

Research Article

Long Term Compression Strength of Mortars Produced Using Coarse Steel Slag as Aggregate

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The paper reports on some experimental results obtained from the production of mortars prepared using a commercial cement, coarse steelmaking slag, superplasticizer, and water. The behaviour of this reference composition was compared to that of some others containing further additives in order to investigate materials compressive strength after long time ageing. It has been demonstrated that an optimized water/cement ratio coupled with slag particles of size lower than 2.5 mm and proper protocol of preparation leads to the production of materials with good mechanical properties after 28, 90, and 180 days of ageing. The resulting materials therefore appeared as good candidates for civil engineering applications. However, the present research also demonstrates that the mortar samples of all of the compositions prepared suffer from decay and compressive strength decrease after long time ageing in water. In the present paper the results are explained taking account of materials residual porosity and alkali silica reaction which occurs in the samples.

1. Introduction

Steelmaking slag (SS) is obtained in large quantities from steel production. It is known that the production of one ton of steel implies the production of 130–200 kg of slag, depending on the composition of the steel and on the steel production process. The great amount of SS produced all over the world implies that their recycling is presently necessary not only due to the rising cost of their possible landfill disposal which is reflected on the cost of the steel produced, but also as a consequence of the “zero-waste” objective which must be the final goal of all future human activities. In fact, it is generally accepted that a possible way for SS recycling is the preparation of roadways embankment which however could have effects on environment due to their natural elution.

SS derives from a high temperature process and, as a consequence of its large lime content, is made unstable by two phenomena: the first is due to the presence of $2\text{CaO}\cdot\text{SiO}_2$, which can transform, on cooling or later, into a phase with a larger specific volume [1] crumbling the slag which results in a mixture of submicronic-micronic-granulated particles; the second phenomenon is due to the presence of free lime which

causes grains swelling for hydration in atmospheric environment after cooling. Such volumetric instabilities are a great disadvantage when slag is used as a raw material for recycling. When the slag also contains free MgO or MgO based compounds, the above phenomena are coupled with a retarded hydration which, in general, occurs when these products are present in mortars or concrete [2, 3]. In order to obtain sufficient stability, various methods have been proposed like long time weathering of the granulated slag outside the slag pits, treatment of the liquid slag by injecting oxygen and silica, autoclaving of the slag in slag baskets [4, 5], and others [6–8].

It is generally accepted that steelmaking slag recycling is convenient provided that the resulting materials have good durability and maintain properties at least equal to the limits established by the official standards for production after long time ageing.

Several authors proposed the use of cement blended with SS for concrete manufacturing [9–11]; for such application, SS needs to be converted, by milling, into a powdered product.

Other authors have found that good properties could be obtained if SS are used for a partial replacement of sand fines with optimized replacement ratios [2, 3, 12–15].

TABLE 1: Mortars mix proportions and samples symbolic names.

	Reference	Chromium oxide	Ultramarine	Aluminium silicate (Na)
Cement (g)	550	550	550	550
Slag (g)	1650	1650	1650	1650
s/c	0.015	0.015	0.015	0.015
w/c	0.36	0.36	0.36	0.36
Chromium oxide (g)	—	66	—	—
Ultramarine (g)	—	—	66	—
Aluminium silicate (Na) (g)	—	—	—	66

Independently on their chemical composition, SS normally displays a wide particle size distribution which also depends on the slag origin, the steel production process, and the time elapsed from slag generation. SS morphologic characteristics are an additional parameter that must be taken under consideration during mortars or concrete preparation.

Nowadays, the use of SS containing small as well as coarse particles, for mortars or concrete production, is still matter of discussion [16].

The aim of the present paper is to report and discuss compressive strength data obtained from mortars containing cement, coarse steel slag, superplasticizer, and water. Mortars were produced using a fixed slag/cement ratio (1/3), as it has been often proposed in literature [13, 17–19]: a fixed superplasticizer/cement ratio (s/c) and a fixed water/cement ratio (w/c). Materials were aged in water for different times up to the limit of 1100 days (d). The reference composition was then modified by the addition of 3 wt% of the total solid material content by several compounds, namely, ultramarine, aluminium silicate (Na), and the green colour (chromium oxide). Hydrated products were characterized by compressive strength and water absorption tests after 28, 90, 360, 720, and 1100 d.

