

# Valorization of steel slag by a combined carbonation and granulation treatment

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## Abstract

This work reports the results of a combined accelerated carbonation and wet granulation treatment applied to Basic Oxygen Furnace (BOF) steel slag after crushing for metal recovery with the aim of producing secondary aggregates for civil engineering applications and of storing CO<sub>2</sub> in a solid and thermodynamically stable form. The tests were carried out in a disc granulator equipped with a lid and CO<sub>2</sub> feeding system. In each test, humidified slag (liquid/solid ratio of 0.12 l/kg) was treated for reaction times varying between 30 and 120 minutes under either atmospheric air or 100% CO<sub>2</sub>. Results indicated that, under both conditions, the particle size of the treatment product increased progressively with the reaction time; specifically, the d<sub>50</sub> values obtained for the products of the combined granulation and carbonation treatment increased from 0.4 mm to 4 mm after 30 min and to 10 mm after 120 min. Significant CO<sub>2</sub> uptake values (between 120 and 144 g CO<sub>2</sub>/kg) were measured even after short reaction times for granules with diameters below 10 mm and for coarser particle size fractions after reaction times of 60 minutes. The density, mineralogical composition, leaching behavior and mechanical resistance of both granulated and granulated-carbonated granules were also investigated.

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## Introduction

Significant amounts of alkaline solid residues are generated during steel manufacturing (around 10-15% by weight of the produced steel, depending on the characteristics of the manufacturing process). Among these, BOF slag, a by-product of iron conversion into steel in a Basic Oxygen Furnace, is one of the most abundant by-products (100-200 kg per ton of steel<sup>1</sup>) without a well-established reuse application. This material is generally ground for metal recovery and then landfilled or reused limitedly for low-end applications, because some of its properties (i.e.: high free lime content, fine particle size and excessive mobility of specific elements) have shown to affect its technical performance and/or environmental behavior, thus hindering its recycling potential. Several studies have indicated that the leaching behavior and mechanical properties of alkaline materials such as steelmaking slag may be affected by accelerated carbonation treatment, which also allows to store CO<sub>2</sub> in a solid and thermodynamically stable form<sup>2-5</sup>. In particular, specific types of steel slag, including BOF, treated by accelerated

carbonation in a static reactor under wet conditions, i.e. applying a liquid to solid (L/S) ratio lower than 1 l/kg, have shown a significant reactivity with CO<sub>2</sub>, even under mild operating conditions (T=20-50 °C; p=1-10 bar)<sup>6</sup>. Although this treatment has shown to affect some of the properties of the slag (e.g. mineralogy and leaching of major and trace compounds), it does not exert an influence on important properties for slag reuse as an aggregate in civil works, such as particle size distribution. In this regard, granulation treatment represents an effective particle size enlargement process, which is achieved by contacting a mixture of a dry powder and a liquid binder in a dynamic device, such as a rotary drum<sup>7,8</sup>. Different studies<sup>9,10</sup> have focused on the application of disc granulation to materials such as fly ash and bottom ash from waste incineration and contaminated soils, with the aim of producing aggregates to use as filling material or for concrete manufacturing. Recently, the combination of granulation with accelerated carbonation has been applied as a treatment to improve the leaching behavior of residues such as waste incineration ash, produce aggregates and reduce CO<sub>2</sub> point-source emissions<sup>11</sup>.

The aim of this work was to evaluate the applicability of combined granulation and carbonation as a valorization technique for BOF slag and to compare the effects of this process with those exerted by granulation under atmospheric air, so to analyze the influence of carbonation on particle aggregation and on the properties of the products. Specifically, the paper reports and discusses the results of granulation and granulation-carbonation experiments carried out in a disc granulator at different reaction times in terms of the effects exerted on the physical properties, CO<sub>2</sub> uptake, mineralogy, leaching and mechanical behavior of the obtained granules.

