODOLOGY

In this research, the main kinetic parameters and the controlling mechanism of the selective roasting/reduction process of NiO are determined with the use of the reducing additive of bituminous coal at 2.0 % by weight and fuel oil at 1.25 wt.% (denoted here as BC2-FO1.25) on a pilot scale. For this purpose, the installation that simulates the Caron process was used in the Centro de Investigaciones del Niquel: Alberto Fernández Montes de Oca, Moa, Cuba (CEDINIQ) [16,26]. The reduction process was carried out in a multi-hearth furnace (Fig. 1) composed of 17 hearths, listed from top to the bottom from hearth 0 (H-0) to 16 (H-16), enclosed in a metal cylinder 11 m high and 2.51 m in diameter, coated internally by a refractory material; the post-combustion air being fed by the furnace hearths 4 (H-4) and 6 (H-6). The residence time in the furnace is 80 min, considering the initial and final time at the exit of hearths 0 and 16, respectively.

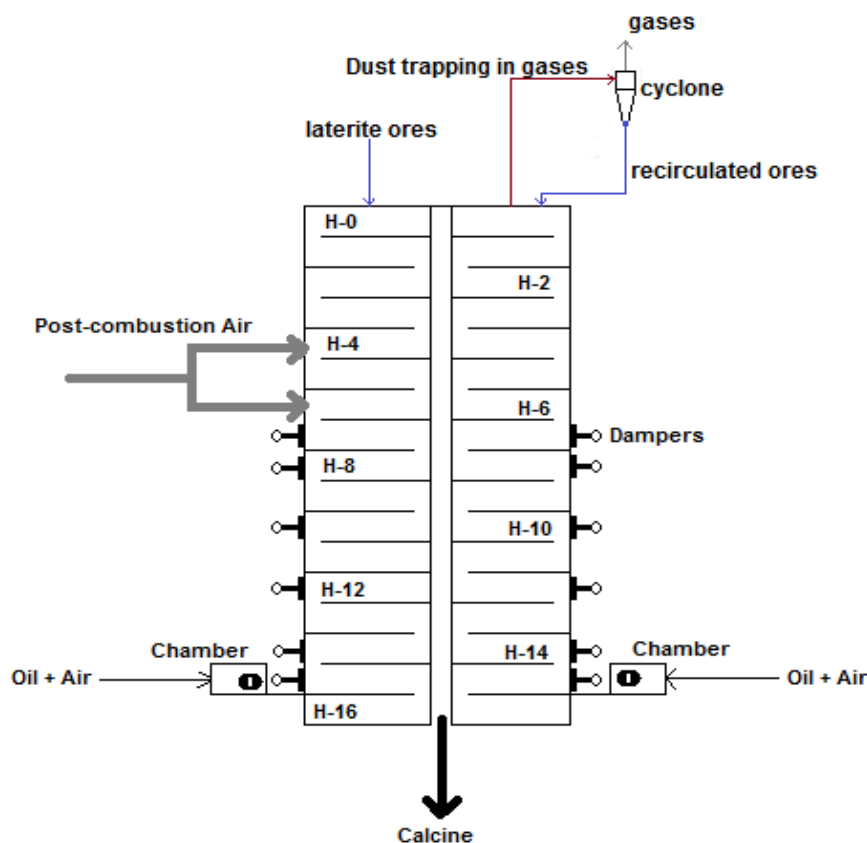


Figure 1. Pilot plant scale reduction furnace; letter H designate hearth [26]

The study was based on evaluating the conversion of NiO by analysing the kinetic data by the integral method, through the selection of kinetic equations with the objective of identifying the best fit of the experimental data, considering that the effect of reoxidation is insignificant. To confirm the controlling mechanism in the roasting/reduction processes, the value of the activation energy was used as a criterion, based on the development of the Arrhenius equation:

$$E_A = \frac{\log\left(\frac{k_{T_2}}{k_{T_1}}\right) \frac{R}{0.434152} \left(\frac{T_2 T_1}{T_2 - T_1}\right)}{1000} \quad (1)$$

where:

T_1 is the final temperature of the mineral reduced by the low thermal profile (LTP), K

T_2 is the final temperature of the mineral reduced by the high thermal profile (HTP), K

k_{T_1} is the reaction rate constant corresponding to the low thermal profile, min^{-1}

k_{T_2} is the reaction rate constant corresponding to the high thermal profile, min^{-1}

R is the universal gas constant, $8.314 \text{ J mol}^{-1} \text{ K}^{-1}$

E_A E_A is the activation energy of the reduction process, kJ mol^{-1}

Thermal profiles shown in Figure 2 were used in the investigation. The high and low thermal profiles were used to facilitate the weakening of the crystalline structure by gradual heating in the temperature ranges that are used in commercial scales [10,12,16,26]. Temperature measurements within different hearths were carried out by using type K thermocouples (Taizhou Best Electric Equipment Co., Ltd., China). Experiments were repeated 3 times for each thermal profile.

