

Conference Paper

Early Age Compressive Strength of Waste-based-glass-powder Magnesium Silicate Binders on Initial Carbonation Curing

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Abstract

In this preliminary study, the effect of glass powder content at early age compressive strength and its effect at strength retention coefficient during water immersion period on magnesium silicate hydroxide cement pastes on carbonation curing was investigated. A magnesium oxide-rich powder with a maximum grain size of 150 μm was used, as well as, a waste glass powder with a maximum grain size of 250 μm , which was obtained from grinded flint glass bottles. Cement pastes were produced with 0, 10, 20, 30, 40, and 50 glass powder weight percentage. The specimens were compacted into cubic moulds ($e = 20 \text{ mm}$) under 70 MPa and, subsequently, cured on accelerate carbonation chamber for 2h at $>99\%$ CO_2 concentration. The compressive strength was determined 3 days after CO_2 , period which the specimens were preserved on room conditions (20°C and $60\%\text{RH}$), and also at 3, 7 and 14 days of water immersion period. Comparison of the results obtained for different mixing compositions, as well as, different water immersion periods are discussed in this work.

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1. Introduction

Nowadays about 7% of the total CO_2 emissions worldwide comes from the cement industry, and it tends to grow as the economies keeping growing, especially in Asia, since concrete has been the manufactured material most used on planet, in terms of volume, and is expected to increase its demand between 12 and 23% until 2050 [1]. On its production, the biggest carbon footprint comes from the Portland cement (PC) [2], which emits CO_2 directly through its production, indirectly by burning fossil fuels used for its energy process, and also from the chemical transformation of limestone (CaCO_3) into lime (CaO), which such process is responsible for about 50% of cement carbon footprint [3].

Besides that, not only the concerns about the climate changes but also due the need to mitigate the CO_2 emissions linked to the conventional Portland Cement manufacture,

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entailed a growth of popularity on magnesium based cements, leading this category to achieve an expressive popularity over the last decade [4], and also allied to its popularity is the high CO₂ uptake ability (up to 1.09 tonne of CO₂/tonne of MgO), turning magnesium based cements a potential carbon-negative cement, but it depends on the method used for magnesium oxide (MgO) manufacture [5].

In the midst of this, one of the main types of magnesium based cement is the Magnesium Silicate Hydrate (M–S–H) cement, which is produced by the mix of a magnesium source (e.g. MgO) and reactive silica source in presence of water, leading the blend to hydrate, and to form brucite, which then reacts with the dissolved silica and forms M-S-H gel [4–6]. Most of the previous studies reported the use of micro silica [6–9], silica fume [10–12], silica flour [9], and amorphous silica [6] as silica source. On top of that, more environmental friendly sources also have been tested, such as rice husk ash [13] and pulverized fuel ash [14].

However, magnesium oxide-based cements, such as magnesium oxychloride cement (MOC), usually present poor water resistance [4], which property has been entailed researchers to find ways to improve it. Although some eco-friendly solutions have been reported, mainly on MOC cements, which are using not only waste-based silica sources, such as glass powder [15], silica fume [16–17], fly ash [17], pulverized fuel ash [15–18], and sewage sludge ash [19], but also CO₂ curing [18] to improve the water resistance in this cement class.

Therefore, in this preliminary study the effect of the weight proportion of an alternative silica source, glass powder (GP) blended with magnesium oxide-rich powder (MRP) was investigated, where were assessed the effect on the compressive strength and also on strength retention coefficient. For this propose, specimens of different proportions of MRP and GP were moulded and subjected not only to CO₂ curing followed by room conditions period but also part of them to water immersion period for up to 14 days.

The use of GP is justified not only due to its chemical composition, which exhibits around 70% of silica [20–22], and its high pozzolanic properties [23], but also due to the municipal solid waste production, which since 2012 had growth around 55% and nowadays represents 5% of global CO₂ emissions, is also associated to climate changes [24–25]. Following similar growth rhythm, the use of glass has also increased around the world as well as the amount of waste glass, which nowadays represents 5% of global municipal solid waste composition [25], where the main kind of manufactured glass is the container glass (e.g. glass bottle), which represents around 62% of glass production, and recent data reveal the annual production of container glass has reached 22.32 million tonnes only in EU-28 [26], meanwhile 31% of its production are deposited

in landfills [27], what means 6.9 million tonnes per year. Moreover each six tonnes of glass powder concrete could result in one tonne of CO₂ reduction from cement Portland production [20].