## Materials and methods

### Materials

The slag used in this study is a by-product of a steel making plant employing the basic oxygen furnace (BOF) process. The steel slag sample was collected from a BOF slag storage site after grinding for iron and steel recovery. The particle size of the untreated material was lower than 2 mm with a d<sub>50</sub> value of 0.45 mm. The main chemical constituents of the BOF slag (see Table 1), as determined by alkaline fusion and ICP-OES analysis of the obtained solutions were: Si (35.7 %), Ca (24 %), Fe (19.6 %), Mg (4.7 %) and Mn (2.24%), with similar concentration values to those reported in other studies regarding this type of steelmaking slag<sup>12,13</sup>.

**Table 1** Elemental composition (g/kg on a dry weight basis) of untreated BOF slag

Element	Concentration (g/kg)	Element	Concentration (g/kg)
Al	1.51	Mg	46.58
Ba	0.06	Mn	22.42
Be	<0.0003	Mo	0.006
Ca	239.264	Na	3.05
Cd	0.0035	Ni	0.019

<b>Cr</b>	1.673	<b>Pb</b>	0.03
<b>Cu</b>	0.27	<b>Si</b>	356.91
<b>Fe</b>	196.03	<b>V</b>	0.32
<b>K</b>	1.281	<b>Zn</b>	0.1

## Methods

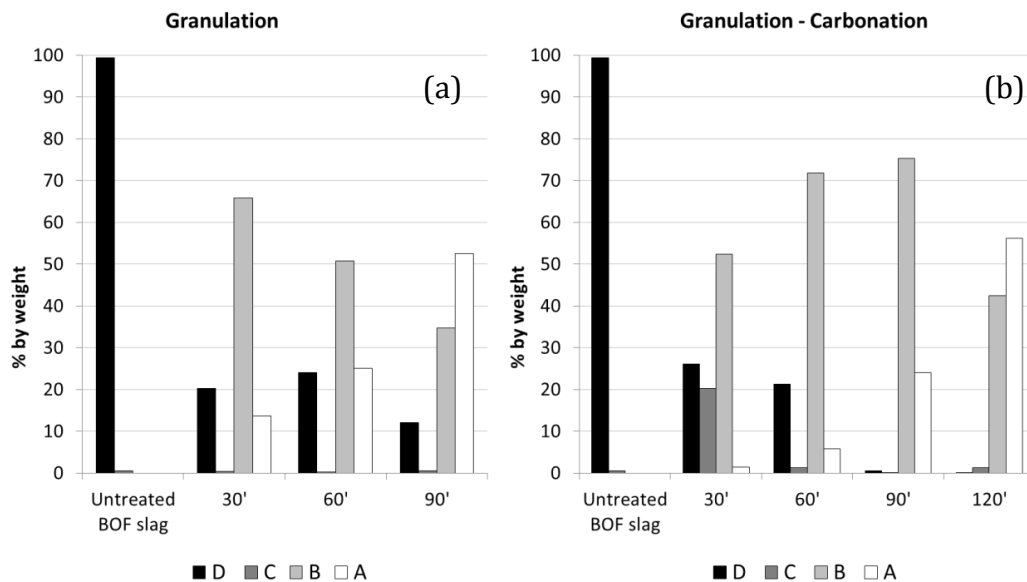
Both the granulation and granulation-carbonation experiments were performed using a disc granulator with a diameter of 0.3 m and a height of 0.23 m, equipped with a blade, and operated at 24 rpm with a tilt of 50° during all experiments. Granulation experiments were performed leaving the reactor under atmospheric air, whereas for the granulation-carbonation experiments, a custom-made Perspex lid with a CO<sub>2</sub> feeding system was placed over the reactor. For both types of experiments, air-dried slag (approximately 500 g) and deionized water were premixed in a plastic bag, at the liquid to solid ratio that resulted optimal for the granulation of this material at the tested conditions, i.e. 0.12 l/kg. In order to obtain a homogeneous starting particle size distribution for all the experiments, the mixture was then pushed through a 2 mm sieve, following the procedure widely adopted in other studies on granulation<sup>8,14</sup>. Experiments were performed at different reaction times (30, 60, 90 and 120 minutes) and the product of each test, after curing under atmospheric conditions for 7 days, was analysed to determine its particle size distribution applying the ASTM D422 standard procedure. Each material was then divided by sieving into the following size fractions: Class A (d>9.53 mm); Class B (4<d<9.53 mm); Class C (2.36<d<4 mm); Class D (d<2.36 mm). Subsequently, each sub-fraction as well as the untreated BOF slag was characterized in terms of its density, carbonate content, mineralogy and leaching behaviour. The density of the samples was assessed by the UNI EN 1097-6 procedure. The carbonate content was evaluated by inorganic carbon analysis using a Shimadzu TOC VCPH analyser equipped with a SSM-5000A solid sampler. The mineralogical composition was determined by powder XRD analysis with Cu K $\alpha$  radiation using a Philips Expert Pro diffractometer equipped with a copper tube operated at 40 kV and 40 mA. The leaching behaviour was assessed by following the EN 12457-2 standard compliance test that involves the grinding of samples with a grain size above 4 mm. Eluate concentrations of major compounds and trace metals were analysed with an Agilent 710-ES ICP-OES. In addition, the leaching behaviour of the unground granules obtained from each treatment was also assessed by applying a L/S ratio of 10 l/kg and a contact time of 24 hours, followed by ICP-OES analysis of eluate concentrations. Moreover, the mechanical strength of the granules was evaluated by performing the Aggregate Crushing Value (ACV) test, applying the British Standard BS 812-110/112.