2. Experimentals

2.1. Materials. Starting materials for mortars preparation were a commercial CEMIIB-LL 32.5N cement; the superplasticizer Glenium 51 (BASF); a steel slag, obtained from the production of a chromium-manganese austenitic steel by a rotary kiln, as aggregate; and water. This reference composition, hereafter called R, was then modified by the addition of 3% of the total starting solid mass with one of the following powders: 817B-ultramarine colour (SIOF spa: Società Italiana Ossidi Ferro, Pozzolo Formigaro-Vr, Italy), Na-aluminium silicate (82% SiO₂, 9.5% Al₂O₃, 8% Na₂O; Fluka Analytical), and 812B-green chromium oxide (SIOF spa: Società Italiana Ossidi Ferro, Pozzolo Formigaro-Vr, Italy). Mortars mix proportions and samples symbolic names are reported in Table 1.

2.2. Characterization of the Starting Materials. The slag was previously naturally weathered for one month outside the production plant; after that it was thoroughly wet and then milled by a hammer mill in order to reduce the maximum particle dimension below the size of 2.5 mm. SS contains 2/3 of fines and 1/3 of coarse particles. In the present research,

TABLE 2: Composition, LOI, and density of CEIIB/B-LL and SS. “Others” indicates the cumulative quantity of all oxides determined in quantity lower than 0.1 wt%.

Component	CEMIIB-LL (wt%)	SS (wt%)
CaO	62.3	38.2
SiO ₂	19.8	14.8
Al ₂ O ₃	4.2	7.4
MgO	2.5	11.7
Na ₂ O	0.6	—
K ₂ O	1.1	—
Fe ₂ O ₃	3.2	9.8
MnO	0.6	10.4
Cr ₂ O ₃	—	2.0
V ₂ O ₅	—	0.8
P ₂ O ₅	—	1.2
SO ₄ ²⁻	2.9	—
Others	2.8	3.7
Free lime*	—	1.9
LOI (%)	4.25	—
Density (g/cm ³)	2.46	3.29

*The amount of free CaO was previously measured following the ethylene glycol method.

fines are considered the slag fraction containing particles with size <1000 μm.

The chemical analysis of the slag and that of the cement, determined by a SpectroMass 2000 induced coupled plasma (ICP) mass spectrometer, is reported, in terms of oxides, in Table 2. The amount of free lime in the slag was also determined following the ethylene glycol method. The density was determined following the ASTM C127 and C128 norms; results are averaged over the fine and coarse slag fractions.

Crystalline phases of slag and cement were investigated by X-ray diffraction (XRD). XRD patterns were recorded on a Philips X’Pert diffractometer operating at 40 kV and 40 mA using Ni-filtered Cu-Kα radiation. Spectra were collected using a step size of 0.02° and a counting time of 40 s per angular abscissa in the range of 20–80°. Philips X’Pert High Score software was used for phase identification.

An Horiba LA950 laser scattering instrument was used to investigate the particles size distribution (PSD) of slag fines and cement: analyses were made in water after 3 min sonication. For clarity of comprehension all PSD curves are represented with logarithmic abscissa and reported in Figure 1.

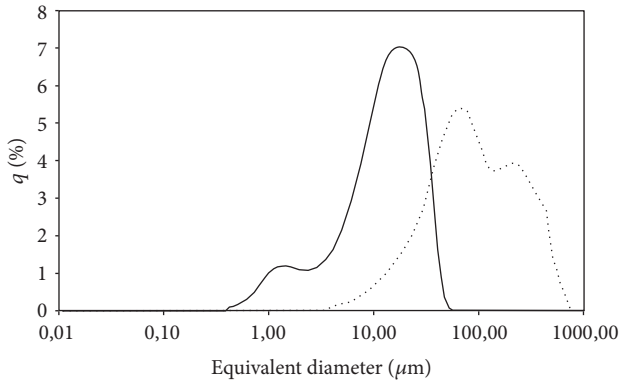


FIGURE 1: Particle size distribution of the fine fraction of slag (interrupted line) and CEMIIB-LL (plain line).