2. Materials and Methods

2.1. Materials

The MRP, commercial name “Magal P”, was obtained from Invivo nSA, which is mainly used as animal feed additive. In order to obtain MRP with a maximum grain size of 150 µm, the material received was grinded into a ball mill grinder and sieved.

The GP was obtained by grinding flint glass bottles, which were collected from garbage. For this purpose, the bottles were washed in tap water not only to clean it but also to remove the paper labels from the external glass surface. Afterwards, the bottles were grinded into a ball mill grinder and sieved in order to obtain glass powder with a maximum grain size of 250 µm. Moreover, as an effort to obtain a standard powder, all grinded bottles collected are from the same supplier.

The MRP and GP chemical compositions are presented in Table 1, which were determined by Energy-dispersive X-ray spectroscopy (SEM-EDX) tests on a Hitachi S-3400N apparatus, where three different spots randomly were selected in each sample to obtain the chemical compositions of the raw materials.

TABLE 1: Chemical composition.

Component	Content (%)	
	MRP	GP
Silica (SiO ₂)	6.36	68.62
Alumina (Al ₂ O ₃)	1.38	2.61
Iron oxide (Fe ₂ O ₃)	3.12	1.65
Calcium oxide (CaO)	3.36	12.03
Magnesium oxide (MgO)	85.78	-
Potassium oxide (K ₂ O)	-	0.76
Sodium oxide (Na ₂ O)	-	14.33

2.2. Mix composition

The mix compositions of all prepared pastes are detailed in Table 2. The weight percentage (wt. %) of MRP and GP varied from 100% to 50% and from 0% to 50% respectively, by contrast, the water to solid (w/s) ratio was kept constant in 0.21.

TABLE 2: Mix compositions.

Mix label	Weight (%)	
	MRP	GP
GP0C	100	0
GP10C	90	10
GP20C	80	20
GP30C	70	30
GP40C	60	40
GP50C	50	50

2.3. Samples preparation

The solids (MRP and GP) were mixed to result on a homogeneous powder, soon after, tap water was gradually added until has been obtained a homogenous blend, then the prepared paste was compacted into 20 x 20 x 20 mm cubic moulds under 70 MPa of static compaction pressure. During this stage, part of the water present in the mix is expelled.

Thereafter, the specimens were subjected to CO₂ curing in a pressurised carbonation chamber for two hours under an atmosphere with CO₂ concentration of > 99%, at partial pressure of 1 bar, and at a temperature of 60°C. As it is widely known, 60°C is an excellent temperature to accelerate chemical reactions, besides this, the partial pressure of 1 bar is strong enough to allow the CO₂ to access the specimen's interior.

Fig. 1 shown a specimen of GP30C after two hours of CO₂ curing, already hardened.

After CO₂ curing, the specimens were preserved on room conditions (temperature of 20°C and relative humidity near 60%) for three days. Then, part of the specimens has been submitted to compressive strength test, meanwhile the other part has been immersed on water period at 20°C for up to 14 days, until has been tested.

2.4. Compressive strength

The compressive strength of the prepared specimens was measured by uniaxial loading in triplicates. The tests occurred at 3rd day after CO₂ curing and at 3rd, 7th, and 14th day during water immersion period. The equipment used for this purpose was a 3000 KN electro-hydraulic mechanical testing machine “ADR Touch 3000 BS EN Compression Machine with Digital Readout and Self Centring Platens”, in accordance with EN 196-1, operated at a loading rate of 0.5 kN/sec.



Figure 1: A cubic specimen of GP30C soon after CO₂ curing.

2.5. Strength retention coefficient

To evaluate the specimens water resistance, the strength retention coefficient (SRC) was calculated by the Eq. (1). Where CS_{H2O} is the compressive strength of specimens immersing in tap water up to 14 days and CS_{CO2} is the compressive strength of specimens three days after CO₂ curing.

$$SRC = CS_{H2O} / CS_{CO2} \quad (1)$$

3. Results and Discussion

3.1. Compressive strength

The compressive strength of all specimens is shown in Fig. 4, which exhibits the comparison of compressive strength on different blends composition.

