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Utilization of Carbonated BOF Slag as Partial Replacement of Aggregate in Cement Mortars

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Abstract

After direct mineral carbonation, a material rich in carbonates and with reduced quantities of free oxides is obtained. The aim of this work was to show that such materials can be used in the construction domain. Basic Oxygen Furnace (BOF) slag from the steelmaking process has been traditionally seen as unfit for bounded applications due to its propensity to swelling, resulting from hydration of its high free lime content. Here, BOF slag was crushed to suitable particle sizes, carbonated in an aqueous solution of carbonic acid, and utilized to replace 50% of natural sand aggregate in cement mortars. The mechanical and chemical properties of these mortars were compared to mortars containing non-carbonated slags, and a standard cement mortar as a reference. Tests were conducted to determine mortar flow and soundness, and cured mortar compressive strength and leaching tendencies. The results showed a satisfactory performance for all considered aspects (comparable with the reference) of the mortar sample containing 37.5 wt% (1.5 in 4 parts solids) carbonated BOF slag of <0.5 mm particle size.

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Introduction

With the purpose of using steelmaking slags in the construction domain, and after much research conducted internationally, different standards have been implemented regarding the conditions that should be met by these industrial wastes before utilization. When intending to use slags as binders, the hydraulic activity of the material is very important.¹ However, with the use of BOF slag as a binder requiring intense grinding, and with the high metallic iron content of BOF slag, this processing step represents a high energy demand, and consequently a high cost. Lower processing costs can be achieved when using BOF as an aggregate, as the extent of particle size reduction (from monolith to millimeter-sized grains) can be reduced. For this application, the main parameters that must meet stringent specifications are the volume stability, referred to as soundness, the basicity, and the leaching of regulated toxic heavy metals and metalloids.² Wang et al.³ developed a usability criterion based

on the free lime content, the specific gravity and bulk relative gravity of the slag. Xie et al.⁴ found that the failure mechanism in BOF dense-graded asphalt mixtures in moist environment is determined by the formation of portlandite ($\text{Ca}(\text{OH})_2$) and calcium-silicate-hydrates (C-S-H), which leads to partial structural failure and facilitates water infiltration.

To obtain acceptable values of hydraulicity and soundness, one possible route is the reaction of the alkaline oxides in BOF slag with CO_2 , a process called mineral carbonation that leads to the formation of geochemically stable carbonates (e.g. CaCO_3), thus preventing the formation of expansive hydroxides and implicitly obtaining a stable material.⁵ An advantage of this approach is that both forms of free lime found in BOF slag (residual and precipitated) can be made to react with CO_2 , thus substantially reducing basicity² and likely propensity for swelling, although the latter has not been reported in literature.

Mineral carbonation has also been reported to yield positive effects in terms of basicity and the leaching behavior of alkaline earth metals, heavy metals and metalloids from steelmaking slags.⁶ However, Van Gerven et al.⁷ observed that the leaching evolution of some metals increases at high carbonation degrees; this means that an optimal level of carbonation might exist that minimizes leaching.

The noble purpose of sequestering carbon dioxide, however, is insufficient for extending the process of mineral carbonation to an industrial scale if the economical balance is not a positive one. For this reason, the materials obtained after mineral carbonation must be valorized by utilizing them in large-scale commercial applications. The purpose of this work was to assess the mechanical and chemical properties of cement mortars containing non-carbonated or carbonated BOF slag as a fine aggregate. This work also aimed at evaluating the effect of slag particle size on the carbonation extent, and the effects of slag particle size and carbonation degree on the mortar properties.

Materials and Methods

BOF Slag Characterization

The BOF slag used in this study was acquisitioned from a steel plant in Belgium. Prior to analyses or experimentation, the material was first crushed using a jaw-crusher (Retsch BB100) to obtain particles smaller than 1.6 mm. The obtained material was further divided into three particle size classes using standard sieves: <0.08 mm, 0.08-0.5 mm, 0.5-1.6 mm.

