INTERNATIONAL JOURNAL OF INTEGRATED ENGINEERING
ISSN: 2229-838X e-ISSN: 2600-7916

IJIE

Vol. 17 No. 5 (2025) 241-255
https://publisher.uthm.edu.my/ojs/index.php/ijie

Study of the Durability Against Acid Sulfuric Attack and Accelerated Carbonation of Eco-Efficient Self-Compacting Concrete Blended with Marble Powder

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DOI: https://doi.org/10.30880/ijie.2025.17.05.019

Article Info

Received: 10 June 2024
Accepted: 25 April 2025
Available online: 30 August 2025

Keywords

Self-compacting concrete, marble powder, durability, acid attack, carbonation

Abstract

Reinforced concrete structures can suffer from numerous problems such as the aggressivity of the environment and the carbonation phenomena. The introduction of fine industrial by-products (FIB) can reduce the permeability and hence improve the concrete durability. Marble industry generates high quantity of fine residue (marble powder) that can be used as FIB to produce an eco-cement. In this concern, five self-compacting concrete (SCC) mixtures were prepared using a constant water to binder ratio of 0.40, one as reference mix and four containing marble powder as a partial replacement of ordinary Portland cement (OPC). The durability of SCC subjected to sulfuric acid attack and accelerated carbonation were assessed. X-Ray diffraction analysis (XRD) were carried out to identify the mineralogical composition before and after durability tests. The results have shown that the use of marble powder is beneficial to improve the resistance of SCC to acid sulfuric attack. However, the incorporation of marble powder seems to accelerate the carbonation of SCC specimens.

1. Introduction

Durability is the main parameter that control the service life of reinforced concrete structures. Reinforced concrete durability is strongly affected by numerous phenomena such as the aggressivity of the environment (sulfate and acid attacks) and the carbonation. In presence of sulfate, cement hydrated products (calcium hydroxide) react with sulfate ions to produce expansive and aggressive elements (gypsum and secondary ettringite). This fills in first pores and micro pores and contributes to strength gain. However, the formation of aggressive elements resulted at long-term in cracks and deterioration in cement matrix which reduced the service life of the concrete. Makhloufi et al. [1] stated that the action of acid solutions (sulfuric, nitric or hydrochloric) on concretes and mortars is quick and more dangerous in comparison to sulfate solutions. It was reported that calcium hydroxide is the most vulnerable product in case of acid attacks [2]. In fact, acid solution penetrates via the pore system of concrete to react with calcium hydroxide (eq. 1) and producing therefore an aggressive and expansive product (gypsum), which in turn reacts with calcium aluminate hydrate (C_3A) to form the secondary ettringite (eq. 2). The volume of the formed products is 2-7 times higher than the initial compounds. This increases the internal pressure and resulted in cracks and spalling, hence reducing the durability of the concrete [3]. The introduction of fine materials (metakaolin, pozzolana, fly ash, silica fume) seems to be a good solution to enhance the resistance to acid attack, because they chemically consume calcium hydroxide and lead to the formation of new generation of C-S-H, this densifies the cement matrix and improves therefore the resistance to external sulfate and acid attacks [4]–[7]. The use of rice husk ash as a partial replacement to OPC in concrete offered superior resistance against acid attack in comparison to concrete without addition [8]. Senhadji et al. [4] stated that mortar

with limestone fine showed superior performance against acid attacks in comparison to the respective mortar and mortar containing silica fume.

$$Ca(OH)_2 + H_2SO_4 \rightarrow CaSO_4.2H_2O \quad (1)$$

$$3CaO.Al_2O_3.12H_2O + 3(CaSO_4.2H_2O) + 14H_2O \rightarrow 3CaO.Al_2O_3.3CaSO_4.32H_2O \quad (2)$$