The specimens cured on CO₂ atmosphere at 60°C temperature for two hours, achieved compressive strengths up to 31.8 MPa, where the best compressive strength belongs to the specimen GP30C.

The specimen GP0C suggests the MRP reacts with the water present in the mix and also with the CO₂ dissolved in the water. Such mix label has reached 25.3 MPa after only two hours on CO₂ curing and 3 days in room conditions. Such behaviour was expected, since the MgO reacts with water to form brucite (Mg(OH)₂) Eq. (2), which reacts with the CO₂ dissolved in the water to form hydrated magnesium carbonates (HMCs), such as nesquehonite (MgCO₃ · 3H₂O) Eq. (3) [28].



Was observed there are a gain of strength insofar as the GP wt. % increases, growing from on 25.3 MPa (GP0C) to 31.8 MPa (GP30C), then after GP wt. 30%, the compressive strength starts decreasing until reach 25.1 MPa (GP50C). Such behaviour might be linked to the effect of particle packing between MRP and GP; i.e. due to different particle size distribution between MRP and GP the best packing density might for GP30C specimen, reason why it shows the highest compressive strength. Apparently, it exists an optimal value of GP for the highest compressive strength, but is not possible to justify such behaviour without a microstructural analyse, since it can be only related with the volumetric proportion between MRP (≤ 150 μm) and GP (≤ 250 μm), because of it, is assumed that all specimens have reacted with the CO₂ (Eqs. 2-3) in the same manner.

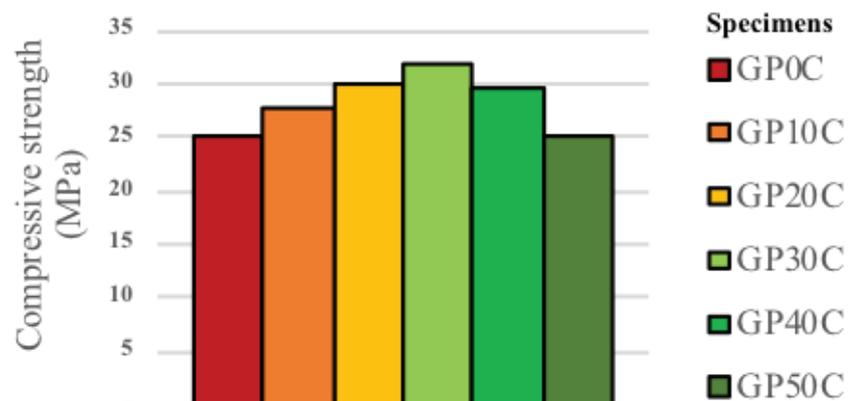


Figure 2: compressive strength on different blends composition.

3.2. Strength retention coefficient

The compressive strength of all the specimens during water immersion period are shown on the Fig. 3 and Fig. 4, which present, respectively, the comparison of compressive strength versus blends composition on different water immersion ages and the comparison of compressive strength versus water immersion age on different blends composition.

The Fig. 5 and Fig. 6 summarize, respectively, the comparison of strength retention coefficient versus blends composition on different water immersion ages comparison of strength retention coefficient versus water immersion age on different blends composition.

As has happened in other works [15–19], the water immersion period of magnesium oxide-based cement resulted in the loss of compressive strength.

Specimens submitted to water immersion period up to 14 days achieved 66.1% as the minimum strength retention coefficient. The lowest result belongs to the specimen GP20C on the 7th day of the water immersion period. The specimen that achieved the best strength retention coefficient result was the GP50C, which has held 99.7% of its initial compressive strength on the water immersion period of 14 days. The specimen that remained with the lowest strength retention coefficient on the water immersion period of 14 days was the GP30C, which has held 76.6% of its initial compressive strength. The specimen that remained with the highest compressive strength on the water immersion period of 14 days was the GP10C, which reached 26.4 MPa.

As has been seen in other works [15–18–19], during the first seven days of the water immersion period, the weak bonds of HMC are broken in contact with the surrounded water, by the same time occurs the reaction of uncarbonated and free MgO with the water, thus forming magnesium hydroxide, reasons why the compressive strength of all prepared pastes decrease. Soon after the 7th day, is expected that the silica present in the mix starts reacting with the system and the M-S-H gels start being formed.