The chemical composition of the slag was obtained by X-ray Fluorescence (XRF, Panalytical PW 2400) analysis. High concentration of calcium (35.0 wt%) was observed, which imparts this material its alkaline properties and high reactivity towards mineral carbonation. Also, BOF slag contains large quantities of iron (20.6 wt%), an undesirable loss of product from the steelmaking process, and a significant quantity of silicon (5.8 wt%), important for the formation of silicate minerals.

The mineralogical composition of the slag was determined by Quantitative X-Ray Diffraction (QXRD) on a Philips PW1830, mineral identification was done in Diffrac-Plus EVA (Bruker) and mineral quantification was performed by Rietveld refinement technique using Topas Academic v4.1 (Coelho Software). Predominant minerals detected consist of Ca–ferrite and Ca–silicates, with Srebrodolskite (32.3 wt%) and β –polymorph of dicalcium silicate (30.8 wt%) being present in the highest quantities. Also observed are high quantities of calcium (8.8 wt%) and magnesium (1.1 wt%) oxides, which are the minerals detrimental to soundness.

Pressurized Slurry Carbonation

Each particle size class of the slag was individually carbonated under pressure and elevated temperature in a 1.1 L stirred batch autoclave reactor (Buchi Ecoclave 300 type 3E). For all the experiments, 200 g of material was added to 800 ml ultrapure water (18.2 M Ω ·cm); the slurry was stirred by a turbine impeller rotating at 1000 rpm. Carbonation was conducted at 90 °C and 20 bar, gauge CO₂ partial pressure, for 2 hours durations. The carbonated slurry was filtered and the recovered solids were dried for 24 hours at 105 °C prior to analyses and further utilization. Carbonation degree of BOF slag was determined by Thermogravimetric Analysis (TGA) using a NETZSCH STA 409 PC. Visualization of trapped carbon and of other elements within a carbonated BOF slag sample was realized through elemental mapping, performed based on Wavelength Dispersive Spectroscopy (WDS) by means of an Electron Probe Micro-Analyzer (EPMA) equipped with a Field Emission Gun (FEG) (JEOL JXA-8530F).

Preparation and testing of mortar samples

In order to test the possibility of using BOF slag as partial replacement of fine aggregate in building materials, mortar specimens were prepared according to the standard EN 196-1:1994, by using a binder/aggregate ratio of 1/3 and a binder/water mass ratio of 0.625. The natural aggregate used was standard quartz sand (CEN 196-1, at 0-2 mm particle diameter), and the binder used was cement CEM I 42,5 R HES (CCB Italcementi Group). The same mix ratios were also used when carbonated and non-carbonated BOF slag was used as replacement for half of the aggregate amount by weight. When one of the two finest slag classes (<0.08 mm and 0.08-0.5 mm) was used to replace the finer half of the natural aggregate, natural aggregate having particle size >0.5 mm (separated by sieving) was used as the other half. When the coarsest slag class was used (0.5-1.6 mm), an equal amount of natural aggregate having particle size <0.5 mm was used.

Consistency of the fresh mortar mixes was tested using a flow table (according to EN 1015-3:1998), and then cast in a mould that permitted four specimens (with dimensions 20x20x160 mm) of the same mortar sample to be obtained. After 60 cycles of shock compaction, the material was kept in the mould for 24 hours, and the demoulded specimens were placed in a moist room at \geq 93% humidity and at constant temperature of 20 °C. Different curing durations (1 to 28 days) were used to assess its effect on the compressive strength. Also, some samples were subjected to accelerated curing in boiling water to assess the effect of this curing condition on compressive strength. The intention was to magnify the effect of unhydrated oxides on volumetric

expansion and compressive strength by boiling in water for an extended period. After curing, specimens were dried for 48 hours at a temperature of 40 °C to remove excess pore water. After drying, each specimen was cut into four cubes (20x20x20 mm), and each cube was tested for compressive strength (according to EN 196-1) in a Schenk Trebel screw tester at a compression rate of 0.2 mm/s.

