



Characterization and activation of Basic Oxygen Furnace slag

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ABSTRACT

BOF slags are by-products of steel conversion. They are a valuable mineral resource for the construction but have chemical properties that directly control their stability or any hydraulic activity. This paper provides a complete characterization and quantification of BOF slag phases in order to better understand their potential hydraulic activity. The acceleration of the hydration by chemical admixtures is also presented. The BOF slag variability is investigated on several samples with different origins and ages.

BOF slag with different origins mainly contain 38–52% C_2S , 20–30% C_2F , 1–7% $Ca(OH)_2$, 11–13% $Fe_{1-x}O$, 2–8% CaO . Calcium silicate is present in β - C_2S form which is the active polymorph present in clinker. BOF slags have poor hydraulic activity at early ages which can be enhanced by calcium chloride addition.

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1. Introduction

The blast furnace slag, a co-product of the pig iron production, has been widely investigated and used for many decades. Granulated or crystallized, it has found much application in construction, essentially as raw components for cement production or as standardized cement component for the granulated state or as aggregate for the crystallized state.

Basic Oxygen Furnace slags are byproducts of the conversion of pig iron to steel in a Basic Oxygen Furnace (BOF). The operating principle of pig iron refining is to blow oxygen in order to oxidize carbon and then decrease the carbon content. During this operation, lime is added in the converter in order to fix undesirable elements in slag and protect refractory lining. At the end of the conversion BOF slag is separated from steel by densimetric separation and poured into a casting pit. Depending on the grade of steel produced, 100–200 kg per ton of BOF slag are generated [1]. Thus, in France for example, 1.2 millions tons were produced in 2005.

The use of BOF slag is still limited and they are still undervalued. Some studies have been conducted on BOF slag use as addition in the raw mix or as hydraulic binder constituents [2,3]. A small amount is used by agriculture in amendment to neutralize soil acidity [4]. Road engineering used to consume an important quantity as aggregate since these materials have good mechanical properties and a low cost [5]. Unfortunately this use is restricted because of free lime content of BOF slag which causes uncontrolled volume expansions [6]. Several studies concerning aggregates

degradation, volumetric instability and BOF slag swelling, are underway to optimize their use [7–9].

With a chemical composition similar to that of Portland cement, the exploitation of BOF slag in some hydraulic binders can also be considered. They nevertheless have poor hydraulic properties, mainly because of their high content of iron oxide and the weak content of tricalcium silicate [10]. Indeed, the wüstite ($Fe_{1-x}O$), which is one of the major phases of BOF slag does not react with water. Furthermore, when materials are finely ground, calcium oxide they contain reacts with air moisture to form hydrated lime $Ca(OH)_2$. This last mineral reacts itself with the ambient CO_2 to form calcite $CaCO_3$. Thus the CaO content decreases over time. These phenomena may change or decrease the reactivity of the unstable materials.

BOF slag valorisation requires a thorough physical and chemical characterization to have a good awareness of their properties. The hydraulic activity of these materials and potential way to activate them should also be known. Most of the literature on this subject only relates to the qualitative characterization of BOF slag and the quantification of free lime content, due to the problem of expansion [11].

Investigations presented in this study are more complete because they relate to the identification and quantification of all mineral phases present in BOF slag (C_2S , C_2F , etc.). They were performed on a variety of products with different origins and ages (fresh and weathered productions). The first part of this paper relates in detail the methodology of proposed analysis. The second part regards the chemical activation tests in order to improve the reactivity of BOF slag seeking the best hydraulic properties of these materials.

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2. Chemical characterization

2.1. Materials

The BOF slag studied are coming from different industrial plants and melts. After cooling and treatment they are reclaimed as 0/100 mm granulates with a minimum fine fraction.

BS1 samples are a mixture of several melts from the same origin but with different ages:

- BS1F: Fresh production
- BS1W: Weathered production

BS2 samples are fresh products, from a second industrial plant and separated melts which differ by the free lime content. This parameter depends on the grade of steel produced:

- BS2H: High lime content
- BS2L: Low lime content

Representative sampling of large granulates were performed for analysis. They were crushed, ground and sieved to 0–125 μm size.

