

Separation of Iron and Calcium from a BSSF Steelmaking Slag Through Acid Leaching

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Considering the BSSF steelmaking slag generated in a Brazilian steelwork, it would be worth to separate iron and calcium from it, with the objective of increasing its iron content, or producing Fe_2O_3 and CaO by selective precipitation from cations in aqueous solution. In this way, materials could be produced and used in-house, e.g., sintering plant. This possibility was explored through acid leaching for an industrial sample. A possibility to increase concentration of iron of the slag is to leach it with a 2 mol.L⁻¹ HCl solution; in this condition, 63.6% of the CaO is extracted from the slag to the aqueous solution, and Fe content of the slag increases from 27.4 to 37.1 wt%. Another possibility is to use HCl 5 mol.L⁻¹; in this condition, 86.8% of the Fe and 90.7% of the CaO are transferred to the liquid solution. Then separation of iron and calcium can be achieved by selective precipitation: firstly, adding ammonia to precipitate $\text{Fe}(\text{OH})_3$, and then calcinating it to produce Fe_2O_3 (~ 200 °C); in the following, adding sodium carbonate to precipitate CaCO_3 , which in turn can be calcinated to produce CaO (~ 900 °C). In this way, Fe_2O_3 and CaO could be produced.

Keywords: *Steelmaking, BSSF, BOF slag, LD slag, leaching, slag valorization.*

1. Introduction

Steel plants produce several wastes and by-products on each process stage, ranging from ironmaking to steelmaking. Blast furnace slags, for example, are wet granulated and utilized in the cement industry; for this by-product, there is value-added market and proven technical solutions. However, this is not the case for BOF (basic oxygen furnace) slags. Among the existing uses for BOF slags, it can be mentioned: (i) as construction material, e.g., road making and railway ballast material; (ii) cement production, with restrictions, since maximum Fe_2O_3 content in cement should not exceed 5%, and, besides, P_2O_5 contained in slag increases corrosion rate of steel structures in concrete; and (iii) restoration of marine ecology¹⁻⁵. Considering that a huge amount of steelmaking slag is generated in the converter – around 120 kg per ton of produced steel –, more initiatives are necessary to consume it. Innovative ideas for conversion of the BOF slag to value-added products are required, considering sustainable growth of the steel industry.

It is difficult to recycle BOF slag in ironmaking or steelmaking processes, due to its high phosphorus content (~ 2% P_2O_5)⁵. Some researchers have shown, in laboratory scale, that it is possible to remove phosphorus via chemical leaching^{6,7}. In this case, the idea is to use the leaching liquor in agriculture.

The usual way for treating BOF slag is simple pouring and cooling. This procedure requires an area in the plant for slag stabilization, which is a process that can last for months. The BOF slag is instable due to the swelling phenomenon,

which is associated with the presence of free lime (CaO) and free periclase (MgO) in the solidified slag. CaO and MgO react with moisture, resulting in expansion due to the formation of hydroxides. The steelmaking slag must be stable before applications such as road construction; the presence of free CaO/MgO is related to instability⁸.

Intensive efforts have been made in the last decades for BOF slag valorization, including industrial facility developments for slag treatment. In Chinese steel plants there are several hot stage processes for treating the BOF slag, for example, the pyrolytic self-slaking process, the HK process, and the Baosteel Slag Short Flow (BSSF). Considering the BSSF process, it treats high temperature BOF molten slag in a special rotating cylinder; under the co-effect of the mechanical forces due to steel balls hitting and water chilling, molten slag is rapidly quenched and crushed, and the slaking (stabilization) of free calcium and magnesia is rapidly completed. Then, magnetic separation is used for dressing of steel particles and iron oxides. The formed BSSF granulated slag is normally smaller than 15 mm and stable; it can be applied in concrete mixture, cement raw material, building materials, fluxes, road construction, and sintering additive^{1,9}. Free lime in the BSSF treated slag is about 1 wt%.