Concrete carbonation is the result of an electrochemical reaction between carbon dioxide present in the atmosphere with moisture and hydrated products that are present in cement paste. It starts when carbon dioxide penetrates throw the surface porosity and reacts in the presence of moisture with calcium hydroxide and calcium silicate hydrated to produce calcium carbonate ($CaCO_3$) [9], [10]. At the first time, calcium carbonate increases the compressive strength of concrete by clogging the pore system, but at later ages it lowers the alkalinity of concrete (pH) from 12-13 to around 9, thus the protective passivation layer surrounding the reinforcing steel begins breaks down, leaving the steel vulnerable to corrosion, which will considerably affect the structural integrity and the bearing capacity of the reinforced concrete elements [11]–[15]. Carbonation is controlled by four factors: (i) carbon dioxide concentration, (ii) permeability of the material, (iii) moisture content and (iv) relative humidity. The process of carbonation can occur even under low concentration of carbon dioxide (0.03% in rural regions). In urban regions, this concentration can reach 1%. Concrete carbonation can advance at a rate of 1 mm to 5 mm per year, dependent on the pore sizes and their connectivity. The impact of carbonation on concrete durability may be reduced by increasing its compactness or using protective anti-carbonation coating system. The most effective way to decrease the porosity of concrete is the incorporation of fine inert materials (FIM).

Fine industrial by-products (FIB) such as silica fume (SF), ground granulated blast furnace slag (BFS), fly ash (FA), marble powder, brick powder have been widely used in self-compacting concrete production as a partial replacement of ordinary Portland cement (OPC) [16]–[20]. One of the primary reasons for the use of FIB in concrete production is to reduce the environmental impacts by reducing cement production and therefore its cost [21]. The filling effect of FIM plays an important role in reducing the concrete porosity. In contrary, fine active or pozzolanic materials have an adverse effect in which the C-H is partially consumed to produce secondary C-S-H, resulting in low content of C-H in the cement paste. In this case, a smaller quantity of CO_2 is sufficient to react with all of C-H, and as a result, the pH drops more quickly.

The resistance to carbonation of concrete made with slag was found lower than corresponding Portland cement concrete [22]. In previous work, Ho and Lewis [23] reported that concrete including fly ash carbonated faster than control concrete. In contrary, Zhang and Li [24] have shown a considerable decrease for the carbonation depth of the concrete by increasing the content of silica fume. They noted a decrease in carbonation depth by 17 and 30% for 6 and 12% silica fume content, respectively. The authors have attributed this finding to the grain size of silica fume particle which is smaller than that of the cement, and as a result, the silica fume particles fill the interspaces around the cement particles to make the hardened concrete much more compact. Therefore, it is difficult for the CO_2 in the atmosphere to permeate into concrete.

Although the influence of fine industrial by-products on the carbonation and acid sulfuric attack of ordinary concrete have been well examined, there is a lack of studies on the durability against carbonation of self-compacting concrete including fine industrial by-products. This paper aims to study the effect of partial replacement of cement by marble powder on the durability against acid sulfuric attack and under accelerated carbonation. Marble powder was incorporated with four percentages 5, 10, 15 and 20% by mass of cement. The study of carbonation of marble powder SCC is recent and the results obtained in this work will contribute to the understanding of carbonation in SCC including binary blended cement.

2. Materials and Methods

2.1 Raw Materials

Ordinary Portland cement (CEMI, 42.5) that complies with the European Standard EN 197-1 was used to produce various SCC mixtures. The marble powder (MP) is a by-product of marble stone sawing, shaping, and lustration. The chemical composition and physical properties of cement and MP are presented in Table 1. Laser distribution analysis was performed on OPC and marble powder to determine their particle size distribution (Fig. 1). The results revealed that MP is finer than OPC. Table 1 gives the diameter of particles that corresponds to 10%, 50% and 90% of passing (Fig. 1). The results showed that almost 50% of the MP particles are smaller than 6.5 µm, and about 50% of the cement particles are smaller than 12.35 µm. Fig. 2 shows the results of the Scanning Electron Microscopy (SEM) test on OPC and MP. It can be seen from Fig. 2 that the particles of marble powder are finer and appear to have a less angular shape in comparison to cement particles. The mineralogical analysis of MP shown in Fig. 3 indicates that it consists mainly of calcite, and with some traces of quartz and dolomite. Natural river sand with a maximum size of 5 mm was used as a fine aggregate. Two classes of coarse aggregates with a maximum

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size of 15 mm were used, the physical properties of these aggregates are given in Table 2. To obtain desired fluidity of SCC, a polycarboxylate-based superplasticizer was used as chemical admixture; it has a specific gravity and pH of 1.07 g/cm^3 and 8, respectively.