2.2. Analytics techniques

Particle size distribution was obtained by the laser particle size technique.

The specific area was identified by Blaine Test according to the standard NF EN 196-6. Absolute density was measured by a hydrostatic weighing in a non reactive liquid.

The X-Ray Fluorescence (XRF) technique is an emission of secondary X-rays characteristic of the atomic elements in the sample. It was used to determine the chemical composition of powders, expressed as a percentage of oxides. The loss of ignition is taken into account in the total composition. Analyses were performed on a Bruker equipment.

The crystalline phases were identified using an Xpert pro X-ray diffractometer using a copper anticathode $K\alpha$ ($\lambda = 1.54506 \text{ \AA}$). The X-ray diffraction patterns obtained have been exploited by the diffraction software Plus-EVA®.

The plan view of different phases in the slag grain was given by an electron probe microanalyzer CAMECA SX100 coupled to EDS analyses in order to identify the chemical composition of minerals and impurities that may present. Powders analyzed were embedded and dryly polished.

The free lime content is found by acid-base titration whose principle is to extract calcium with glucose and to titrate the filtrate with a hydrochloric acid 0.1 N. This method must be complemented by TGA-DTA (Thermo-Gravimetric Analysis coupled with Differential Thermal Analysis), in order to find out the hydroxide and oxide calcium content separately. This last method allowed us also to determine the carbonate (CaCO_3) content. The principle is to follow up and to quantify mass changes due to dehydration and decarbonation of samples as a function of temperature. The tests were performed on a thermobalance Setaram TG92®.

The main characteristic of steel slag is the high iron oxide content, which exists in both the di- and trivalent states. This content is found by redox titrations of Fe^{III} with a potassium dichromate solution $\text{K}_2\text{Cr}_2\text{O}_7$ in the presence of sodium diphenylamine sulfonate indicator. The Fe^{III} is reduced to Fe^{II} by stannous chloride SnCl_2 in order to titrate both di and trivalent states contents.

The calcium silicate content is found by SAM extraction with methanolic solution of salicylic acid [12–14]. Calcium phases are dissolved by salicylic acid and the C_2S is calculated by difference with the already known lime content.

3. Results and discussions

3.1. Physical properties

The absolute density measured on these BOF slag equals 3700 kg/m^3 on average. This value is relatively high, due to the high iron content. Note here that, due the presence of iron oxide, a longer grinding time is needed comparing to the clinker one. The density is measured on a freshly ground powder; samples aging causes hydration and carbonation of free lime and, consequently, a decrease of density. This parameter may reach an average of 3300 kg/m^3 after 6 months aging.

The particles size distribution is given in Fig. 1. Despite the same grinding conditions for both samples, BS1 presents a larger fines amount.

The specific surface area of 0–0.125 mm fresh powder equals to almost $3800 \text{ cm}^2/\text{g}$.

3.2. Minerals phases' identification

The chemical composition of BOF slag expressed as a percentage of oxides obtained by XRF analysis is given in Table 1. For comparison, results obtained on a cement clinker (K) and a granulated blast furnace slag (GBFS) are also reported in Table 1. The major differences between these three materials are in the contents of alumina, silicon and iron oxides. SiO_2 and Al_2O_3 contents of BOF slag are respectively six times and three times weaker than those of GBFS, and Fe_2O_3 content is much higher.

The chemical composition is rather similar for the four samples of BOF slag despite their differences. Calcium, silicon and iron oxides alone account more than 80% of BOF slag elements. Weathered samples have a higher Loss Of Ignition (LOI) due to carbonation.

If we disregard the high iron content, we note that BOF Slag composition is close to the clinker one. However the iron oxide

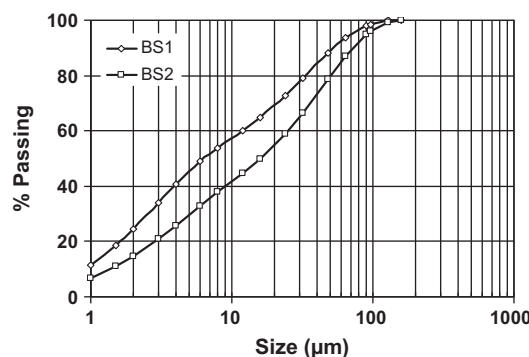


Fig. 1. Particle size distribution of BS1 (fresh production) and BS2 (high lime content production).