Each mortar mixture was also used for soundness testing (Le Chatelier test, according to EN 196-3). Samples mounted in the appropriate moulds and under load were kept in a water bath at 20 °C for 24 hours, and then boiled for 3 hours. Distances between indicator arms before and after boiling were measured by caliper and the difference (equivalent to sample expansion/swelling) were recorded.

Basicity and leaching behavior of the non-carbonated and carbonated slag powders and that of the mortar samples were tested using batch leaching test methodology (EN 12457-4). The crushed samples from compressive strength tests were first jaw-crushed and sieved to <1.6 mm. The solids were added to sealed bottles containing ultrapure water at a ratio of 10 g/100 ml and shaken for 24 hours at 160 rpm and 25 °C on a shaking table (Gerhardt Laboshake). Basicity was determined by pH measurement of the final slurry. The slurry was then centrifuged for 5 minutes at 4000 rpm (Heraeus Labofuge 400) to separate the supernatant, which was then diluted in 0.3 M nitric acid solution and analyzed in triplicate for metal and metalloid content by Inductively Coupled Plasma Mass Spectrometry (ICP-MS, Thermo Electron X Series).

Water absorption of non-carbonated and carbonated slag powders, and of the standard sand, was measured using an adapted methodology based on norms EN 1097-6 and BS:812 Part 2. Dry samples of the materials were mixed with DI water at a mass to volume ratio of 25 g to 100 ml in sealed bottles. The samples were allowed to hydrate statically for 24 hours. The resulting slurry was then vacuum-filtered on Whatman paper 5 to recover the moist solids. The difference in mass between the wet solids and the initially dry solids is taken as the total water absorption. The moist solids were then dried in an oven at 105 °C for 24 hours and re-weighed. The difference in mass between the moist and the dried solids is taken as physically absorbed water, while the difference in mass between the dried and the initially dry solids is taken as chemically absorbed water (i.e. water that is consumed by the formation of hydroxides and hydrates).

Results and Discussion

CO₂ Uptake

The CO₂ uptake of carbonated materials was determined, and thus the evolution of CO₂ uptake based on particle size distribution was obtained. An increase of CO₂ uptake is observed once the particle size is reduced (10.0 wt% uptake for BOF slag <1.6 mm, compared with 16.2 wt% uptake for BOF slag <0.08 mm). This trend is easily explainable by the material's exposed surface increase that occurs when particles size is diminished, thus promoting contact between carbonic acid from the solution with the reactive minerals on the particle surfaces. Once the carbonation extent increases, the formation of passivating layers prevents access of the carbonic acid to the

unreacted particle core.⁸ Greater mineral conversion is thus obtained for finer particles. Based on the calcium content of the fresh BOF slag, the maximum theoretical CO₂ uptake of the slag is 27.8 wt%. Accordingly, the Ca-conversion of the finest sample is 58.3 %, compared to 36.0 % for the coarsest sample.

EPMA analysis allowed insight on the distribution of the main constituent elements and mineral phases in the carbonated BOF slag <0.5 mm sample. Notably, no particle containing calcium but absent in other elements, which would correspond to free lime, is found. This signifies extensive free lime conversion. This result is corroborated by the elimination of chemical water absorption of the carbonated slags, according to Figure 1, which occurs in non-carbonated slag due to the hydration of free CaO into Ca(OH)₂.