## Results and discussion

### Effects of the tested treatments on the physical properties of the materials

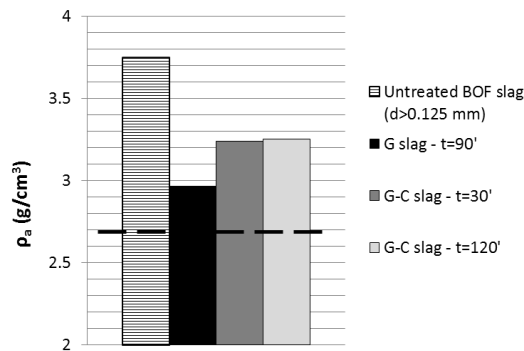
The average diameter of the granules obtained from granulation and granulation-carbonation experiments resulted between 4 mm and 10 mm and showed to increase with the reaction time. The d<sub>50</sub> value of the treatment product was an order of magnitude greater than that of the initial BOF slag (0.4 mm). In Figure 1 the weight

distribution in the 4 classes of the untreated slag is compared with that of the treated material obtained after different reaction times. For the granulation experiments (Figure 1a), it can be seen that the content of the finest fraction (class D) decreased from almost 100% in the untreated slag to about 10% in samples obtained after 90 minutes. Granules with a size fraction between 2.36 and 4 mm (Class C) were not found after any of the tested reaction times, while a significant decrease of class B granules and a corresponding increase of class A particles was observed for an increase of the treatment time, highlighting the ongoing agglomeration process. The effect of the granulation-carbonation treatment on granule size distribution is reported in Figure 1b. The content of classes D and C showed to decrease significantly after 90 minutes, while the amount of class B granules, differently from the granulation tests, showed to increase over time up to a reaction time of 90 minutes. As underlined by the trend of the content of the coarser fraction (class A), the kinetics of particle growth in the presence of CO<sub>2</sub> was slower than that resulting under atmospheric air.



**Figure 1** Comparison of the weight distribution of the untreated slag with (a) granulated slag and (b) granulated-carbonated slag at different reaction times. Class A ( $d > 9.53 \text{ mm}$ ); Class B ( $4 < d < 9.53 \text{ mm}$ ); Class C ( $2.36 < d < 4 \text{ mm}$ ); Class D ( $d < 2.36 \text{ mm}$ )

The apparent particle density of both the untreated slag ( $d > 0.125 \text{ mm}$ ) and samples obtained after granulation and granulation-carbonation was evaluated and compared with that of natural gravel (see Figure 2). The value found for the untreated slag was quite high ( $3.75 \text{ g/cm}^3$ ) if compared to that of natural gravel ( $2.7 \text{ g/cm}^3$ ), probably in relation to the significant iron content of the slag<sup>13</sup>. The density of the material obtained after a granulation treatment of 90 minutes ( $2.9 \text{ g/cm}^3$ ) was lower than that of the untreated slag, probably owing to the porosity of the granules formed during the process. An increase in density was observed for granules obtained after the combined treatment ( $3.2 \text{ g/cm}^3$ ) that can be related to the carbonation reaction occurring in the material, as discussed below. In addition, it can be noted that the apparent particle density did not seem to be affected by the reaction time since the values found after 30 and 120 minutes were very similar.