Table 1
Chemical composition of BOF slag investigated measured by X-ray Fluorescence.

wt.%	BS1F	BS1W	BS2L	BS2H	GBFS	K
LOI	1.1	8.3	0.0	0.0	0	0.2
MgO	4.5	5.5	6.0	7.5	7.5	4.3
Al_2O_3	1.9	1.9	1.7	2.1	12.2	4.7
SiO_2	10.8	8.6	13.1	11.3	35.1	20.8
P_2O_5	1.4	1.4	2.4	2.0	0	0.2
SO_3	0.4	0.4	0.4	1.2	1.7	1.3
CaO	45.0	40.1	41.7	42.9	41.1	63.6
TiO_2	0.5	0.5	0.9	0.7	0.6	0.3
MnO	2.6	2.0	4.1	3.7	0.4	0.1
Fe_2O_3	32.0	30.8	29.3	28.3	0.4	3

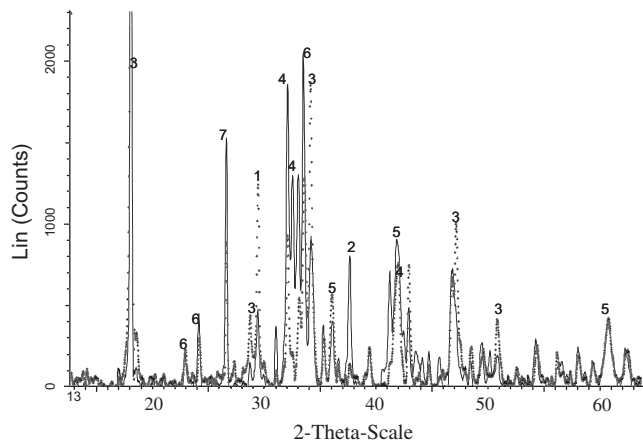


Fig. 2. X-ray diffraction pattern of BS1; solid line BS1F, dotted line BS1W 1-CaCO₃, 2-CaO, 3-Ca(OH)₂, 4-C₂S, 5-FeO, 6-C₂F, 7-SiO₂.

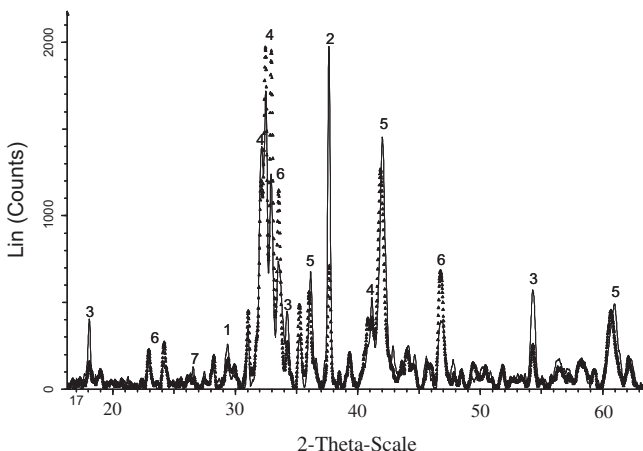


Fig. 3. X-ray diffraction pattern of BS2; solid line BS2H, dotted line BS2L.

Fe₂O₃ minimizes the hydraulic activity of these materials since iron phases are inert with water contact.

The X-ray diffraction pattern of BS1 and BS2 slag are given in Figs. 2 and 3 respectively.

The main minerals phases are: dicalcium silicate C₂S, dicalcium ferrite C₂F, calcium hydroxide Ca(OH)₂, calcium oxide CaO and wüstite Fe_(1-x)O. Some minor phases as calcite CaCO₃, periclase MgO and quartz SiO₂ were also identified in particular samples.

In the same way Fe₃O₄ was detected in small amounts in some samples. Its presence explains the magnetic behavior of BOF slag and also the difficulty of crushing that represent such materials.