2.3. Mortars Preparation. For the mixture preparation and w/c optimization a 5 L Hobart planetary conforming to ASTM C305 standards was used. The optimized amount of water was determined by the ASTM C1437 slump test performed on the reference blend R. The paste is said to have the right workability if the cake width is 180 (± 20) mm, according to UNI 7044:1972 and ASTM C230 norms. The identified optimal w/c ratio of the reference blend (R) was 0.36; this same ratio was applied to all the compositions.

All mortar pastes were prepared by the following protocol: the full amount of slag was put into the mixer together with the total amount of water and superplasticizer and mixed for two minutes; after this previous homogenization, also the additive was introduced into the mixer and the paste was stirred for 15 min. This time elapsed, cement was added, and the product was worked on for 5 min.

Pastes were then poured under vibration into the moulds with dimensions of 100 \times 100 \times 100 mm (for compression tests) or 40 \times 40 \times 40 mm (for water absorption tests), sealed with a plastic film to ensure mass curing, and aged 24 h for a first hydration. Samples were then demoulded and cured in air for 24 h and then in water at room temperature for 28, 90, 180, 360, 720, and 1100 d. After production, materials ageing was performed separately to avoid contamination between the different compositions. The ageing water was maintained at the constant temperature of 25°C ($\pm 3^\circ\text{C}$) and replaced with fresh water every 7 d. After curing, before their characterization, samples were dried with a cloth and aged in the atmosphere for 24 h.

Materials ageing by submersion in water was set up in order to study their behaviour under severe conditions and consequently to test their behaviour in an eventual use under moist environment. In absence of an autoclave treatment such ageing was considered suitable for products where the retarded hydration could have negative impact on their durability.

2.4. Characterization of Hydrated Materials. A modified ASTM C 642 norm was used to test water absorption of the hydrated samples. After curing, samples were put in an oven at 80 \pm 5°C for 24 h and weighed (W_1); they were then aged in an autoclave, at 120°C and 2 kPa for 2 h using 2 L of water.

After boiling, they were cooled down to room temperature (in water), dried with a cloth, and weighed again (W_2). Water absorption was evaluated using the following equation:

$$W (\%) = \frac{100 (W_2 - W_1)}{W_1} \quad (1)$$

Compression tests were performed, in accordance with the ASTM C39 norm, using Test Mark CM8000 apparatus; data were averaged over 3 measurements.

Expansion was measured by a caliper, before the compression tests after 360 d of curing, on 100 \times 100 \times 100 mm cubic samples.

3. Results and Discussion

During weathering, the slag was often turned over in order to favour any possible natural hydration of the unstable components. Such compounds, if present in the fine fraction of the slag, simply contribute to materials' expansion during the first hydration step when materials are not yet hardened [2, 3]. However, if they present as clusters of particles, more or less stuck together, they suffer from uncontrollable hydration which could occur after mortars hydration when materials are brittle. The reduction of the maximum particles dimension below the size of 2.5 mm permits to optimize the contact with water during weathering, during hammer milling, and then, after that, during the stirring steps of mortars preparation.

Table 2 shows that the slag contains large amounts of CaO, SiO₂, Fe₂O₃, MgO, MnO, and Al₂O₃. All the other oxides have been detected in lower quantities even if the amount of Cr₂O₃ cannot be considered negligible. The absence of relevant amounts of toxic elements can also be remarked. Table 2 also shows that free lime is 1.9% and the ratio (CaO + MgO)/SiO₂ is superior to 1.4 which would lead to expectation of hydraulic activity [20–23]. It can also be observed that density is 3.29 g/cm³, in line with those reported by other researchers [10, 13, 24]. CEM II/B-LL conforms to European Standards EN-197/1.

Figure 1 shows the PSD curves of the fine fraction of slag and cement. It can be observed that cement displays a maximum concentration of particles at around 20 μm but contains also a little fraction of smaller particles highlighted by a small peak at around 1.5 μm ; the milled and sieved (1000 μm) SS has a PSD with a very large particle size distribution highlighted by a double peak with maximum at around 80 and 350 μm , respectively.