**Figure 2** Apparent particle density results for: untreated BOF slag; granulated slag after 90 minutes (G granules – t=90’); granulated-carbonated slag after 30 (G-C granules – t=30’) and 120 minutes (G-C granules – t=120’); natural gravel (dotted line).

### Effects of the tested treatments on the CO<sub>2</sub> uptake of the materials

The main results of the tested treatments in terms of the reaction of the alkaline phases of the slag with the CO<sub>2</sub> contained in the gas phase were assessed by measuring the Inorganic Carbon (IC) content of the material before and after the granulation and granulation-carbonation treatments. The results reported in Table 2 indicate that the IC content of the products of both types of treatment was significantly higher than that of the untreated slag, implying that carbonation occurred also when the material was subjected to the granulation treatment under atmospheric air. However, the IC content of the granulated-carbonated materials (3.5-4%) was almost double of that obtained from the granulation tests (1.7-2%).

On the basis of the IC content of each treated sample, its CO<sub>2</sub> uptake was evaluated by applying Eq. 1, in which the IC concentration of the untreated (CO<sub>2</sub> initial) and treated (CO<sub>2</sub> final) sample is expressed in terms of its CO<sub>2</sub> weight percent.

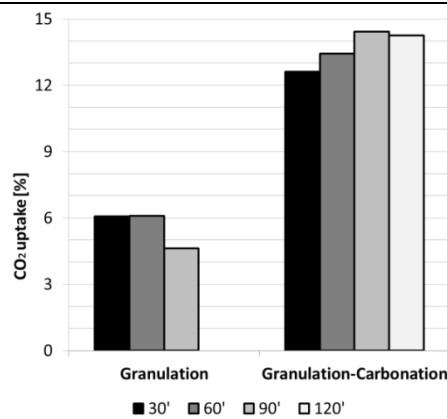
$$CO_{2 \text{ uptake}} = \frac{CO_{2 \text{ final}}(\%) - CO_{2 \text{ initial}}(\%)}{100 - CO_{2 \text{ final}}(\%)} \times 100 \quad (1)$$

The CO<sub>2</sub> uptake values measured for each type of treatment as a function of the reaction time are reported in Figure 3. For the granulation test, the maximum uptake value (6%) was obtained after short reaction times (30-60 minutes) and showed to decrease after a reaction time of 90 minutes. This behavior may be ascribed to the larger size of the obtained granules and hence lower specific surface available for the reaction. The CO<sub>2</sub> uptake of the granulated-carbonated granules was above 12% after a reaction time of 30 minutes and increased slightly to the maximum value of 14.4% after 90 minutes. It is worth noting that these results, achieved with a considerable amount of material (≈500 g) in a dynamic device under ambient T and P conditions, are comparable with those obtained treating 1 g-milled samples under enhanced operating conditions (T=50 °C and P=3 bar) in a static reactor applying a L/S ratio of 0.3 l/kg. In addition, differently from the static batch tests, prior to the granulation-carbonation tests the BOF slag was not subjected to any type of mechanical pre-

treatment. Hence, compared to other process routes investigated for accelerated carbonation of residues (i.e. slurry<sup>15</sup> and wet process in static conditions<sup>5</sup>), the tested treatment appears to be promising in consideration of the significant CO<sub>2</sub> uptakes and Ca to CaCO<sub>3</sub> conversion yields achieved (53-60% based on total Ca content) notwithstanding the mild conditions adopted.

**Table 2** Comparison of the IC content (% by weight) before (untreated slag) and after treatment (granulation and granulation-carbonation) at different reaction times. Results are reported as weighted averages of the values achieved for each class.