Table 1 *Chemical composition and physical properties of cement and MP*

Chemical composition (%)		OPC	MP
SiO_2		20.14	0.42
CaO		63.47	56.01
MgO		2.12	0.12
Al_2O_3		3.71	0.13
Fe_2O_3		4.74	0.06
SO_3		2.67	0.01
K_2O		0.47	0.01
T_iO_2		0.21	0.01
Na_2O		0.69	0.43
P_2O_5		0.06	0.03
Loss ignition		1.72	42.78
Physical properties			
Specific gravity (g/cm^3)		3.1	2.7
Fineness characteristics	Fineness (m^2/kg)	330	360
	d_{10} (µm)	1.19	1.36
	d_{50} (µm)	12.35	6.50
	d_{90} (µm)	40.53	21.09

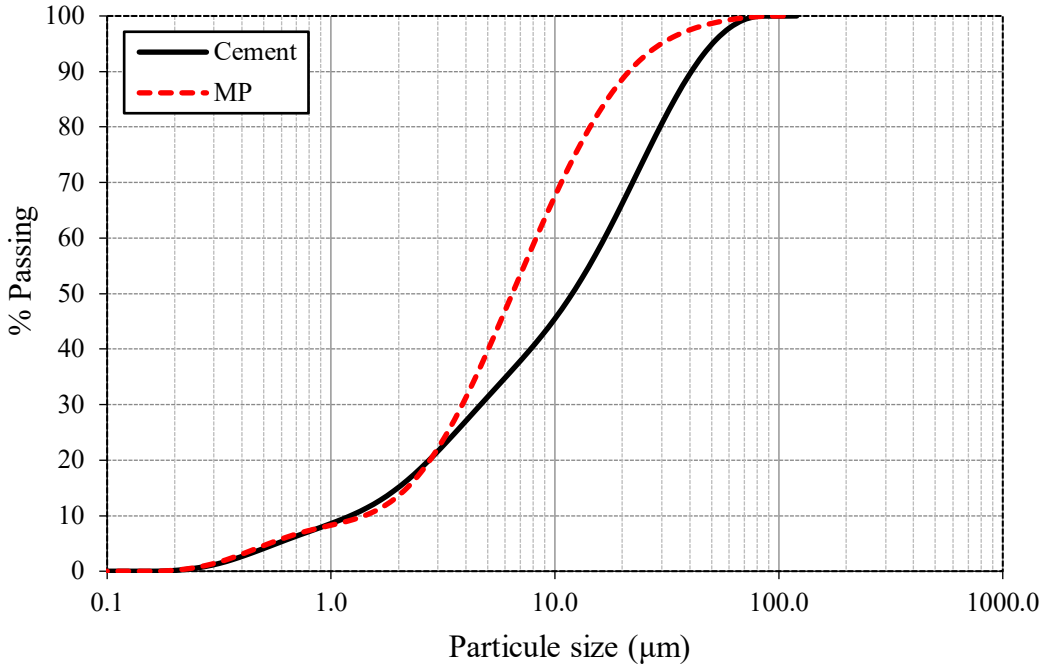
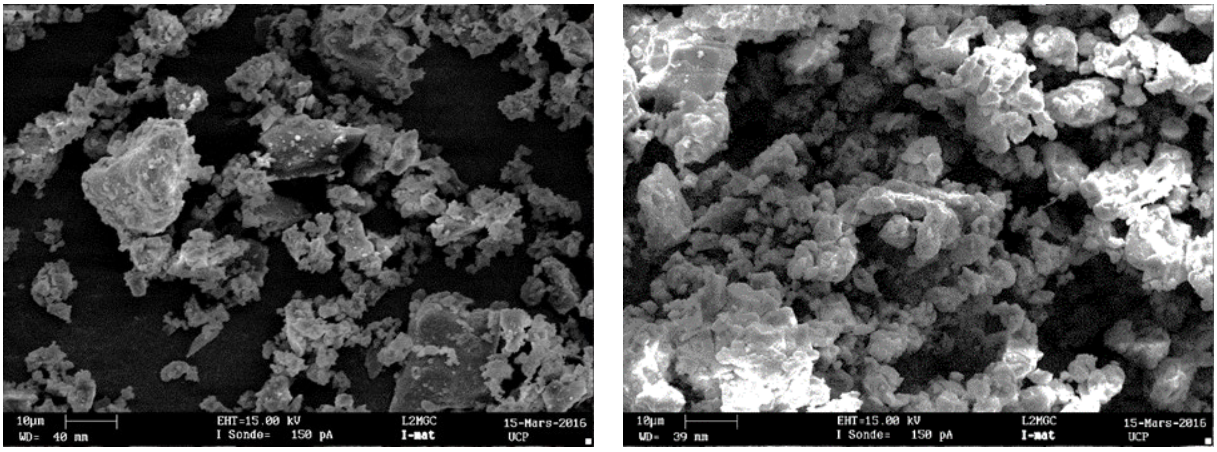