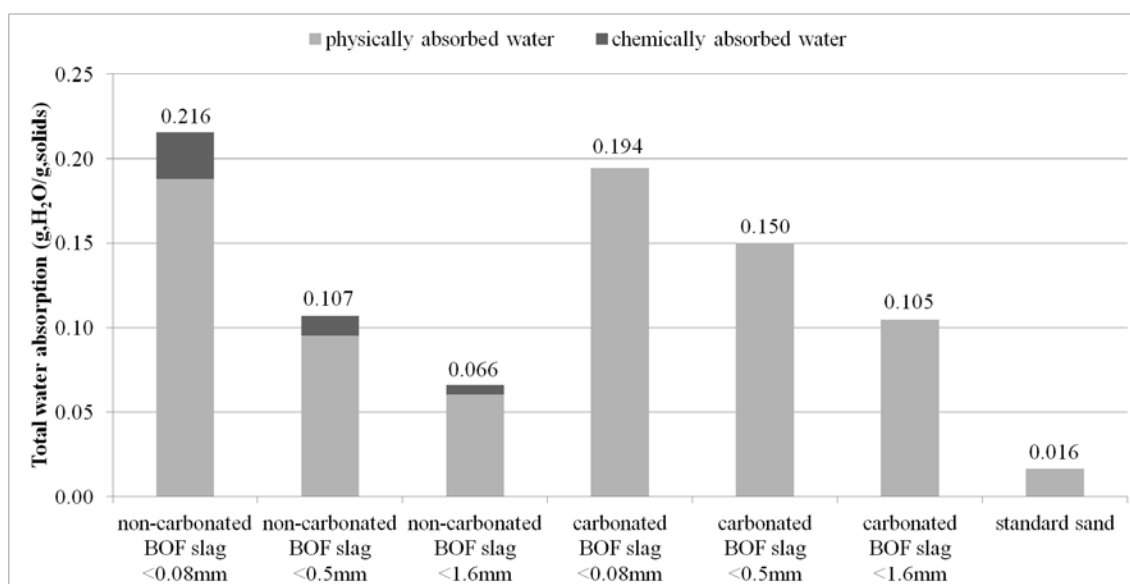


Figure 1. Water absorption (physical, chemical and total) of non-carbonated and carbonated BOF slags and of standard sand

Evidence was found by EPMA that lime may readily dissolve in the carbonation medium, and precipitates in the bulk solution as large crystals. In contrast, CaCO₃ originating from silicates precipitates immediately on the silicate surfaces, seeing that all dicalcium silicate grains were enveloped by CaCO₃ layers. Visual evidence was also found that dicalcium silicate reacts more extensively than srebrodolskite.

Mortar Paste Consistency

For all mortar samples, a constant water/cement weight ratio was used (0.625); hence consistency of mortar pastes was not controlled, but rather was a response variable. Flow table values were recorded to assess the impact of aggregate replacement (sand by slag) on the particle size distribution of the aggregate fraction, which has important implications on mortar consistency and strength properties. The presence of small sized aggregate particles in an excess amount leads to increased physical water absorption, as shown in Figure 1. This effect can lead to a decrease in mortar flow, and can further affect its density through formation of larger voids that reduce compressive strength.

Good agreement between paste consistency and mortar density was observed for samples containing non-carbonated or carbonated BOF slag <0.08 mm, and for that containing carbonated <1.6 mm BOF slag. For these samples, results from flow table (110 mm, which signifies no flow) and density (1.5-2.2 g/cm³) measurements were the smallest. Small specimen densities are a consequence of higher voids, which is a direct result of poor flow behavior of the mortar. Flow table values obtained for mortar samples containing non-carbonated <1.6 mm BOF slag or non-carbonated/carbonated <0.5 mm BOF slag (123-136 mm, compared to 161 mm for the reference specimen) were reasonable for mortar applications. These specimens had densities of 2.4 g/cm³, compared to 2.3 g/cm³ for the reference. These results have important effects on a specimen's compressive strength.