The objective of the present work is to study possibilities for separating iron and calcium oxides from a BSSF steelmaking slag, considering a sample got from a Brazilian steel plant, through acid leaching. Leaching behavior for this slag was examined considering aqueous solutions containing hydrochloric acid and acetic acid, for different concentrations.

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2. Materials and Methods

2.1. As-received sample and leaching solid residue characterization

A representative BSSF slag sample, 20 kg, was collected from a Brazilian steelwork. Firstly, it was treated with H_2O_2 to remove carbon. Then, it was crushed and sieved to < 250 mesh. Through X-ray Diffraction (XRD), mineral phases were determined, with a diffractometer XPert Pro MPD-PANalytical, with a copper tube. Identification of the crystalline phases was carried out with the X'Pert High Score Plus software. Elemental composition of the BSSF slag sample was analyzed by a quantitative X-ray Fluorescence (XRF) apparatus, for the as-received material and for the remaining slag after the leaching processes (solid residue). Titration was used to determine Fe_{total} .

Chemical composition (wt%) of the as-received BSSF slag sample (quantitative XRF and titration for total iron) is shown at Table 1. Besides, typical range of chemical composition for BOF slags produced in Brazilian plants can be seen¹⁰. The high iron oxide content is due to the oxidizing conditions in the converter during production of steel.

2.2. Leaching tests

Leaching experimental setup is shown at Figure 1. It was performed with orbital agitating and air bubbling for 3 hours. For each leaching test, 20 g of slag sample was mixed with 100 ml of acid aqueous solution, in an Erlenmeyer. For hydrochloric acid and for acetic acid, concentrations in the range 0.5-5.0 mol.L⁻¹ were tested. After the leaching processes, the leaching liquor (supernatant) and the solid residue were separated by vacuum filtration (38 L min⁻¹, reaching 695 mmHg). The solutions with acetic acid for the concentrations of 3, 4, and 5 mol.L⁻¹ were submitted to vacuum filtration once again since presented red precipitate, i.e., $Fe(OH)_3$ after dilution. This is explained by the fact that iron cations can precipitate in the form of hydroxide from pH >2¹¹; this precipitate was added to the solid residue. After the leaching processes, supernatant and solid residue were analyzed.

2.3. Qualitative analysis for iron and calcium in leaching liquor

To evaluate the presence of calcium and iron cations in the leaching liquor, firstly qualitative analysis tests were performed. Regarding iron, an aliquot quantity of 1 ml was taken; it was diluted, acidified, and then drops of potassium thiocyanate (KSCN) were added.

Considering qualitative analysis for calcium cations, with an aliquot quantity of 1 ml of leaching liquor, it was diluted, and then drops of concentrated sodium hydroxide

0.1 mol.L⁻¹ until pH 8 were added. Na_2CO_3 was used to identify calcium by $CaCO_3$ precipitation.

2.4. Quantitative analysis for iron and calcium in leaching liquor

The samples were diluted (1:50) and then analyzed in duplicate, using standard methods for water analysis¹². Total iron content was determined by the spectrophotometric (orthophenanthroline) method, and calcium by the EDTA titration method.

2.5. Separation of iron and calcium from the liquor

Separation of iron and calcium ions from the liquor was performed using the difference between the pH ranges related to the precipitation of the respective hydroxides, i.e., selective precipitation. Ammonium hydroxide would be the indicated option for this separation (since it is a weak base), however, the use of a low concentration solution of sodium hydroxide produces the same effect. The use of NaOH 1.0 mol.L⁻¹ solution facilitates the procedure because it does not require the use of an exhaust system, which would be mandatory for the use of ammonia hydroxide solution, for it releases toxic gases.