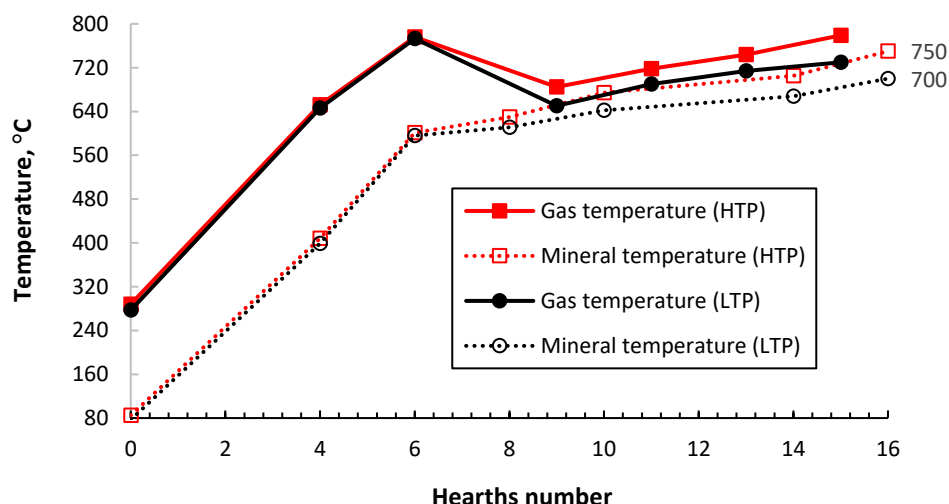


Figure 2. Temperature profiles in the reduction furnace

2. 1. Physical chemistry characteristics of the fed lateritic ore

Lateritic ore with a degree of homogenization greater than 90 % was fed to the furnace at a rate of 750 kg h^{-1} , after being dried and ground until the moisture content was less than 4 mass. % and the size distribution of particle less than $75 \mu\text{m}$ was from 84 to 88 %. Table 1 shows the main average (avg) characteristics of the mineral showing insignificant differences between the processed samples during the evaluation of the two thermal profiles in terms of the contents of Ni, Co, Fe, MgO, SiO_2 , Al_2O_3 , H_2O , C and S. The average component content (avg) of lateritic mineral in both LTP and HTP was similar with standard deviation (σ) values less than 1.56 and particle size distribution of the ore suitable for processing by the Caron process.

Table 1. Characteristics of the lateritic mineral with the two thermal profiles evaluated in the reduction process.

Thermal profile		Content, wt. %										Particle size, μm			
		Ni	Co	Fe	MgO	SiO_2	Al_2O_3	H_2O	S	C		150	75	45	<45
LTP	avg	1.165	0.098	38.921	3.153	7.696	7.916	3.225	0.255	2.081	3.844	10.078	6.598	79.481	
	σ	0.014	0.005	0.873	0.168	0.367	0.357	0.420	0.016	0.010	0.694	1.552	1.365	0.918	
HTP	avg	1.167	0.099	39.323	2.828	7.788	8.080	3.660	0.251	2.088	3.425	9.783	7.392	79.400	
	σ	0.013	0.001	0.416	0.513	0.249	0.228	0.190	0.018	0.032	0.698	0.585	1.133	1.500	

2. 2. Chemical analysis and mineralogical characteristics of the reduced/leached samples

The reduced ore samples were taken from the even hearths of the furnace using the procedure proposed previously [16,18]. Each sample was then leached with the ammonium carbonate solution at a concentration of NH_3 from 80 to 85 g dm^{-3} and CO_2 from 40 to 42 g dm^{-3} for 2 h at a liquid/solid mass ratio (L/S) of 10/1.

The individual elements of interest in the leached mineral were determined by atomic absorption spectrometry (AAS, model SOLAR 929, Solar System ATI, Unicam Analytical Technology Inc., Cambridge, UK).