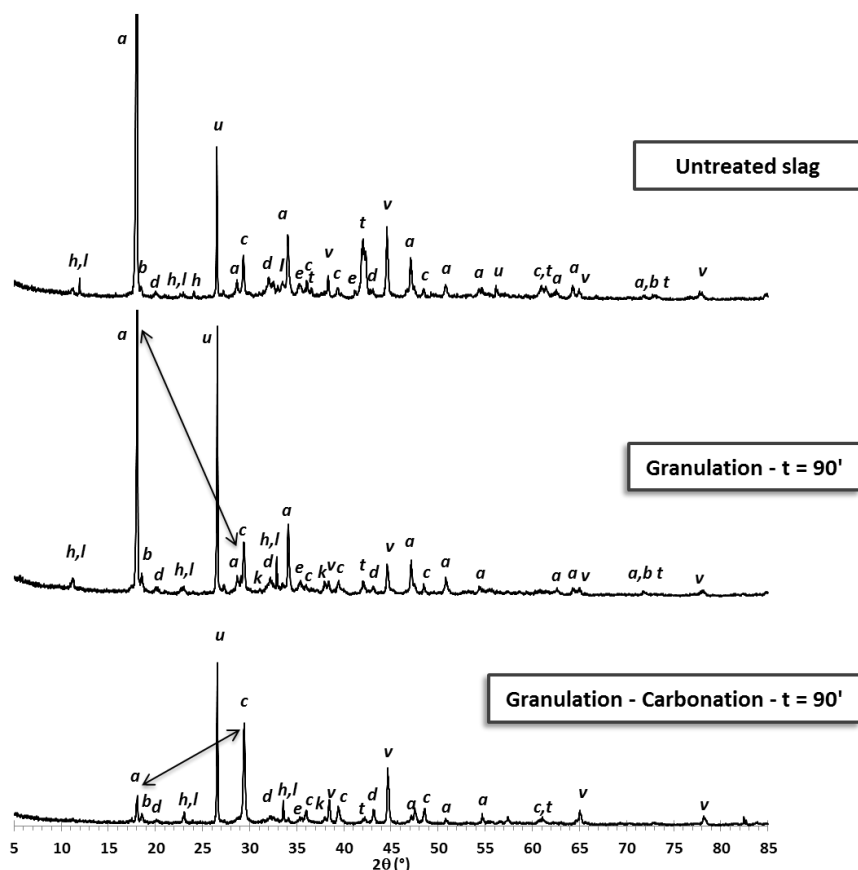
IC (%)		
Untreated slag		
0.54		
t [min.]	Granulation	Granulation-Carbonation
30'	2.06	3.53
60'	2.08	3.7
90'	1.71	3.91
120'	-	3.87



**Figure 3** CO<sub>2</sub> uptake as a function of time (min.), obtained from the granulation and granulation-carbonation tests, calculated as weighted averages of the CO<sub>2</sub> uptakes resulting for each class.

### Effects of the tested treatments on the mineralogy of the materials

XRD patterns of the untreated slag and of granules obtained after granulation and granulation-carbonation carried out for a reaction time of 90 minutes are presented in Figure 4. The mineralogy of the granulation product was very similar to that of the untreated slag, exhibiting a predominance of Ca phases (Ca hydroxide, Ca-containing oxides, Ca-silicate and calcite) and other oxides, such as coesite, vanadium oxide, magnetite and Mn-Mg oxide. After the granulation-carbonation treatment, the portlandite content decreased significantly while the ratio between calcite and portlandite peaks increased (see e.g. those highlighted by the arrows reported in Figure 4), indicating the reaction of Ca(OH)<sub>2</sub> with CO<sub>2</sub>. This result is in good agreement with the higher IC value measured in the product of the granulation-carbonation test compared to that of the granulation treatment (see Table 2).