3. Results and Discussion

3.1. Characterization of the as-received material

The elemental chemical composition of the as-received BSSF slag is shown in Table 1. Main elements are iron, which is contained in the form of iron oxides, and calcium. The X-ray diffractogram for the as-received sample is shown in Figure 2. The major detected phases are β - Ca_2SiO_4 , α - Ca_2SiO_4 , merwinite, and sebrodskite. Minor phases are hatrurite, wustite, and lime.

All the phases which were detected by XRD were expected, according to the review of Yildirim and Prezzi¹³; they reported that the common mineral phases present in steel slags typically include merwinite ($3CaO \cdot MgO \cdot 2SiO_2$), olivine ($2MgO \cdot 2FeO \cdot SiO_2$), dicalcium silicate (α and β), $4CaO \cdot Al_2O_3 \cdot Fe_2O_3$, $2CaO \cdot Fe_2O_3$, CaO (free lime), MgO, FeO (wustite), $3CaO \cdot SiO_2$ (hatrurite) and the RO phase (a solid solution of $CaO \cdot FeO \cdot MnO \cdot MgO$). Besides, solid solutions of FeO (wustite) are observed.

BOF slags with basicity between 3.1 and 3.5 typically contain 7–13% of free CaO based on the steelmaking practice and cause volume instability on hydration by forming calcium hydroxide. The free lime content increases sharply with increasing of CaO/SiO₂ ratio from 3.5 to 4.5 and its amount is significantly enhanced by a slow cooling rate¹⁴. In the present work, basicity value is 4.8. However, cooling rate

Table 1. Chemical composition (wt%) of the as-received BSSF slag sample, and typical range of chemical composition (wt%) for BOF slags produced in Brazil¹⁰.

	Fe_{total}	CaO	SiO ₂	MgO	Al ₂ O ₃	P ₂ O ₅	MnO
As-received BSSF slag sample (wt%)	27.42	42.14	8.86	8.68	1.75	1.98	1.95
BOF typical chemical composition (wt%)	14.0-22.0	23.0- 47.0	11.0- 18.0	5.0- 13.0	1.0- 4.0	1.0- 3.0	3.0- 7.0

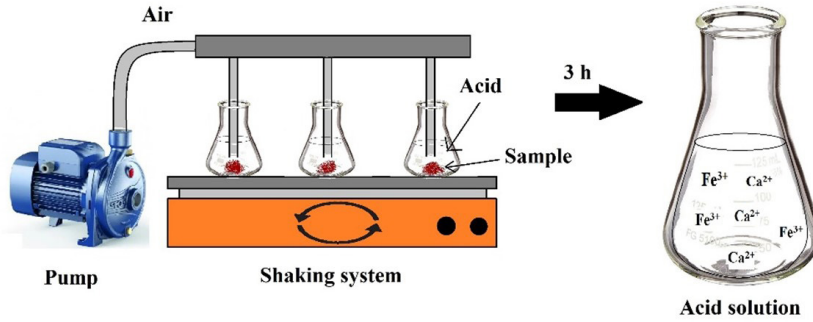


Figure 1. Experimental setup for the leaching process applied for the BSSF slag sample.

is high in the BSSF reactor, thus free lime content should not be high. Free lime was detected by X-ray diffraction, but it is not a major phase. Besides, while slag is cooled with water, it is ground simultaneously; this should influence on the phase transformations.

Crystal formation is a function of both the chemical composition of the melt and its cooling rate. Silica rich blast-furnace slag vitrifies (forms a glassy phase) easily when it is rapidly cooled. Steel slag has a lower silica content than blast furnace slag and, hence, steel slag seldom vitrifies when rapidly cooled. Even for the remarkably high cooling rate of the BSSF reactor, crystallization from melt is still intense, according to the diffractogram of Figure 2.