The crystalline structure and composition of the reduced/leached minerals were analysed by powder X-ray diffraction (XRD) by a PANalytical X'PERT3 diffractometer (Malvern Panalytical, UK) with Goni type sweep in 2θ angular registration from 4.0042° to 79.9962° , with step distance of 0.0080° with Cu radiation and nickel filter, potential difference of 40 kV, current of 30 mA and a calibration checked by an external silicon standard scanning. Fractions of nickel, iron, and cobalt reduced to the metallic state of the ore in H16 were determined by leaching the reacted samples with a bromine-ethanol solution [27] followed by AAS analysis with flame and generator hydride (model SOLAR 929, Solar System ATI, Unicam Analytical Technology Inc., Cambridge, UK).

2. 3. Determination of the controlling mechanism in the roasting/reduction process

The controlling mechanism of the roasting/reduction process was determined by evaluating the equations of the unreacted core model reported by Levenspiel [28] and the nucleation models reported by Bo *et al.* [29], typical of heterogeneous solid-fluid reactions. Table 2 shows the equations used.

Table 2. Controlling mechanisms analysed

Model type	Controlling mechanism	Equation
Unreacted core	Diffusion through the gas film (DGF)	$x_{\text{Ni}} = k_{\text{DGF}} t$
	Diffusion through the ash layer (DAL)	$1 - 3(1 - x_{\text{Ni}})^{2/3} + 2(1 - x_{\text{Ni}}) = k_{\text{DAL}} t$
	Chemical reaction (ChR)	$1 - (1 - x_{\text{Ni}})^{1/3} = k_{\text{ChR}} t$
Nucleation	2-D growth of nuclei (GN-2)	$[-\ln(1 - x_{\text{Ni}})]^{1/2} = k_{\text{GN-2}} t$
	3-D growth of nuclei (GN-3)	$[-\ln(1 - x_{\text{Ni}})]^{1/3} = k_{\text{GN-3}} t$
	5-D growth of nuclei (GN-5)	$[-\ln(1 - x_{\text{Ni}})]^{1/5} = k_{\text{GN-5}} t$

In Table 2 x_{Ni} is the conversion ratio of Ni (degree of reduction) and t is the time, while k_{DGF} , k_{DAL} , k_{ChR} , $k_{\text{GN-2}}$, $k_{\text{GN-3}}$ and $k_{\text{GN-5}}$ are the rate constants for each proposed mechanism.

3. RESULTS AND DISCUSSIONS

3. 1. Effect of the thermal profile with the use of the BC2-FO1.25 additive

Figure 3 shows the XRD patterns of the fed and reduced/leached lateritic ore samples corresponding to each thermal profile analysed.

It is observed that iron oxides and oxihydroxides (goethite, maghemite and hematite) as well as aluminium hydroxide (gibbsite) predominated in the lateritic ore fed. The secondary phases grouped the contents of silicon and magnesium, identifying the phases of hydrated magnesium silicate (lizardite), aluminium and magnesium oxide (spinel) and some silicon oxide (quartz).

The main mineralogical phases identified in the reduced/leached mineral, regardless of the thermal profile evaluated, were iron oxides (magnetite and maghemite, syn), magnesium iron aluminium chromium oxide (magnesiocromite, ferroan) and magnesium iron silicate (fayalite). These identified phases coincide with those reported by different studies [26,30] and indicate that during the evaluation of both thermal profiles an adequate roasting reduction process is achieved.