**Legend:** *a*: portlandite [ $\text{Ca}(\text{OH})_2$ ]; *b*: brucite [ $\text{Mg}(\text{OH})_2$ ]; *c*: calcite [ $\text{CaCO}_3$ ]; *d*: larnite [ $\text{Ca}_2\text{SiO}_4$ ];  
*e*: magnetite [ $\text{Fe}_3\text{O}_4$ ]; *h*: Ca-Al-Fe oxide [ $\text{Ca}_2\text{Fe}_{1.4}\text{Al}_{0.6}\text{O}_5$ ]; *k*: corundum [ $\text{Al}_2\text{O}_3$ ];  
*l*: Ca-Cr-Fe oxide [ $\text{Ca}_2\text{Cr}_{0.5}\text{Fe}_{1.5}\text{O}_5$ ]; *t*: Mn-Mg oxide [ $(\text{MgO})_{0.725}(\text{MnO})_{0.275}$ ];  
*u*: coesite [ $\text{SiO}_2$ ]; *v*: V oxide [ $\text{VO}$ ]

**Figure 4** XRD pattern of untreated slag and of granules obtained from the granulation and granulation-carbonation treatments after a reaction time of 90 minutes.

### Effects of the tested treatments on the leaching behaviour of the materials

The results of the EN 12457-2 compliance test carried out on the untreated slag and on crushed material ( $d < 4$  mm) obtained from granulation and granulation-carbonation experiments at different reaction times are reported in Table 3. For all of the tested samples, only a few elements presented leachate concentrations higher than the instrumental quantification limit: major slag constituents (Ca, Na, K, Si, Al) and Ba. As can be noted in Table 3, the pH of the untreated slag resulted alkaline (12.4), in good accordance with its high  $\text{Ca}(\text{OH})_2$  content detected by XRD. The pH values measured in the treated samples showed to decrease slightly, but remained well above 12, indicating that portlandite was still the solubility controlling phase. As for the leaching of major slag constituents, Al and Si concentrations showed to increase after both types of treatment, while K and Na presented lower values particularly in the eluates of the products of the combined treatment. Furthermore, from Table 3 it can be observed that Ca concentrations did not show to vary significantly after neither type of treatment, in agreement with the pH values that remained above 12. As for Ba, eluate

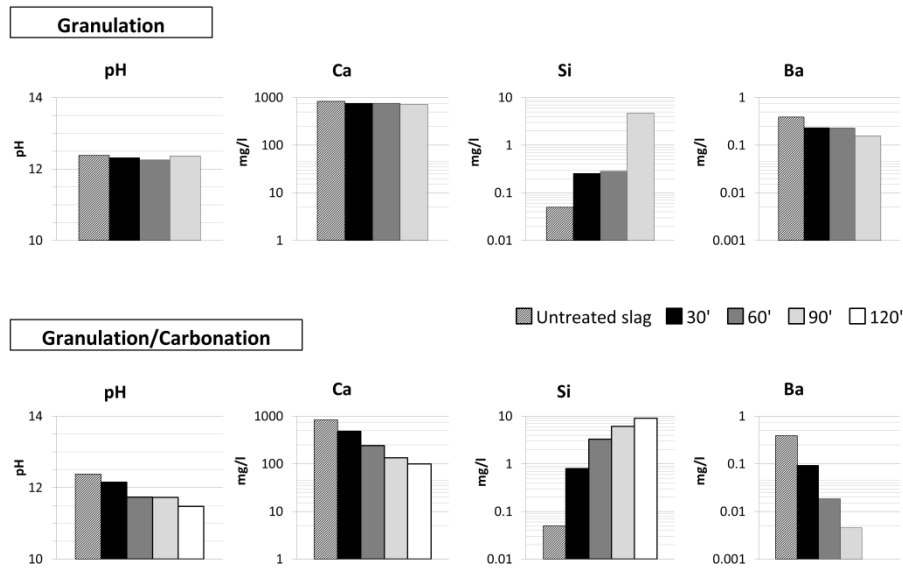
concentrations showed to decrease slightly after the granulation treatment and over a factor of 2 upon the combined treatment. These results indicate that the two processes, and in particular the combined one, led to the formation of more soluble Al and Si containing phases probably owing to hydration reactions, while other elements (i.e.: K, Na and Ba) appeared to be more tightly bound to the solid matrix.