There are few reports in the literature regarding the influence of cooling rate in relation to the properties of slag, especially when it comes to steelmaking slags – BOF and EAF (electric arc furnace) slags. Engström et al.¹⁵ studied the influence of the cooling rate, regarding mineralogy of steelmaking slags. For the semi-rapidly cooled BOF slag (cooling rate was approximately 0.3 K/s within the first hour and 0.04 K/s thereafter), they found the following crystalline phases: a solid solution of magnesium, iron and manganese oxide (Mg,Fe,Mn)O, calcium ferrite (Ca₂Fe₂O₅), calcium manganese oxide (Ca,Mn)O and β-dicalcium silicate (β-Ca₂SiO₄). In the rapidly cooled BOF slag (500 K/s), three crystalline phases were detected: tricalcium silicate (Ca₃SiO₅), α-dicalcium silicate (α-Ca₂SiO₄), and a solid solution of magnesium, iron and manganese oxide (Mg,Fe,Mn)O. All these phases are expected for BOF slags¹³. Ca₃SiO₅ and α-Ca₂SiO₄ are metastable phases at room temperature, because they are stable at higher temperatures and remain in slag due to fast cooling¹⁵; both were detected in the present work (Figure 2).

In Portland cement, the hydraulically important polymorph of dicalcium silicate is β-Ca₂SiO₄. On cooling from elevated temperatures (during clinker production), α-Ca₂SiO₄ passes through several closely related polymorphs before transforming to β-Ca₂SiO₄ at 630 °C. It is also possible a transformation at lower temperature to the γ-modification, accompanied by a volume increase responsible for the well-known phenomenon of ‘dusting’¹⁶.

3.2. Qualitative analysis for iron and calcium in leaching liquor

The relevant acid leaching reactions can be represented in the following way:

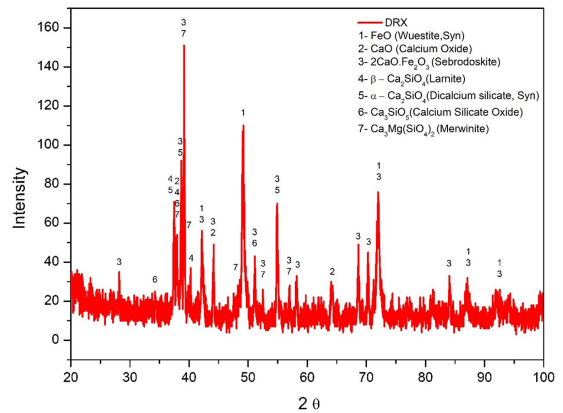
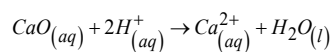
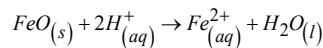


Figure 2. XRD diffractogram for the as-received BSSF steelmaking slag.



Due to air bubbling ferrous iron is transformed to ferric iron¹¹:

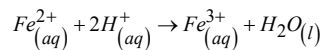


Figure 3 shows results for the qualitative test for iron, for the samples leached with HCl. The higher the concentration of HCl, more intense the red color becomes, since it is related with iron thiocyanate.

The red color which is observed in the qualitative test for iron is related to the complexation reaction of iron with potassium thiocyanate. In slightly acidic solution, intense red appears quickly due to the formation of a non-dissociated complex of iron thiocyanate¹¹, according to the reaction $Fe^{3+}(aq) + 3SCN^-(aq) \rightarrow Fe(SCN)_3(aq)$. Figure 3 shows that leaching is more effective when increasing acid concentration. For acetic acid, the behavior is similar, however, red color is less intense for the same values of molar concentrations, i.e., acetic acid removes lower amount of iron during leaching process.