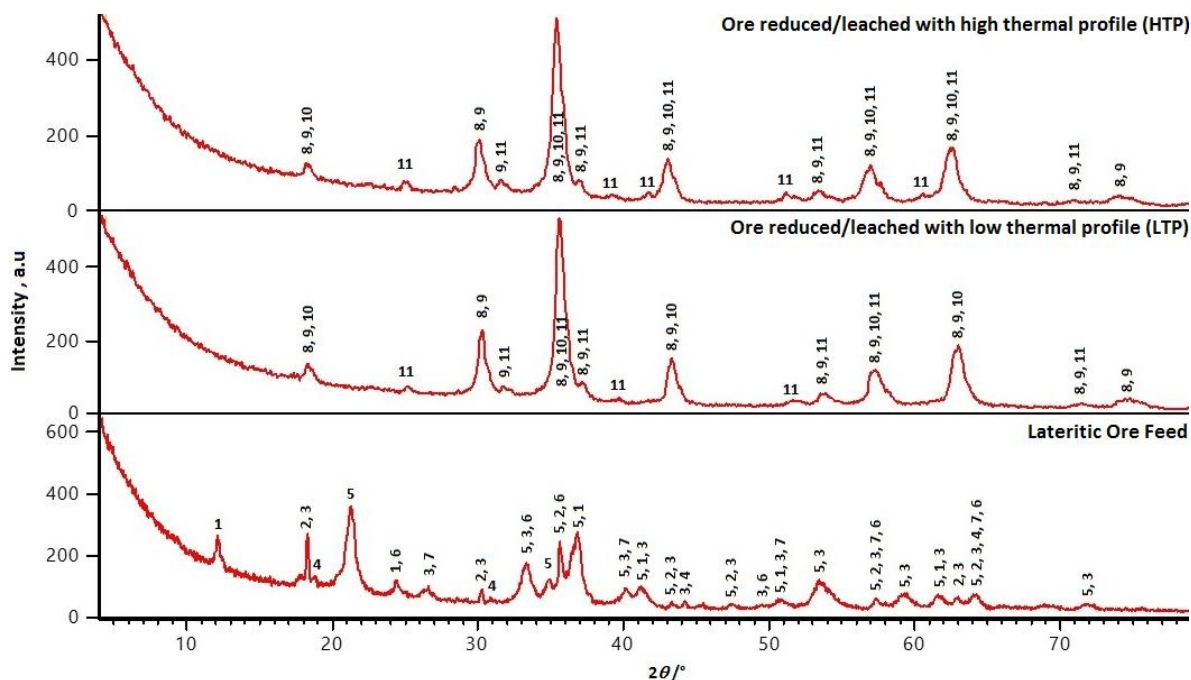


Figure 3. XRD patterns of reduced/leached lateritic mineral samples with the use of BC2-FO1.25 additive and two thermal profiles. 1 - lizardite: $\text{Mg}_3\text{Si}_2\text{O}_5(\text{OH})_4$; 2 - maghemite: $\text{Fe}_{21.16}\text{O}_{31.92}$; 3 - gibbsite: $\text{Al}(\text{OH})_3$; 4 - spinel: MgAl_2O_4 ; 5 - goethite: FeOOH ; 6 - hematite: Fe_2O_3 ; 7 - quartz: SiO_2 ; 8 - magnetite, syn: Fe_3O_4 ; 9 - maghemite, syn: Fe_2O_3 ; 10 - magnesiochromite, ferroan: $[(\text{Mg}, \text{Fe})(\text{Cr}, \text{Al})_2\text{O}_4]$ and 11 - fayalite: $\text{Mg}_{26}\text{Fe}_{1.74}(\text{SiO}_4)$.

Formation of fayalite and magnesiumchromite, ferroane in the reduced and leached mineral (Fig. 3), justify the presence of non-leachable Ni contents in the process. These results agree with those obtained by Rhamdhani *et al.* [31-33], when identifying that the mineralogical phases of olivine and ferro-chromite spinels are not leached by the carbonate-ammonia technology. The presence of fayalite in both thermal profiles confirms that the reduced mineral reaches temperatures greater than and equal to 700 °C and that the use of the additive BC2-FO1.25 allows an adequate transformation of the serpentinitic phases. This finding coincides with the results of Valix & Cheung [34,35] during the evaluation of S as a reducing additive in the Caron process in the temperature range of 600 to 800 °C.

Although both thermal profiles present similarity, the shape of the peaks in the high thermal profile (HTP) became sharper, indicating an increase in the crystalline nature of the roasted product.

3. 2. Activation energy corresponding to the roasting/reduction process with the use of the additive BC2-FO1.25

Figure 4 relates the average $\text{Ni}_{\text{residual}}$ (residual Ni mass percentage after the reduction/leaching process) in the pilot Herreshoff furnace as a function of time, for the two thermal profiles evaluated. All data are presented in Supplementary Material S1 and S2. The decrease in the percentage of residual Ni as the residence time increases for both thermal profiles is observed as a trend, a regularity typical of an adequate reduction process in which the effects of reoxidation of the reduced mineral are minimized.

The behaviour presented by the high and low thermal profile curves indicates that the kinetic process of the general transformation of NiO into Ni^0 adjusts to a first-order reaction with a coefficient of determination greater than 0.9490; this behaviour coincides with that reported by Shoubao [36] and Castellanos [37] during the evaluation of the roasting/reduction process of nickel-bearing lateritic ores. The average feed Ni percentage ranged from 1.1550 to 1.1780 wt.%, which corresponds to the chemical characteristics of the lateritic ore used in the study, see Table 1.