The XRD analysis demonstrates that the slag contains several phases, some of them in very small amount so that it has not been possible to doubtless recognize them; the present paper, therefore, reports only those that were identified by a minimum of four representative peaks. In detail, the presence of aluminium calcium oxide (PDF 00-006-0495 Ca₃Al₂O₆), pyroxmangite (PDF 01-084-1774 Mg_{0.97}Mg_{0.03}SiO₃), glaucocroite (PDF 00-014-0376 (Ca, Mn)₂SiO₄), iron oxide (PDF 01-084-0308 Fe₂O₃), calcium silicate (PDF 01-076-0799 Ca₂SiO₄), and calcium magnesium silicate (PDF 00-013-0498 Ca_{6.1}Mg_{1.1}Si_{3.6}O_{14.4}) has been

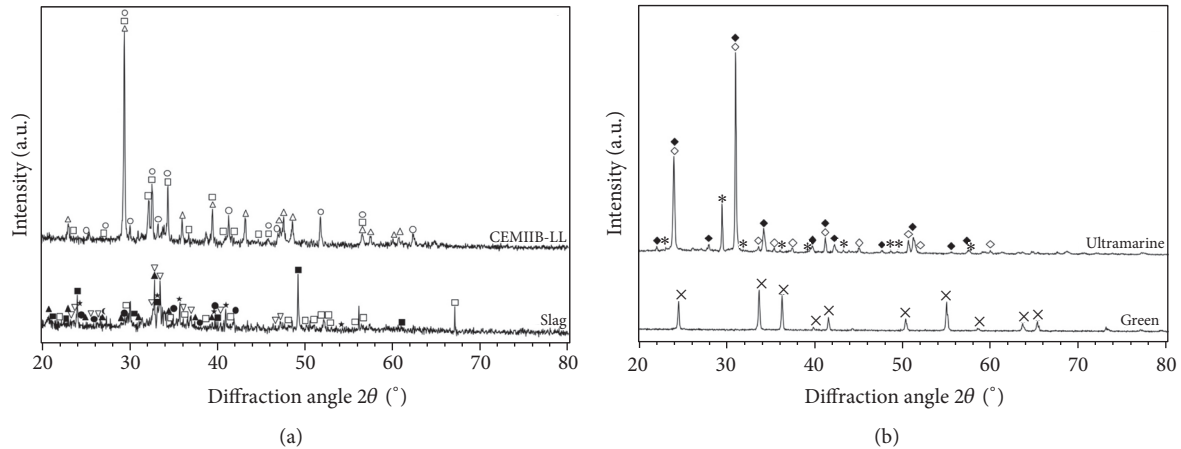


FIGURE 2: (a) X-ray diffraction patterns of CEMIIB-LL and slag. It is shown that cement contains calcite (Δ), C3S (\circ), and C2S (\square) whereas the slag contains calcium aluminium oxide (\blacktriangle), pyroxmangite (\bullet), glaucochroite (\blacksquare), iron oxide (\star), calcium silicate (\square), and calcium magnesium silicate (∇); (b) X-ray diffraction patterns of ultramarine and green colour: ultramarine contains ankerite (\diamond), sodium aluminium silicate (\blacklozenge), and magnesium calcite ($*$); green colour is mainly chromium oxide whose peaks are identified by (\times).

revealed. The presence of the above phases is documented by Figure 2(a) which also shows that CEMIIB-LL contains C3S (PDF 01-073-1929 Ca_3SiO_5), C2S (see above), and calcite (PDF 01-088-1807). X-ray diffraction also confirmed that green colour is mainly chromium oxide (PDF 01-074-0326 Cr_2O_3), whereas ultramarine, which is known to be a complex sulfur-containing sodium aluminium silicate ($\text{Na}_{8-10}\text{Al}_6\text{Si}_6\text{O}_{24}\text{S}_{2-4}$), also revealed the presence of ankerite (PDF 00-012-0088 $\text{Ca}(\text{Mg}_{0.67}\text{Fe}_{0.33}^{+2})(\text{CO}_3)_2$) and a calcite modified by a small amount of Mg (PDF 01-089-1306 ($\text{Mg}_{0.06}\text{Ca}_{0.94})(\text{CO}_3)$) as it can be observed in Figure 2(b).