Soundness of Mortars

For a material to be considered stable, the difference between the value read before boiling and the value read after boiling, during the Le Chatelier test, should not be greater than 5 mm.⁹ In this study case, the reference mortar sample indicated a swelling expansion of only 0.9 mm. Other samples that remained under 5 mm variation were the mortar samples with 50% aggregate replaced by carbonated BOF slags of particle sizes <0.08 mm (-0.3 mm contraction) and <0.5 mm (1.1 mm swelling). All remaining mortar samples, including all which contained non-carbonated slag, registered swelling values substantially greater than 5 mm (11.4 to 91.7 mm swelling), placing them in the category of materials unsuited for use in the construction domain.

Notably, coarser slags had greater swelling. It had been rather expected that smaller particle size of the untreated BOF slag would conduce to greater swelling, since greater contact surface would be available to react with the aqueous solution. Instead, it appears that the finer slag becomes extensively hydrated, but not completely, already during paste preparation, and thus it suffers diminished delayed hydration (i.e. during curing). These results confirm the theory that free oxides, particularly lime, have a detrimental effect regarding volume stability.

A swelling value of 15.7 mm was obtained for the mortar sample containing carbonated <1.6 mm BOF slag. In this case, the reason for volume instability can be attributed to insufficient carbonation extent of slag. This means that a more intense process should be used for carbonation of materials with larger particle sizes, or further comminution should be done prior to, or during carbonation.

Compressive Strength of Mortars

The main objective of the compressive strength test was to assess the effect of aggregate replacement relative to the reference composition. The compressive strength data from each mortar mixture is presented in Figure 2. These are average values of the four specimens cured under different conditions (duration and standard or accelerated curing); no significant statistical difference was observed from the different curing conditions. On the other hand, significant differences were observed between mortars of different compositions.

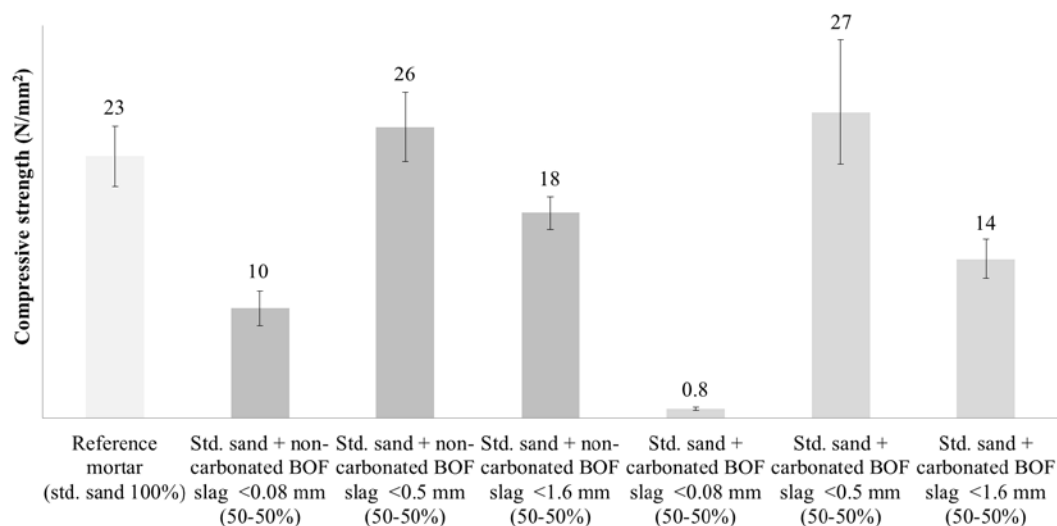


Figure 2. Average values of compressive strength for all mortar specimens (reference, non-carbonated BOF slag aggregate replacement, and carbonated BOF slag aggregate replacement); error bars are based on data from samples treated under different curing conditions (1 and 7 days followed by boiling, and 7 and 28 days without boiling) and quadruplicates of each measurement