The average reaction rate constant at the low thermal profile (k_{T1}) was 0.0176 min^{-1} , which is for about 8.3 % lower than that achieved when using the high temperature profile (Figure 4); aspect that confirms compliance with the Arrhenius equation. Substitution of the values of the reaction rate constant and the final temperature of the reduced mineral in Equation (1) allows obtaining an activation energy of $14.4060 \text{ kJ mol}^{-1}$ when using the additive BC2-FO1.25 in the process of roasting/reduction (Table 3).

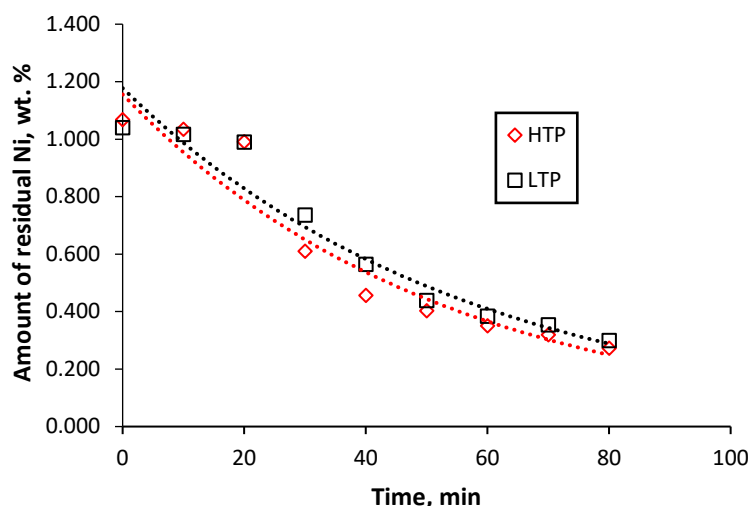


Figure 4. NiO reduction kinetics on a pilot scale at two temperature profiles and with the use of the BC2-FO1.25 additive

Table 3. Activation energy and reaction rate constants with the reducing additive BC2-FO1.25

Thermal profile				E_A / kJ mol ⁻¹
Low		High		
k_{T_1} / min ⁻¹	T_1 / °C	k_{T_2} / min ⁻¹	T_2 / °C	
0.0176	700	0.0192	750	14.4060

The value of the activation energy with the BC2-FO1.25 additive corresponds to those reported, at laboratory scale during the evaluation of the reduction process in the temperature range of 675 to 800 °C, by Castellanos [37] (17.1544 kJ mol⁻¹ for laterites from the Nicaro region with the fuel oil additive at 2.5 wt.%) and Ilyas [21] (13.80 kJ mol⁻¹ for laterites from Bajaur, Pakistan with a mixture of 10 wt.% of coal and 9 wt.% of Na₂SO₄).

3. 3. Determination of the controlling stage of the roasting/reduction process with the use of the additive BC2-FO1.25

Table 4 shows the results of determination of the controlling stage in the roasting/reduction process for the high and low thermal profiles, when considering the unreacted nucleus models for particles of constant size and growth of nuclei.

Table 4. Predicted times for reaction completion according to different controlling mechanisms shown in Table 2

Thermal profile	Complete reaction time, min					
	Chemical reaction	Diffusion		Growth of nuclei		
		Through the gas film	Through the ash layer	GN-2	GN-3	GN-5
High	203	98	277	59	59	59
Low	230	103	336	64	62	61

The analysis of the complete reaction times allows us to identify that the controlling stage of the process is diffusion through the ash layer (DAL), with times necessary to complete the reaction of 277 and 336 min for the high and low thermal profiles, respectively. The controlling stage in the process was directly related to the activation energy value obtained in this study (value less than 17 kJ mol⁻¹, Table 3) and to the mechanism reported during the roasting/reduction of lateritic ores in different investigations [14,21,36-38].

To identify the mechanism that best describes the lateritic ore roasting/reduction process with the use of the additive BC2-FO1.25 and the high and low thermal profiles, possible combinations between the ChR, DGF, DAL and GN models were evaluated. To identify the mechanism that best describes the lateritic ore roasting/reduction process with the use of the additive BC2-FO1.25 and the high and low thermal profiles, possible combinations between the ChR, DGF, DAL and GN models were evaluated, according to the procedure described by Levenspiel [28]. The mechanism that best describes the roasting/reduction process in the low thermal profile (LTP) is the combination of the diffusion model through the ash layer (DAL) and growth of the nuclei (GN-5) with a coefficient of determination of 0.9838. In the case

of the high thermal profile (HTP), it is observed that the most accurate combination to describe the process is diffusion through the ash layer (DAL) and the growth of nuclei (GN-3) with a correlation of 0.9623. (See Figure 5). These results are logical and justified because the increase in temperature causes an increase in the work per gram-molecule necessary to form a growth nucleus.