In the present experimental work, components homogenization, during mortars preparation, has an important but not the most important role for the production of stable materials. In the experimental section, it has been already described that slag was previously worked in order to reduce the size of coarse particles and, at the same time, to favour hydration of the major part of its hydraulic compounds. However, after milling, the slag still contains large clusters whose core could never be wet by water thus maintaining hydraulic activity even after mortar pastes production. Such hydraulic activity could then emerge when materials are in use. In fact, if their hydration occurs after demoulding on hydrated-hardened materials, unwanted fractures, spalling, and/or chipping could appear. The preparation protocol followed in the present research favours an almost good hydration of the hydraulic compounds of the slag; nevertheless, after long term ageing in water, several compositions displayed the above phenomena as documented by Figures 3(a) and 3(b) which show the surface of the reference samples after 1 year of ageing in water.

It is clear that hydration is favoured by a high quantity of water coupled with a long mixing time. On the other hand, it is known that a high amount of water is in conflict with the production of materials with low open porosity and good mechanical performances. The addition of superplasticizer

was therefore considered necessary for the production of fluid pastes and, after hydration, of materials with strength in line with the official norms.

In addition, it has been observed that, during mortar paste homogenization, at the early stage of mixing, the mixture containing slag, superplasticizer, water, and additive behaves like moist soil that turns into a fluid slurry after about 10 min. This particular rheological behaviour is probably due to bleeding which is amplified by the quite high level of MgO which is contained in the slag [9, 18].

Figure 4 shows compressive strength (a) and water absorption (b) trends of samples tested after 28, 90, 180, 360, 720, and 1100 d; error bars are also reported.

In Figure 4(a), it is observed that the reference composition displays a strength of 35 MPa after 28 d which raises to 47 after 90 d and remains constant up to 180 d but decreases for longer ageing times: samples tested after 360 and 720 d show a strength of 44 and 26 MPa, respectively. After such ageing time, cubic specimens self-crumbed and were not tested. In parallel, the corresponding water absorption data (Figure 4(b)) are 6.5 after 28 d and 5.2 after 90; this value remains almost constant after 180 to 360 d but grows for longer ageing times reaching the value of 7.7 after 720 d.

The behaviour of the composition containing aluminium silicate (Na) can be also observed in Figures 4(a) and 4(b). It is displayed that samples have a constant compressive strength of 55 MPa between 28 and 180 d but decreases to 48 MPa after 360. For longer times, samples self-crumble on ageing and therefore were not tested at 720 and 1100 d; the water absorption is 4.2 after 28 d and remains almost constant up to 180 d but increases to 4.8 at 360 d.

Materials containing ultramarine display a strength of 68 MPa after 28 d which raises to 76 after 90 and 78 after 180 but decreases to 76 after 360 d, 63 after 720 d, and 49 after 1100 d; the corresponding water absorption is 3.6 after 28 d, 3.2 after 90 d, and 2.9 after 180 d; this value remains

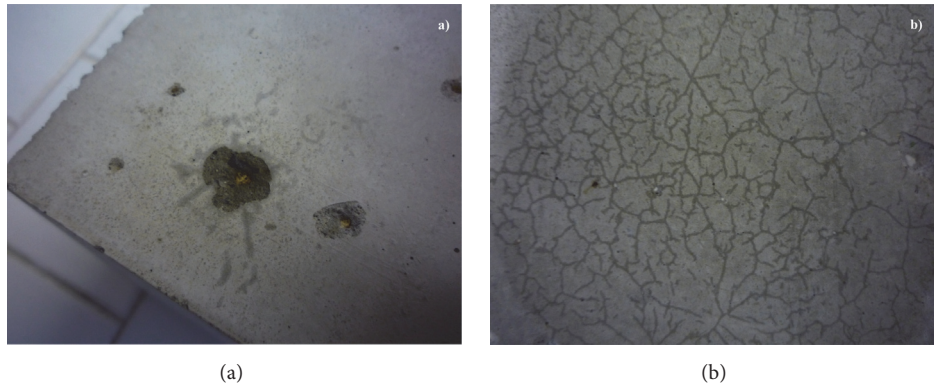


FIGURE 3: Image of the surface of the reference mortar sample after 1 year of ageing in water: (a) spalling; (b) chipping.