Regarding the qualitative test for calcium, the white precipitate (CaCO₃), which is related to the presence of calcium

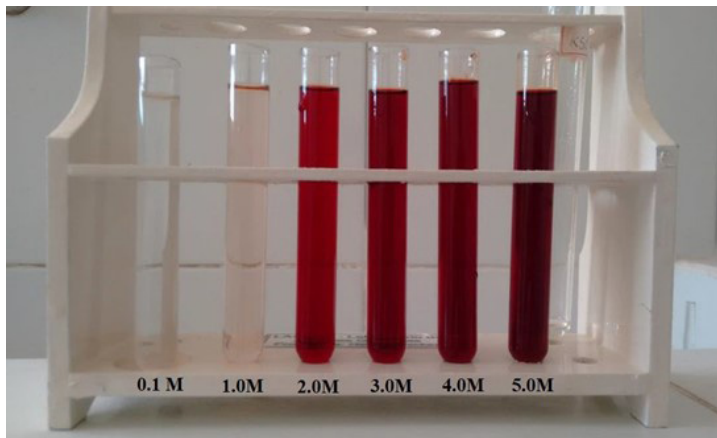


Figure 3. Qualitative test results for Fe^{3+} when using HCl for leaching, showing iron thiocyanate (red color).

cations in supernatant, was observed for all concentrations and for both acids. Calcium cations do not precipitate under presence of hydroxide ion, because calcium hydroxide presents high solubility in pH below 12.5¹¹. Solubility product of $\text{Ca}(\text{OH})_2$ is around 1×10^{-6} , and for $\text{Fe}(\text{OH})_3$ is only 1×10^{-39} . Therefore, iron hydroxide precipitates first. Thus, when adding NaOH to remove iron from solution, this will not affect the content of calcium cation in solution. The test tubes were centrifuged at 4000 rpm during 5 min, and then another aliquot was taken from the supernatant. Then precipitation of calcium was performed adding sodium carbonate, generating calcium carbonate according to the reaction $\text{Ca}^{2+}_{(aq)} + \text{CO}_3^{2-}_{(aq)} \rightarrow \text{CaCO}_3(s)$.

In this way, the white precipitate in the test tube was related to the presence of calcium cations in the supernatant.

3.3. Quantitative analysis for iron and calcium in leaching liquor

Figures 4 and 5 show comparisons between HCl and CH_3COOH , regarding iron and calcium extraction from the sample of BSSF slag. Both elements are more easily transferred to leaching liquor when using HCl. For Figure 4, the %Fe (y-axis) was calculated considering 5.48 g of Fe as 100%, which is the mass of Fe contained in 20.0 g of the as-received sample. For Figure 5, the %CaO (y-axis) was calculated considering 8.43 g of CaO as 100%, which is the mass of CaO contained in 20.0 g of the as-received sample.

As in the analysis of iron content, hydrochloric acid is more effective in removing calcium than acetic acid. However, with acetic acid 55.1% of CaO can be removed from the as-received slag (when using 5 M) and simultaneously maintaining most of the iron in solid slag (only 5 wt% Fe is removed to the solution). This fact could be used to increase the iron content of the BSSF slag, considering in-house solutions such as sintering plant. However, the phosphorus content is a problem.

3.4. Quantitative analysis for the solid residue after leaching

Table 2 shows percentage change of Fe e CaO after leaching, which was calculated from XRF results and titration for Fe_{total}

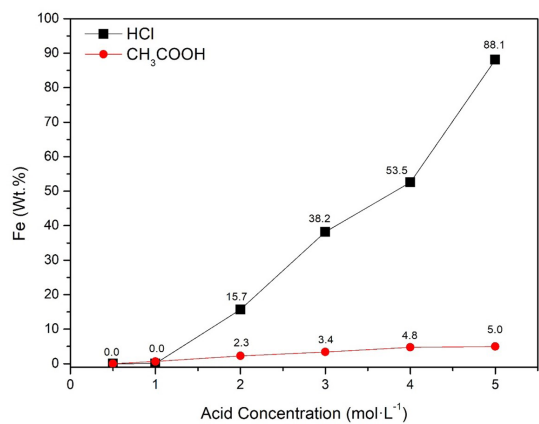


Figure 4. Iron content (wt.%) of the filtrate (leaching liquor) when using HCl and CH_3COOH for leaching, with different molar concentrations, according to the spectrophotometric (orthophenanthroline) method.