For all investigated samples, X-ray diffraction (XRD) indicates that the major mineral in BOF slag is the polymorph β-C₂S. This form of mineral is the active one which is present in cement. Unlike others slag such electric furnace slag, BOF slag contain in most cases the β form [15]. Usually β-C₂S changes to γ-silicate below 675 °C during cooling [16]. However this disintegration is not found in the presence of high Fe_{1-x}O content and presence of impurities which inhibit the reaction [15]. The peaks of Fe_{1-x}O are slightly offset from the pure phase which indicates that this mineral is present in a solid solution.

Fig. 2 presents the X-ray diffraction pattern of fresh and weathered BS1 samples. The main differences are observed in calcic phases:

- The calcite content is higher for BS1W slag due to the hydroxide calcium carbonation during weathering. This result is in agreement with the high LOI (Loss Of Ignition) value obtained by XRF (Table 1)
- The hydroxide calcium content is higher for the BS1W. It is produced by the hydration of calcium oxide and calcium silicate. Therefore, the peaks of these two last phases are smaller.

The X-ray diffraction patterns of BS2 samples are presented in Fig. 3. As expected, BS2H contains more calcium hydroxide and oxide. The amount of calcite is relatively low since these slag are considered fresh ones.

Most authors mention the presence of C₃S and C₄AF [10,1]. These two phases do not appear in the diffraction pattern because of peaks overlapping with C₂S and C₂F ones respectively. However C₃S phase can stand out against C₂S with the presence of a peak at

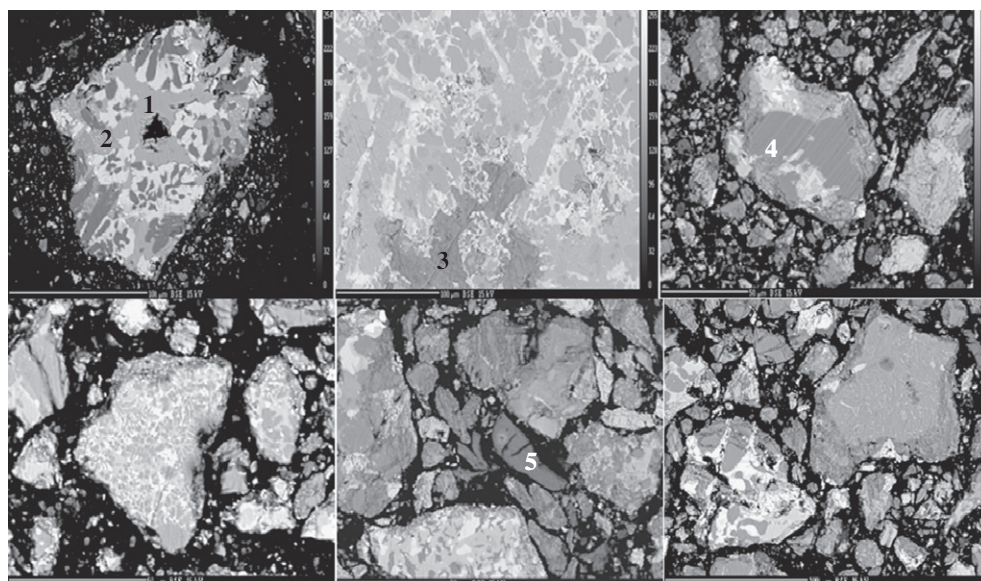


Fig. 4. Plan view of BOF slag with microprobe analysis 1-C₂F, 2-SS (CaO/FeO), 3-C₂S Hydrated, 4-C₂F, 5-Ca(OH)₂.

Table 2(a): Free lime contents by chemical titration and (b): $\text{Ca}(\text{OH})_2$, CaO and CaCO_3 contents by TGA analysis.

(a)			
Free lime content (wt.%)			
BS1F			6.8
BS1W			7.8
BS2L			5.5
BS2H			10.3
(b)			
TGA (wt.%)	$\text{Ca}(\text{OH})_2$	CaO	CaCO_3
BS1F	6.3	2.0	2.9
BS1W	7.3	2.3	4.0
BS2L	1.1	4.6	1.9
BS2H	2.9	8.1	2.6

1.76 Å [1]. This one is not visible in the given X-ray diffraction pattern probably due to the low content of this phase. C_3S is particularly present in basic BOF slag with a CaO/SiO_2 ratio higher than 2.7 [6].