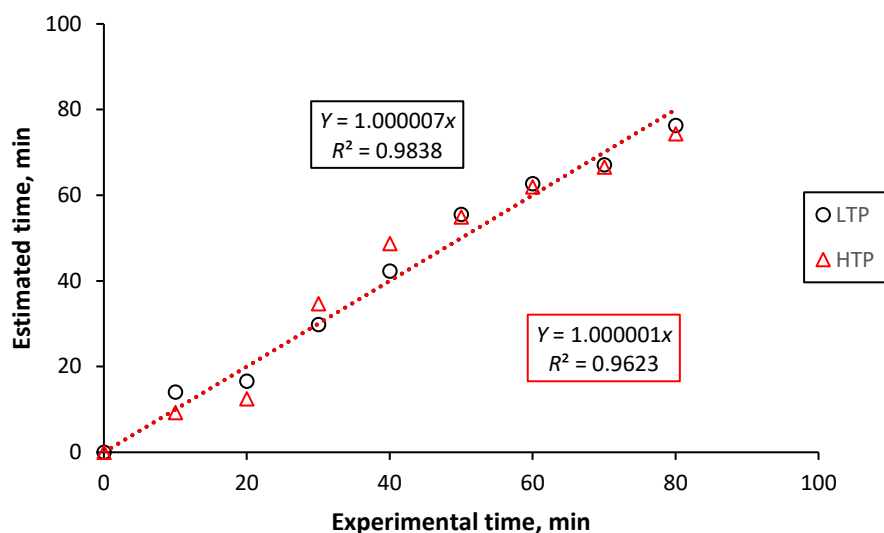


Figure 5. Combinations of mechanisms (DAL and GN) to describe the roasting/reduction process of lateritic ore with the use of the additive BC2-FO1.25

4. CONCLUSIONS

The BC2-FO1.25 additive can guarantee adequate mineralogical transformations in the roasting/reduction process of lateritic ores with a predominance of iron oxides-oxihydroxides at the use of high and low thermal profiles. The results of this study suggest that the relationship between the average Ni_{residual} percentage after the reduction/leaching process and residence time is described by a first-order reaction with determination coefficients greater than 0.9490. The pilot-scale kinetic behaviours for both thermal profiles evaluated indicate that the controlling mechanism is diffusion through the ash layer, with complete reaction times that ranged from 276 to 336 min and an activation energy of $14.4060 \text{ kJ mol}^{-1}$.

Supplementary material

Additional data are available electronically at <https://www.ache-pub.org.rs/index.php/HemInd/article/view/1385> or from the corresponding author on request.

Declaration of interest statement: The authors declare they have no conflict of interest.

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Kinetika procesa pečenja/selektivne redukcije uz upotrebu mešavine bitumenskog uglja i lož ulja kao aditiva

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(Stručni rad)

Izvod

Lateritna ruda je trenutno glavna sirovina koja omogućava ekstrakciju nikla Caron procesom. Do danas, kinetičko ponašanje procesa pečenja/selektivne redukcije lateritnih ruda nikla u pilot postrojenju je nedovoljno istraženo. U ovoj studiji, kinetičko ponašanje, faza koja kontrolira i mehanizmi koji opisuju ovaj proces primenom mešavine 2,00 mas.% bitumenskog uglja i 1,25 mas.% mazuta kao redukcionog aditiva određivani su tokom evaluacije visokog i niskog toplotnog profila. Faze redukovanih/izluženih minerala i polazne rude analizirane su rendgenskom difrakcijom praha. Pokazano je da smeša koja se koristi kao redukcionim aditiv garantuje adekvatnu transformaciju u oba termička profila; odnos između zaostalog nikla i vremena zadržavanja je opisan reakcijom prvog reda sa koeficijentima determinacije većim od 0,949. Iako nije analiziran uticaj vazduha posle sagorevanja, faza koja kontrolira proces je bila difuzija kroz sloj pepela sa energijom aktivacije od 14,4060 kJ mol⁻¹. Dakle, najpreciznija kombinacija za opisivanje procesa je difuzija kroz sloj pepela i rast jezgara.

Ključne reči: kinetika i mehanizmi reakcije; rad peći; pirometalurgija; lateritna ruda nikla; Caron proces