Table 2. Percentage change of Fe e CaO after BSSF slag leaching.

[HCl]	Sample mass wt% change	Fe wt% change	CaO wt% change
0.5 M	-8.5	0	-32.2
1.0 M	-15.2	0	-50.3
2.0 M	-35.9	-13.3	-68.3
3.0 M	-52.7	-43.5	-71.6
4.0 M	-66.6	-57.0	-83.6
5.0 M	-86.6	-85.6	-91.7
[CH ₃ COOH]			
0.5 M	-11.4	-0.6	-31.4
2.0 M	-19.3	-0.7	-43.5
3.0 M	-20.3	-1.1	-44.0
4.0 M	-24.1	-4.7	-48.8
5.0 M	-25.5	-4.5	-51.0

for the remaining solid residue of BSSF slag. The residue mass decreases when increasing acid concentration, in a more intense way for HCl. When comparing Table 2 with Figures 4 and 5, the different methods for determination of CaO and Fe are coherent since comparable results are obtained.

Crystalline phases have an important role in slag leaching; the content of a certain element can never be directly correlated to the leaching behavior without considering in what phase it is present⁸. Solubility of steel slags have been studied; it was found, for some steel slag samples¹⁷, that the Ca-bearing mineral larnite was the most soluble phase in deionized water, followed by brownmillerite. Larnite and hatrurite are examples of hydraulic phases, where the hydration process facilitates the migration of species such as Ca into water. Merwinite shows high reactivity at pH 4 and pH 7¹⁸. On the other side, experiments indicate that FeO (wustite) is insoluble at pH=4¹⁷.

These findings can help to explain the results of the present work. Larnite and hatrurite are hydraulic crystals which are present in the BSSF slag sample, as major and minor phase respectively; their relatively fast dissolution would explain the high levels of dissolved calcium even with low acid concentrations. A similar behavior is expected for merwinite, another major phase identified by X-ray diffraction.

With higher acid concentrations, dissolution of the other Ca-bearing and Fe-bearing crystals should occur.

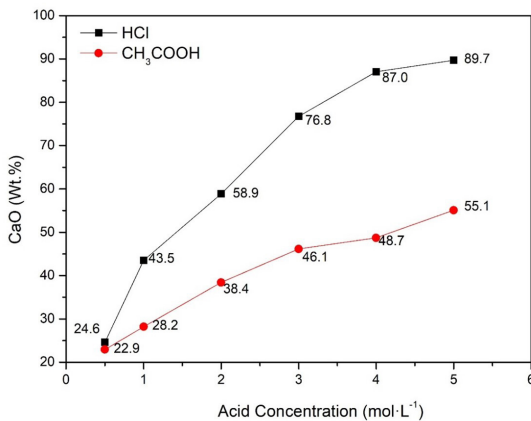


Figure 5. Calcium oxide content (wt.%) of the filtrate (leaching liquor) when using HCl and CH₃COOH for leaching, with different molar concentrations, according to the EDTA titration method (elemental calcium was determined, and then Ca was converted to CaO).

Considering that the only Fe-bearing detected minerals are calcium ferrite (sebrodoskite) and wustite, and the fact that wustite present low solubility and it is a minor phase, iron should be transferred mainly from sebrodoskite. A further analysis with SEM (scanning electron microscopy) with samples submitted to different pH values for different periods would support these conclusions.

Dissolution rates of highly soluble minerals tend to be relatively fast; it means there is high probability of being transport-limited for larnite and lime (minor phase for the investigated sample) instead of being interface limited, as occurs for low solubility minerals¹⁹. Available data for minerals occurring in slags systems (solubility/thermodynamic/sorption) are often incomplete or missing. This is a problem also for studies with simulation of leaching behavior of slags using geochemical modelling¹⁸.