We note that, unlike the Granulated Blast Furnace Slag, no amorphous phase was detected by this technique. In fact, the narrow peaks show that the identified phases are crystallized.

To evaluate the specific chemical compositions of phases identified above, the samples were characterized by the micro probe technique. A plan view, given in Fig. 4, shows the structural heterogeneity of BOF slag which is common for all samples whatever their origins. Phases are distinguished by the contrast of the gray color. Indeed, for all grains, phases are intimately mixed, as it can be seen in the images. Only few independent phases have been observed but they are never pure.

The calcium silicate phases are homogeneous with “Ca/Si” ratio varying from 1.8 to 3.5 (average 2.4). This value proves the presence of C_3S , but its content is too low to be identified by XRD. The only detected impurity is phosphorus. For BOF slag rich in P_2O_5 and C_2S can be found in solid solutions with the C_3P [4]. The presence of this impurity confirms the stabilization of silicate in the β form.

The calcium ferrite phases are also fairly homogeneous and contain Al in variable content. Locally, the Fe/Ca ratio is close to that of C_4AF phase. But this last phase can not be identified by XRD because of peaks overlapping with those of C_2F [1].

The solid solutions (Fe, Mg, Mn and Ca) O, with varying percentages of these elements are common mineral phases in the BOF slag [2]. Two kinds of solid solutions are generally mentioned in the literature: a wüstite phase (Fe, Mn and Mg) O and a phase dominated by CaO [17].

3.3. Phases quantification

The free lime content ($\text{CaO} + \text{Ca}(\text{OH})_2$) expressed in equivalent CaO is determined by glucose extraction and filtrate's titration by hydrochloric acid. The free MgO content is also measured by this method, but will be neglected in our calculations since the MgO is trapped in solid solutions and is rarely free. The results are given in Table 2a. The free lime content in the sample BS2R is, as expected, higher than that for BS2P. The CaO content of BS1 has an intermediate value to those of the two BS2, which seems logical since BS1 is a mixture of different melts.

This chemical titration is completed by a thermogravimetric analysis in order to differentiate CaO and $\text{Ca}(\text{OH})_2$ contents. A typical TGA curve of BOF slag is presented in Fig. 5. In the temperature range 20–800 °C, two reactions associated with lime and accompanied with loss of mass, are distinguished:

– 450 °C: $\text{Ca}(\text{OH})_2$ dehydration



– 750–800 °C: CaCO_3 decarbonation



According to reactions 1 and 2, $\text{Ca}(\text{OH})_2$ and CaCO_3 contents can be calculated with mass balance. CaO content is deducted from the free lime content measured above. Results are given in Table 2b.

The results of FeII and FeIII chemical titrations are given in Table 3a. From those results and based on the fact that FeII is only present in the $\text{Fe}_{(1-x)}\text{O}$ phase and FeIII in the C_2F phase, these last two phases contents are calculated in Table 3b.

The last phase to quantify is calcium silicate. The method adopted is an extraction of calcic phases with a methanolic solution of salicylic acid. Powder extracted was analyzed with XRD. C_2S , $\text{Ca}(\text{OH})_2$ and CaO were dissolved by the acid and C_2F , FeO , CaCO_3 and SiO_2 remain in the powder. C_2S and SiO_2 contents can be calculated with mass balance of the lost and recovered mass