In this preliminary study, it is not possible to conclude clearly the relation between the gain of compressive strength after the 7th day of water immersion period and the silica content, since specimens with low (GP0C), as well as, with high silica content (GP50C) have presented a gain of compressive strength. The SRC after the 7th day of water immersion period decrease insofar the GP content increases, until reach 20%, after this point the SRC starts increasing and reaches 99.7% on GP50C. Therefore, it might indicate that both silica and alumina may react during the water immersion period, forming different kinds of gels, such as M-S-H gel. Such gels are denser and should

be the responsible for the compressive strength gain after the 7th day of the water immersion period. However, further studies on microscopy level are needed to fully understand the results obtained.

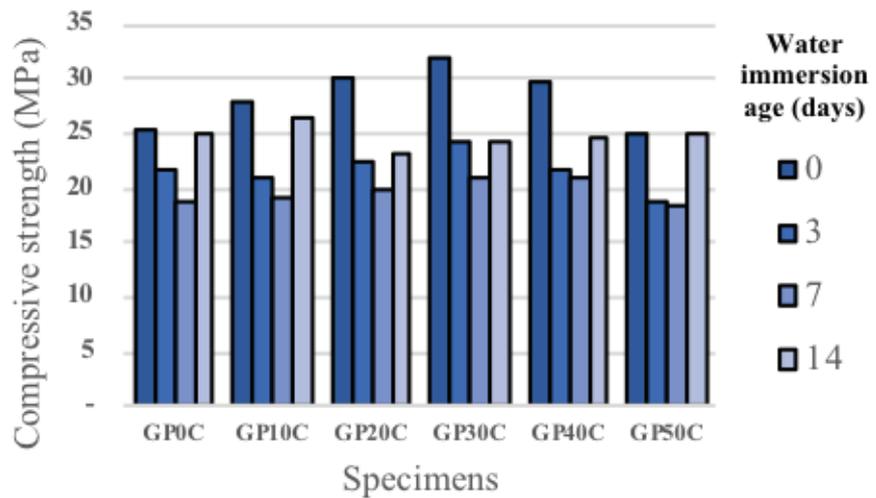


Figure 3: Comparison of compressive strength versus blends composition on different water immersion ages.

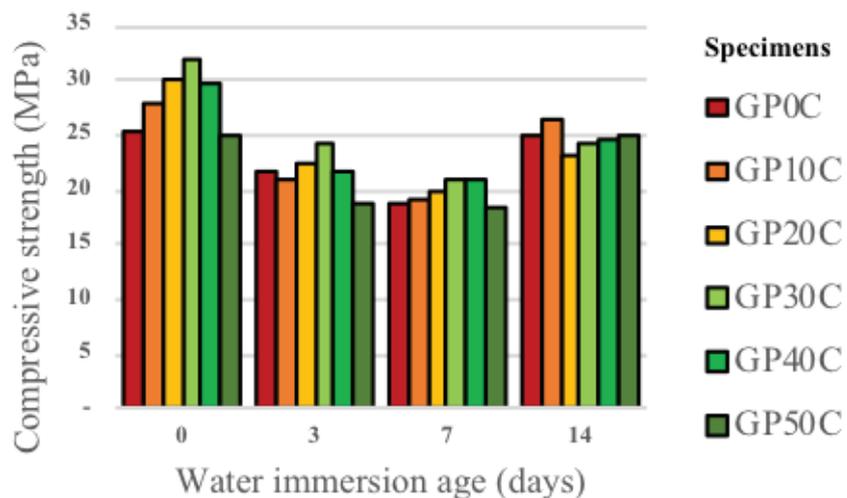


Figure 4: Comparison of compressive strength versus water immersion age on different blends composition.