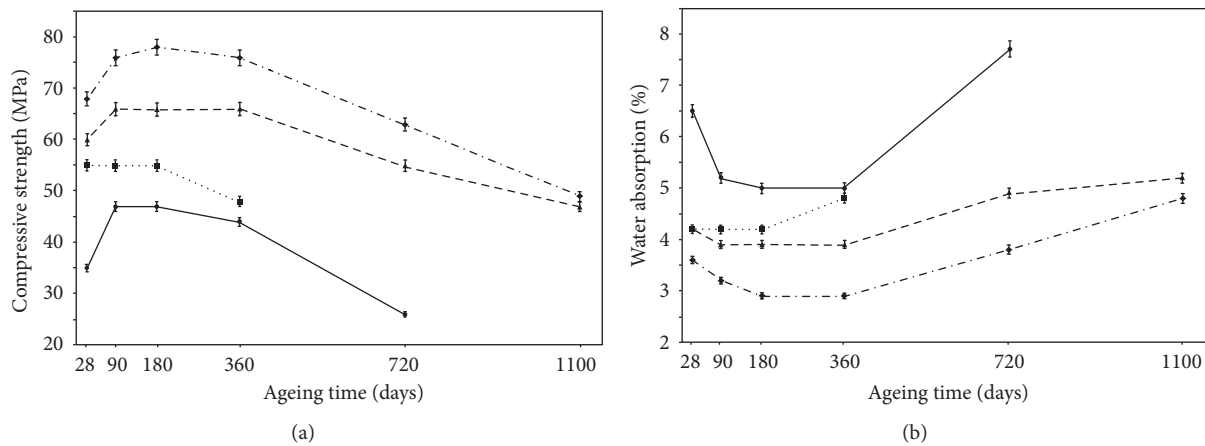


FIGURE 4: Trends of compressive strength (a) and water absorption (b) displayed as a function of curing time. Each composition refers to the following graphic: reference = —; chromium oxide = ---; ultramarine = -.-.-; aluminium silicate (Na) =

almost constant from 180 to 360 d but grows for longer ageing times reaching the values of 3.8 and 4.8 after 720 and 1100 d, respectively.

Materials containing chromium oxide display a strength of 60 MPa after 28 d which raises to 66 after 90; strength remains at this level after 180 and 360 d of ageing but slowly decreases for longer times showing 55 MPa after 720 and 47 after 1100 d, respectively. Water absorption is 4.2 after 28 d, 3.9 between 90 and 360 d, 4.9 after 720 d, and 5.2 after 1100 d.

Expansions of 0.044 and 0.021% were, respectively, measured on reference composition and on that containing aluminium silicate (Na) whereas materials made with ultramarine and green colour showed 0.014 and 0.017, respectively, with all of them being below the 0.05% limit established by the ASTM C33 official norm.

The behaviour of the reference composition is reasonably due to the synergic effect of the above described phenomena, that is, extended fracturing, chipping, and crazing, which cause catastrophic expansion in the samples during long time ageing. Data reported demonstrate that the mortars preparation protocol followed in the present research is not sufficient to favour a complete hydration of the hydraulic components contained into the coarse slag particles.

In materials containing aluminium silicate (Na), the presence of Na favours the ASR which increases the initial residual porosity and, in turn, materials permeability, accelerating, with respect to the reference composition, the catastrophic degradation of the samples during ageing. It follows that the addition of aluminium silicate (Na) does not contribute to materials durability.