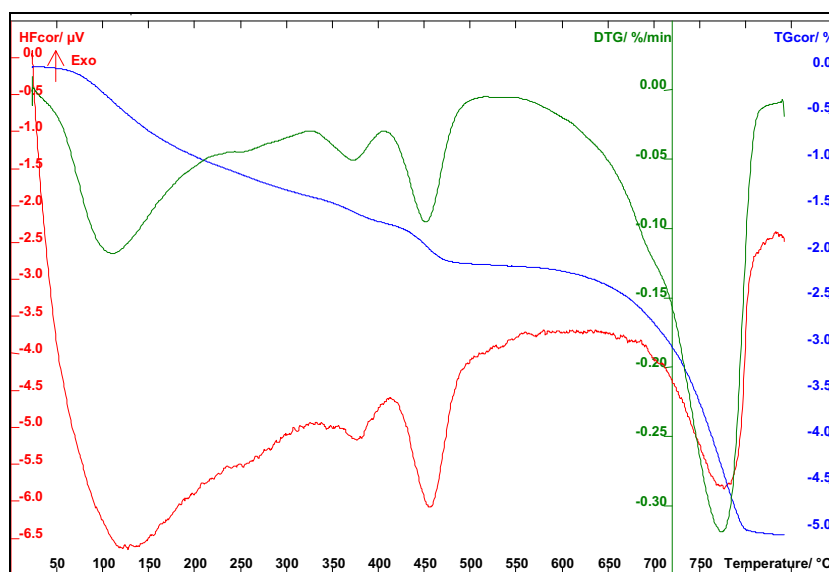


Fig. 5. Thermogravimetric curve of BOF slag.

Table 3

(a): Fe titration's results and (b): Iron phases contents.

(a) wt.%	Total iron	Fell
BS1F	21.8	9.3
BS1W	20.1	10.0
BS2L	17.3	8.9
BS2H	19.4	10.1
(b) wt.%	Fe _{1-x} O	C ₂ F
BS1F	11.9	30.3
BS1W	12.9	24.5
BS2L	11.5	20.2
BS2H	13.1	22.5

Table 4

Mineralogical composition of BOF slag.

wt.%	C ₂ S	C ₂ F	FeO	Ca (OH) ₂	CaO	CaCO ₃	SiO ₂
BS1F	41.8	30.3	11.9	6.3	2.0	2.9	4.7
BS1W	38.1	24.5	12.9	7.3	2.3	4.0	10.9
BS2L	52.0	20.2	11.5	1.1	4.7	1.9	8.5
BS2H	51.1	22.5	13.1	2.9	8.1	2.7	0

respectively (Table 4). Magnetite Fe₃O₄ has not been taken into account in this calculation; its content may be associated to the SiO₂ one hence the variability of this parameter.

3.4. Summary

Phase contents of the four analyzed samples are given in Table 4. The chemical compositions of different BOF slag are quite close (Table 1), despite the different origins and ages of the samples.

Main differences remain on the mineral phases contents and in particular the C₂S content and the free lime type phases:

- C₂S contents depend on the origin of sample (an average of 40% for BS1 and 51.5% for BS2)
- free lime contents are substantially equal for both BS1. These values are an average of those of BS2H and BS2L. However, only BS2R has a high content of CaO, while for the other samples it's the Ca(OH)₂ content which is most important particularly for the old production (BS1V). It is also the sample with the highest calcium carbonate content (4%). Clearly, the age product has an influence on the nature of the calcic phases.

The C₂S and C₂F contents decrease in time, showing the reactivity and the hydration of these two phases. This observation confirms the evolving nature of such materials which can influence their hydraulic behavior. The difference of the two slag's fineness observed by laser granulometry can also be explained by the variability of the phases contents and in particular the C₂S one [18].

The sum of C₂F and Fe_{1-x}O contents confirms the results obtained by XRF (Fe₂O₃, Table 1). BS1F, which has the highest Fe₂O₃ content, has also the highest C₂F content.

The SiO₂ contents are variable. But these values are deducted from the other mineral phases. Therefore, they probably include iron metal and magnetite which were not quantified in this study (low levels a priori).

The MgO phase was also neglected. Indeed, this mineral is often trapped in solid solutions and is rarely found in free form.

4. Chemical activation of BOF slag

β-C₂S phase, the main phase in the BOF slag, is known by its latent hydraulicity. The activation of this phase, if possible, is an interesting solution for the early reactivity improvement of these materials.

In this context, several admixtures commonly used in civil engineering have been tested.

4.1. Raw materials

Only BS1F is investigated in this part of study.

The various accelerators admixtures used are:

- calcium chloride dihydrate CaCl₂·2H₂O 99 + %
- sodium chloride NaCl 99.5 + %

According to literature, NaCl and CaCl₂ accelerate the setting and increase the resistance at early ages [19]. The comparison between these two products has helped to show up the contribution of the accompanying cation, Ca²⁺ and Na⁺.