4. Conclusions and Future Studies

This preliminary study has successfully utilized a magnesium oxide-rich powder (MRP) and waste-based silica source (GP), to produce waste-based-glass-powder magnesium silicate hydroxide binders on CO₂ curing followed by a water immersion period, which

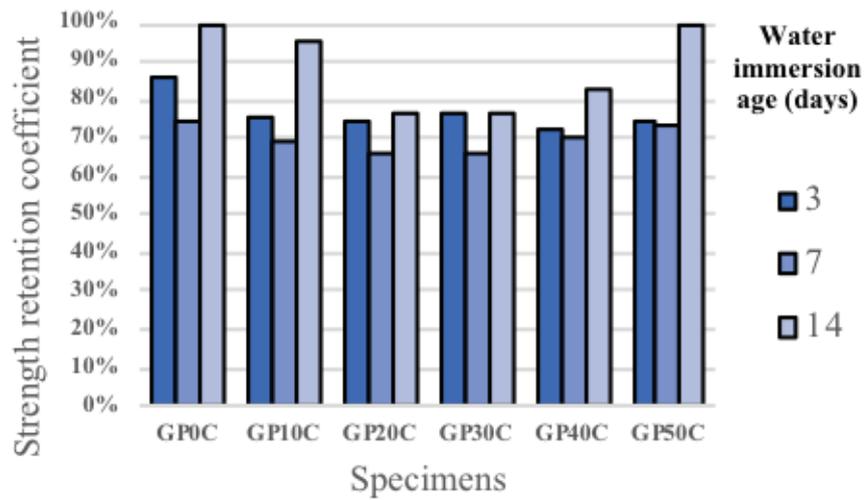


Figure 5: Comparison of strength retention coefficient versus blends composition on different water immersion ages.

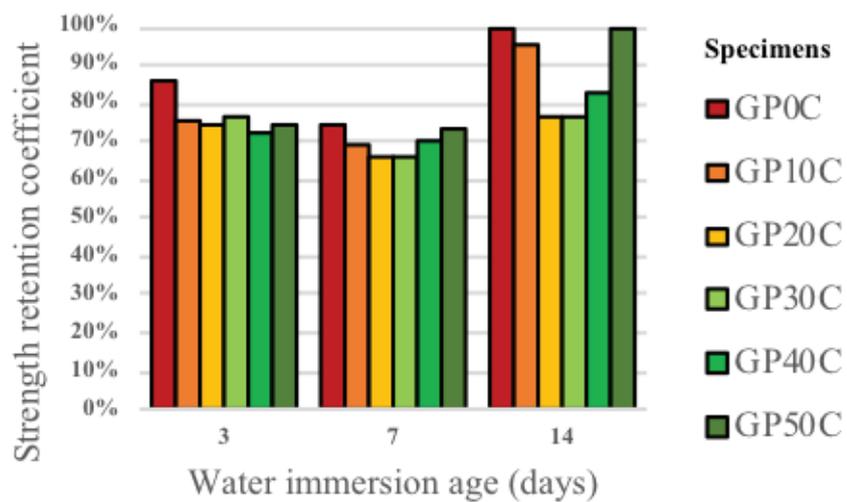


Figure 6: Comparison of strength retention coefficient versus water immersion age on different blends composition.

compressive strength has been evaluated during the process stages, this way allowing to calculate the strength retention coefficient. Therefore, the results of this study are summarized below:

- Initial 2 hours CO₂ curing reveals beneficial properties, since all of the specimens achieved more than 25 MPa of compressive strength;
- The highest compressive strength was achieved on the specimen GP30C, which reached 31.8 MPa, after initial CO₂ curing;

- Since every specimens were submitted to the same compaction pressure it can be assumed that GP30C presents better initial compressive strength, due to different particle size distribution between MRP and GP which resulted in best packing density;
- The highest strength retention coefficient was achieved on the specimen GP50C, which held 99.7% after the 14th day of water immersion period;
- The highest compressive strength after the 14th day of water immersion period was achieved on the specimen GP10, which reached 23.1 MPa;
- Initial CO₂ curing may have not generated stable hydrated magnesium carbonates, which convert to brucite [19] when the specimens were submitted to the water immersion period, mainly on the first seven days, the reason why occurs the loss of compressive strength up to the 7th day of the water immersion period;
- Over the water immersion period, mainly after the 7th day, the silica may react with the system to form denser phases of M-S-H gels, which are more stable, resulting in compressive strength gain on every mix label.

However, future studies are needed to understand better the behaviour of the binders produced in this preliminary study.

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