From Figure 2 it is observed that the smallest values of compressive strength belong to the fairly dry mortar mixtures that showed insufficient flow, which included the two samples prepared with the finest BOF slag fractions (<0.08 mm). At the same time, the mortar samples prepared with the coarsest BOF slags (<1.6 mm) had compressive strengths significantly lower than the reference. This is in agreement with their volume instability being greater than that of samples prepared with the finer BOF slag fractions. Hence, the only mortar specimen that obtained similar flow value, soundness and compressive strength values as the reference mortar specimen, was the one with carbonated BOF slag of <0.5 mm particle size. For this mortar, the compressive strength value was averaged at 27.3 N/mm², even higher than the value obtained for the reference mortar (23.4 N/mm²). This result seems to be due to the fact that mortars with carbonated slag as aggregate possess less pore water, as indicated with their flow table values, which in turn can be attributed to higher water absorption of the carbonated slag particles when compared to that of sand particles.

Results from compressive strength test, supported by the other tests (flow table and soundness), demonstrates the utility of the carbonation process as a treatment method for BOF slag towards its use as aggregate in the construction domain. Also, it can be concluded that if BOF slag is used as stated, this material should present a particle size distribution according to the standard EN 196-1:1994, and the free lime conversion after mineral carbonation should be high enough to ensure an acceptable value of soundness for the final building material.

Leaching from Mortars

Based on the abovementioned results, the only BOF slag fraction (carbonated and non-carbonated) used in leaching tests was that with particle size <0.5 mm, along with the crushed and ground mortar samples containing this type of slag, and the crushed and

ground reference mortar sample used for comparison. The aim was to compare the leaching tendency of some regulated metalloid and heavy metals from the slag, and assess the effect played by both mineral carbonation and partial containment in a cementitious matrix on this tendency. At the same time, by comparing the leaching results from the reference mortar sample with those from the slag containing mortar samples, the differential influence on leaching tendency by including the BOF slag in the mortar mixture can be deduced.

It was found (Figure 3) that after mineral carbonation the leaching tendency of several elements drops, namely barium, nickel and lead, while it remains nearly unchanged for chromium and copper, and it increases substantially for vanadium. The leaching tendencies of cobalt and molybdenum remained below the detection limit (0.1 mg/kg). However, including the BOF slags in a cementitious mortar matrix led to full containment of the metalloid's leaching tendency. Leaching of lead and nickel similarly decreased from the slags contained in mortars.

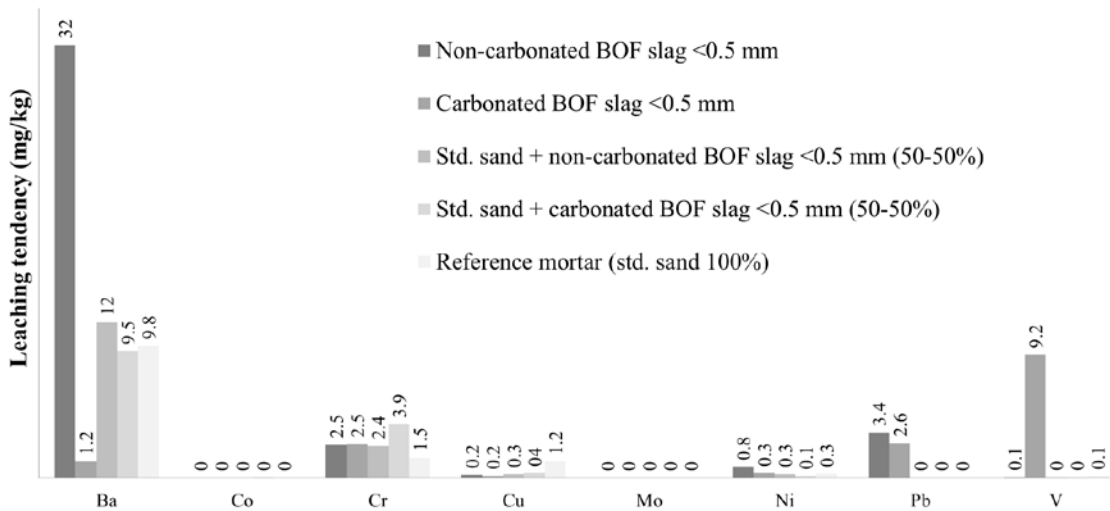


Figure 3. Leaching tendency of eight metalloids and heavy metals from <0.5 mm BOF slag before and after carbonation, from mortar samples containing <0.5 mm BOF slag (carbonated or non-carbonated) as partial aggregate replacement, and from reference mortar sample; value of '0' means <0.1 mg/kg