- sodium metasilicate nonahydrate Na₂SiO₃·9H₂O 44–47% is an admixture conventionally used in a proportion of 6 to 15% in the hydraulic binder. At high doses it may cause an important decrease of strength.

Special hydraulic binder – high alumina cement – quicklime and a commercial admixture accelerator of setting for concrete have been used to complete this study.

4.2. Tests performed

The steel slag pastes were prepared by mixing steel slag with water. The water/steel slag ratio of paste was 0.25. With this ratio BOF slag pastes have normal consistency according to the standard NF EN 196 cement. The activators, except the lime and alumina cement, were added to the batching water for each test with proportions given in Table 5.

Pastes were prepared with a standardized mixer following cycle of the standard NF EN 196, then poured into cylindrical specimens 34 × 34 mm and kept at 40 °C in a 96% humid atmosphere (3 specimens per admixture). The compressive strengths tests were performed at 7 days after polishing bearing section. The broken samples were transferred into acetone to stop hydration and then were analyzed by X-ray diffraction in order to identify the phases formed. These results were compared to a control with no chemical additives.

4.3. Results and discussion

4.3.1. Compressive strength

The 7-day strengths of hydrated BOF slag with different admixtures are given in Fig. 6. They are presented related to a control sample performance which is equal on average to 9 MPa. In detail:

Table 5

Admixtures' proportions of BOF slag pastes.

Admixtures	wt.%
CaCl ₂	2
NaCl	2
Na ₂ SiO ₃	10
CaOv	2
Alumina cement	2
Commercial admixture	1

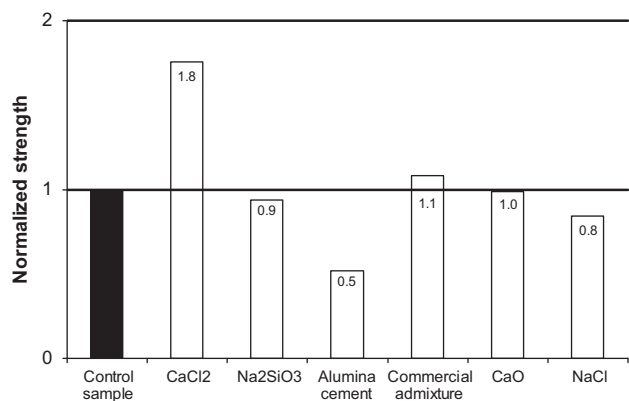


Fig. 6. Normalized 7-day compressive strength of activated BOF slag pastes.

- calcium chloride almost doubles the mechanical properties of the control samples
- commercial admixture has a marginal effect
- lime has no effect
- Na₂SiO₃ has a slightly negative effect on strength. However it has improved paste' consistency which was more fluid comparing to other pastes. This product is often used as water-reducing admixture [19].
- NaCl has a stronger negative effect which confirms that the di and trivalent cations are more effective than the monovalent on accelerating paste' hardening. [20]. This observation will be confirmed later by XRD analysis
- high alumina cement could not counteract the aluminium oxide lack in BOF slag. Contrariwise it apparently has caused paste destruction and consequently decreased mechanical performances since no chemical reaction have been observed by XRD as it will be shown in the next paragraph.

4.3.2. Chemical characterization of hydrate products

Chemical characterization of control paste hydrated at 40 °C for 7 days shows an early consumption of the C₂S phase and simultaneously emergence of CSH type phase containing aluminium and iron with chemical formula Ca₃AlFe(SiO₄)(OH)₈. Corresponding X-ray diffraction peaks are distinguished at 2θ = 20° and 29°. The second product of hydration identified is the epidote with chemical formula Ca₂FeAl₂Si₃O₁₂(OH) This phase diffracts at 2θ = 11°.