Materials containing ultramarine show the highest compressive strength at any time of ageing. Ultramarine and aluminium silicate (Na) have similar composition, their main difference being the presence of S_3^- ions in ultramarine. Such ions are responsible of instability and reactivity of ultramarine blue which is amplified in the mortars production environment. In fact the environmental conditions caused by the chemical reactions during the hydration process are strongly aggressive. In such environment the S_3^- radical anion, containing an unpaired electron [25], is greatly reactive and reasonably accelerate some of the hydration processes between the mortars components during production. The mechanical performances of materials containing ultramarine are therefore reasonably due to two synergic phenomena: the first of chemical origin, which depends on the accelerated reactions between some of the mortars components, namely,

MgO and CaO containing compounds [2, 3, 9, 26, 27], during the early stages of hydration when materials are not yet hardened; and the second of physical nature due to the minimized materials open porosity which limits permeability during ageing. However the presence of Na favours long term ASR which causes a continuous progressive increase of open porosity and reduction of strength. The present results are in agreement with those obtained by other researches which observed a similar behaviour in concrete containing recycled slag [28].

Materials containing chromium oxide display good but not the best properties among all the materials tested in the present research. In fact their absolute performances are inferior to those of the composition containing ultramarine. However, if the ratio between the strength value obtained after 1100 d of ageing and the maximum strength measured (i.e., after 180 d) is evaluated, it can be observed that materials containing chromium oxide reach 0.71 whereas those containing ultramarine show the value of 0.63. It means that the presence of chromium oxide permits to obtain materials that suffer from a lower decay with respect to the others. This result is in line with the one obtained by other authors who demonstrated that the use of chromium oxide tends to mitigate the ASR [29].

Another detail which emerged from the data reported in present research is that all compositions display an inversion of the trend of compression strength and water absorption after 180 d of curing. This inversion is reasonably related to the retarded hydration of some compounds and depends on materials permeability which is function of their open porosity. The time required for permeation falls between 180 and 360 d which therefore depends on the preparation protocol followed and not on materials' composition.

It may be concluded that cement based materials containing coarse steel slag as aggregate can develop good long term compression strength if their permeability is limited. Permeability is promoted by material's residual open porosity which could be limited optimizing preparation. In parallel, the ASR must be limited by reducing the presence of alkalis in the starting components or by the addition of compounds which could limit their negative effects.

4. Concluding Remarks

The present research demonstrates that mortars prepared using a commercial CEMIIB-LL cement, coarse ($\emptyset < 2.5$ mm) steel slag as aggregate, Glenium 51 as superplasticizer, and water display good compression strength after ageing for 28, 90, and 180 d in water at 20°C if prepared following a different protocol route with respect to those normally used for the production of mortars or concrete. However, if aged for longer times, several samples spontaneously crumble during ageing due to some localized catastrophic expansion caused by the retarded hydration of some compounds contained in the starting slag.

A limited addition (3 wt% of the total solid materials content) of further products (i.e., ultramarine blue colour, chromium oxide green colour, and aluminium silicate (Na)) to the above described blank composition showed that reducing

materials residual open porosity and limiting the ASR during ageing lead to optimize long term compressive strength of compositions containing ultramarine and chromium oxide.

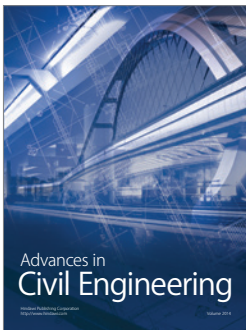
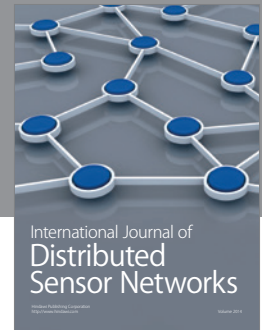
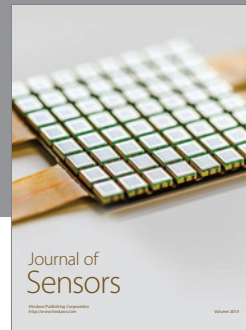
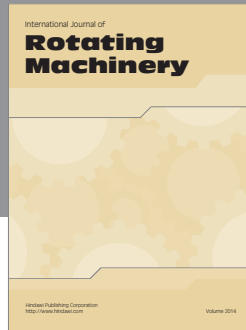
Competing Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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