3.5. Separation of Fe and Ca from the liquor

When using a high acid concentration, for example HCl 5 M, 87% of the iron and 91% of the CaO are transferred to solution. Since iron and calcium are in considerable extent dissolved in the supernatant, the separation in this condition can be achieved by selective precipitation. A 25 mL volume of solution resulting from leaching (supernatant) was dispensed in a 100 mL beaker. Subsequently, NaOH 1.0 mol.L⁻¹ was drip until the solution reached pH close to 8. The precipitation range of iron hydroxide (III) occurs from pH 3 to pH 7. One can observe the formation of a reddish-brown precipitate, characteristic of Fe(OH)₃. The precipitate was filtered and separated for later identification and the supernatant solution was separated for precipitation of the remaining Ca²⁺ ions.

The filtered solution containing Ca²⁺ ions with pH around 8, was separated into another beaker of 100 mL, and subsequently it was added sodium carbonate until total precipitation. It can be observed the formation of a white precipitate. Both precipitates were calcined at 900°C for one hour, cooled and subsequently sent for identification using XRD; Figure 6 shows their diffractograms. The iron precipitate is Fe₂O₃. The calcium precipitate presents three distinct solid phases: CaO, CaCO₃, and Ca(OH)₂, since calcination time and temperature were not enough for the complete conversion to CaO.

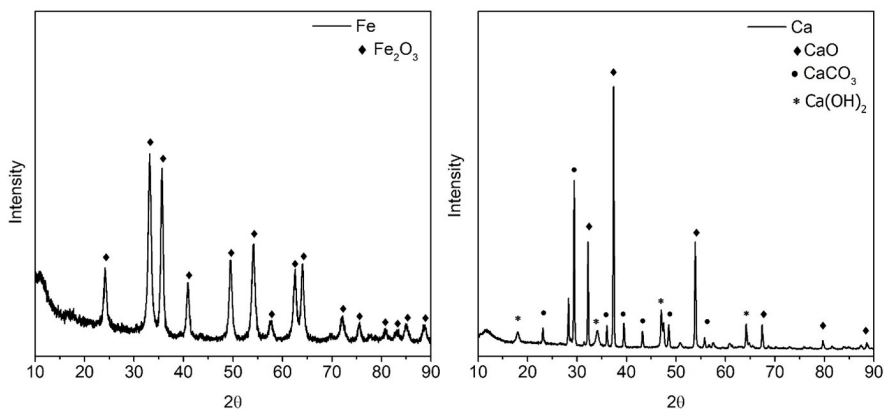


Figure 6. XRD diffractogram for the calcined precipitates.

4. Conclusions

For the BSSF steelmaking slag sample analyzed in the present work, the following conclusions can be drawn:

- A possibility to increase concentration of iron of the slag is to leach it with a 2 M HCl solution; in this condition, 68.3% of the CaO is extracted from the slag to the aqueous solution, and Fe content of the slag increases from 27.4 to 37.1 wt%.
- Another possibility is to use HCl 5 M; in this condition, 86.8% of the Fe and 90.7% of the CaO are transferred to the liquid solution. Separation of iron and calcium can be achieved by selective precipitation: firstly, adding ammonia to precipitate $\text{Fe}(\text{OH})_3$, and then calcinating it to produce Fe_2O_3 (~200 °C); in the following, adding sodium carbonate to precipitate CaCO_3 , which in turn is calcinated to produce CaO (~1000 °C). In this way, Fe_2O_3 and CaO could be produced and separated.
- Despite of the extremely high cooling rate which is applied to BSSF slags, the detected crystalline phases are also typical phases for BOF slags that suffered the usual semi-rapid cooling: $\beta\text{-Ca}_2\text{SiO}_4$, $\alpha\text{-Ca}_2\text{SiO}_4$, merwinite, sebrodoskite (major phases); and hatrurite, wuestite, lime (minor phases).

5. Acknowledgements

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