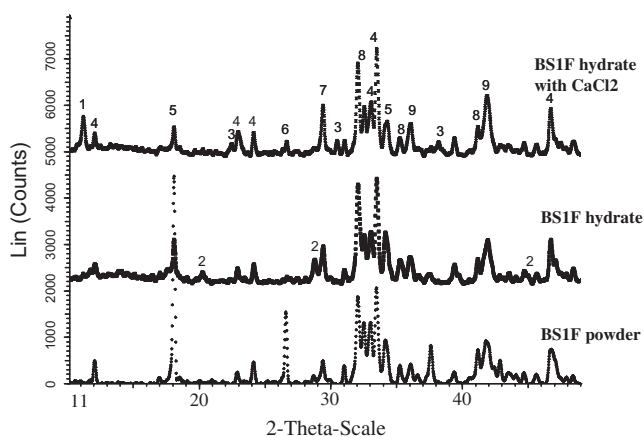


Fig. 7. X ray patterns of raw material, BOF slag paste and CaCl₂ activated paste 1-Epidote Ca₂FeAl₂Si₃O₁₂(OH) 2-Calcium Aluminium Iron Silicate Hydroxide Ca₃AlFe(SiO₄)(OH)₈; 3-Hydrate product with CaCl₂; 4-C₂F; 5-Ca(OH)₂; 6-SiO₂; 7-CaCO₃; 8-β-C₂S; 9-FeO.

Fig. 7 shows the X-ray superposed patterns of BOF slag initial powder, hydrated paste without admixture and hydrated paste in the presence of CaCl₂ as additive. We note that:

- CaCl₂ enhances the formation of the epidote phase and forms a new hydrate. Unfortunately this last one could not be identified by XRD. Ca₃AlFe(SiO₄)(OH)₈ phase no longer appears
- NaCl and Na₂SiO₃ enhance as well the epidote phase formation but no other hydrated products were identified by XRD
- CaO has clearly no effect because the diffraction pattern of the activated paste with this compound is similar to the control sample one.
- High alumina cement and commercial admixture developed no new hydrated phase

4.3.3. Results interpretations

BOF slag activation study showed that only CaCl₂ is really effective to improve mechanical properties at early ages. Some previous research has shown that Ca(OH)₂ and CaCl₂ form a complex which could be responsible for the acceleration of the reactions [21]. As a consequence Ca(OH)₂ content is lower in the presence of CaCl₂ as observed by X-ray diffraction. This observation is significant in our case since BOF slag have high lime content. This confirms the finding that CaCl₂ accelerates the pozzolanic reaction of Ca(OH)₂ [22].

In general, cement phases hydration results on CSH gel formation. This gel is nanoporous, amorphous and is deposited on the non-hydrated particles surfaces. Crystals of Ca(OH)₂ are formed and grow in porous space of this gel. In presence of CaCl₂ these phenomena are modified: (i) the period of nucleation-germination is decreased [23]; (ii) the CSH gel has non homogeneous zones with high and low density; (iii) ions from unhydrated phases diffuse easily through the initial layer of CSH [24].

5. Conclusion

An investigation on BOF slag characterization and improvement of its hydraulicity was presented in this paper.

The different techniques used in this study represent a rational approach for a comprehensive BOF slag characterization. Four samples with different origins and ages were investigated. Results showed that chemical compositions are ultimately close, which suppose that these products are quite similar, whatever their origins. The most significant difference is the C₂S content which varies from almost 40–50%. This silicate is the main mineral and it is present in the β-C₂S form which is the active phase usually found in hydraulic binders. C₃S phase was detected by microprobe analysis but its content is too low to be detected by X-ray diffraction. The second mineral present is the C₂F. This phase contains locally alumina; detected by microprobe. This association is close to C₄AF phase known by its hydraulic activity. This last mineral can not be identified by XRD because of peaks overlapping with the C₂F phase ones.

The accelerating admixtures investigated in this study are: NaCl, CaCl₂, CaO and Na₂SiO₃, high alumina cement and commercial admixture. Strength properties of BOF slag activated pastes were compared to a hydrated control paste. All of them were kept at 40 °C during 7 days. Hydrated products were characterized by X-ray diffraction. Results showed that CaCl₂ is an effective admixture for BOF slag activation. It improves mechanical properties and accelerates phases' hydration.

These investigations confirm that BOF slag has a latent reactivity that can be accelerated by CaCl₂. These properties have been obtained through the combination of analytical techniques

presented in the context of this work. This study is a step towards the valorisation of BOF slag as a building material.

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