In order to evaluate the effects of the agglomeration process on the release of major and trace components, leaching tests were also performed on each particle size fraction of the granules obtained after the treatments without grinding. In Figure 5 the pH and eluate concentrations of Ca, Si and Ba resulting for Class B granules produced after granulation and granulation-carbonation at different times are reported and compared to the values found for the untreated slag. After granulation, pH, Ca and Ba concentrations did not appear to vary significantly compared to the values obtained for the untreated slag, while Si release increased of up to one order of magnitude after 90 minutes of the treatment. The leaching behavior of the granules appeared to be considerably affected by the carbonation treatment, showing to vary increasingly with the reaction time. In particular, the pH values decreased from above 12 to 11 while Ca concentrations were reduced of up to one order of magnitude, as a result of the reaction of portlandite with CO<sub>2</sub> and consequent formation of calcite, a less soluble Ca-phase. Si concentrations increased of over two orders of magnitude upon carbonation, with a similar trend to that exhibited by the products of the granulation treatment, suggesting that the reaction of silicate phases such as larnite with CO<sub>2</sub> was quite limited as also indicated by the results of XRD analysis. Ba leaching from the granules showed to be greatly influenced by the carbonation treatment, decreasing to values lower than 0.5 µg/l after 120 minutes. The differences between the results of these tests and those reported in Table 3 regarding the product of the combined treatment may be related, besides to the effects exerted by particle size, to the higher carbonation degree resulting on the surface of the formed granules with increasing reaction times.

**Table 3** Main results of the EN 12457-2 leaching test on untreated slag and on crushed granules obtained after granulation and granulation-carbonation at different reaction times

	pH	Al mg/l	Ba mg/l	Ca mg/l	K mg/l	Na mg/l	Si mg/l
<b>Untreated slag</b>							
	12.38	0.013	0.39	828.77	16.47	226.04	0.05
<b>Granulation</b>							
<b>30'</b>	12.31	0.10	0.29	829.12	9.19	140.92	0.24
<b>60'</b>	12.31	0.09	0.30	827.26	10.38	149.78	0.22
<b>90'</b>	12.34	0.07	0.34	826.65	11.83	182.98	0.15
<b>Granulation-Carbonation</b>							
<b>30'</b>	12.33	0.13	0.21	806.75	10.58	96.88	0.24
<b>60'</b>	12.31	0.21	0.17	749.35	12.26	97.95	0.31
<b>90'</b>	12.30	0.20	0.16	790.60	8.13	100.06	0.31
<b>120'</b>	12.28	0.19	0.16	828.62	9.05	106.16	0.30





**Figure 5** Comparison of pH values and eluate concentrations of Ca, Si and Ba for untreated slag and class B granules obtained after granulation and granulation-carbonation treatment at different reaction times

### Effects of the tested treatments on the mechanical behaviour of the materials

One of the goals of the application of the tested treatments on BOF slag is the production of aggregates to reuse in construction works, in substitution of primary raw materials. Hence, in order to compare the mechanical properties of the treatment products with those of natural gravel, it was decided to perform the Aggregate Crushing Value (ACV) test on both types of materials. First results showed that the amount of fine particles ( $d < 2.36\text{mm}$ ) obtained applying the standardized confined compressive test on the treatment products was significantly higher (over 70% by weight of the material) than that achieved for natural gravel (20% by weight of the material). The ACV obtained for the granules resulting from the combined treatment appeared to be slightly higher than that of the material produced by the granulation test; however, further tests are being performed in order to better understand the effects of the two tested processes on the mechanical strength of the products.

### Conclusions

The aim of this work was to analyze the effects of a combined granulation and carbonation treatment applied to BOF steel slag as a  $\text{CO}_2$  storage technique and to produce aggregates for civil engineering applications. Hence, the main physical, chemical, environmental and mechanical properties of the granules were evaluated and compared to those resulting for the products of granulation tests carried out under atmospheric air. Results indicated that the combined treatment was able to produce granules with a tenfold larger particle size compared to the untreated slag and significant  $\text{CO}_2$  uptakes (12-14% wt.) even after short reaction times at ambient conditions, due to calcite formation upon  $\text{Ca}(\text{OH})_2$  reaction with  $\text{CO}_2$ . This reaction showed also to affect the leaching behavior of the produced granules in terms of pH and of the release of major components (Ca and Si in particular), as well as to cause a

decrease of Ba mobility. Finally, ACV tests are being performed to evaluate the effects of the tested treatments on granule strength.

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