The only result of concern was that of chromium leaching from the carbonated BOF slag-containing mortar. Its Cr leaching tendency was the highest of all tested materials. Yet, the leaching extent from this material is still comparatively low; the Belgian-Walloon limit for Cr(VI) leaching from re-used waste materials is 20 mg/kg.¹⁰ According to Fernández-Bertos et al.¹¹ and Cornelis et al.¹², leaching of chromium at high pH in alkaline materials is controlled by Cr substitution in calcium-silicate-hydrates (CSH), the main component of hydrated cement. Thus, over time, it is possible that chromium leached from BOF slag can become chemically bonded to the surrounding matrix, thus minimizing the leaching risk from the construction material for the duration of its life cycle, including its CDW (construction and demolition waste) phase.

Differing leaching tendencies from slag-containing mortar samples compared to leaching values of the same elements from the pure slags are linked to the basicity of the materials, and thus the pH values of the leaching solutions. A decrease of approximately one pH unit was registered for the carbonated BOF slag compared to the non-carbonated slag. The mortar samples, however, had elevated pH ranging from 12.4 for the reference sample to 12.7 for the mortar containing non-carbonated BOF slag. The high pH values of the mortars represent the reason for which vanadium leaching was kept low, since V is known to mobilize in response to pH reduction.¹³

The results regarding leaching from mortar samples containing carbonated BOF slag as partial replacement of natural sand aggregate provides further confirmation that mineral carbonation process can be considered an effective treatment method for metallurgical slags prior to utilization of this material in the construction domain. A further research direction that can be investigated is the inclusion of sorbent materials as partial aggregate replacement to further attenuate the leaching tendency of elements of concern, such as chromium in the present case.

Conclusions

The research presented in this paper aimed to demonstrate that mineral carbonation can be considered a route to treat steelmaking converter slags in order to further use this residual material as a partial aggregate replacement in the domain of civil constructions. Mineral carbonation of BOF slags leads to the sequestration of meaningful amounts of CO₂, 0.11–0.19 kg,CO₂/kg,slag at the conversions achieved herein. Taking into account a 50% replacement ratio of natural aggregate, and an aggregate to binder ratio of 3, the prepared mortars contained up to 74 grams of CO₂ per kilo; this categorizes it as a carbon sink.

The mineral carbonation process also represents a means of treatment by forming stable carbonates through reaction of calcium and magnesium oxides with CO₂. This is especially important for the stabilization of free lime, which was shown to have detrimental effects on the hydration-driven volume expansion of mortars prepared with non-carbonated BOF slag. In contrast, for mortar samples containing carbonated slag, soundness increased in proportion to the carbonation conversion, reaching values similar to the soundness of reference cement mortar.

Particles size had an important influence on flow value of the fresh mortar and, implicitly, on compressive strength of the hardened mortar specimens. From this point of view, it is concluded that maintaining the particle size distribution of the aggregate mixture according to the standard norms is critical for ensuring suitable consistency and mechanical strength.

The best result obtained in terms of the combination of tested parameters (consistency via flow table test, soundness, compressive strength and leaching of toxic elements) corresponded to the mortar mixture prepared with carbonated BOF slag of <0.5 mm particle size. In light of these results, it is possible to conclude that BOF slag utilization as partial aggregate replacement in construction domain can be a realistic method to valorize and recycle this kind of industrial residue.

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