Fig. 1 *Particle size distribution of cement and MP*



(a) **(b)**

Fig. 2 *Particle's shape of (a) Cement; and (b) Marble powder*

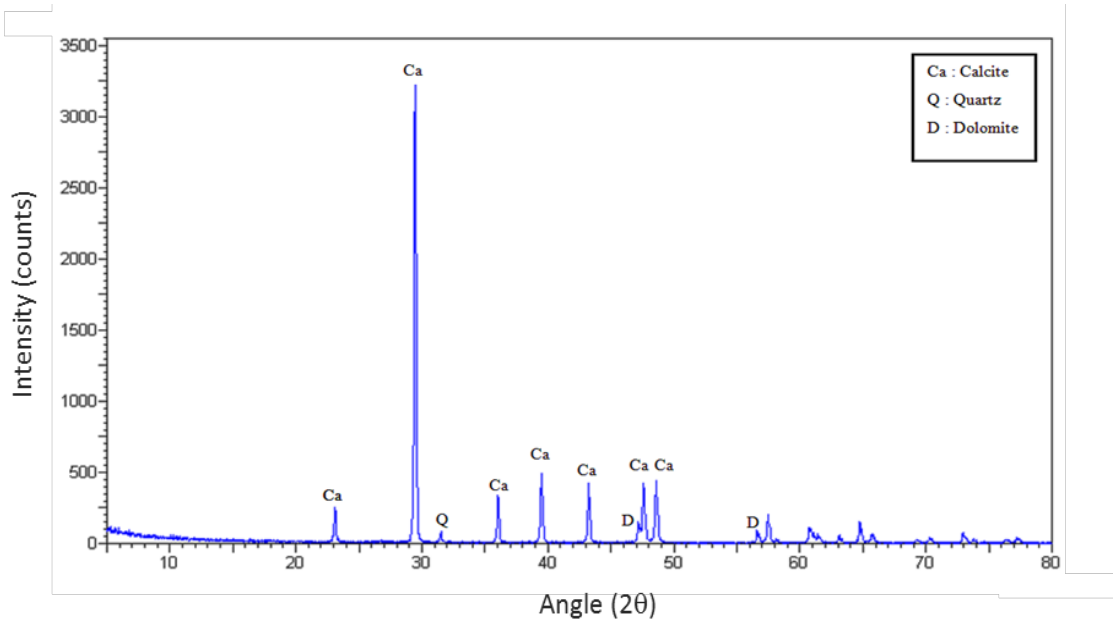


Fig. 3 *XRD analysis of marble powder*

Table 2 *Physical properties of aggregates*

Properties	Sand 0/5	Gravel 3/8	Gravel 8/15
Absorption coefficient (%)	0.60	1.50	2.30
Specific gravity (g/cm^3)	2.60	2.70	2.70
Apparent density (g/cm^3)	1.58	1.32	1.30

2.2 Mix Design Proportions

For investigating the effect that marble powder on some durability properties of SCC, five mixtures were prepared with water to binder (w/b) ratio of 0.4 and a total binder content of 470 kg/m^3. The control mixture only included OPC as binder, whereas in the other mixtures OPC was partially substituted by marble powder at ratios of 5, 10, 15 and 20%. To ensure the desired fluidity of all mixtures, the superplasticizer was used at a dosage of 0.9% by mass of binder. The proportions of all mixtures are shown in Table 3.

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Table 3 *Mix proportions of SCC*

Materials (kg/m³)		SCC Mixes				
		0% MP	5% MP	10% MP	15% MP	20% MP
OPC		470	446.5	423	399.5	376
Marble powder	(%)	0	5	10	15	20
	(kg)	0	23.5	47	70.5	94
Sand 0/5				882.05		
Gravel 8/15				540.61		
Gravel 3/8				272.52		
Water				208.23		
Superplasticizer				4.25		

2.3 Preparation of Specimens and Curing

Prismatic specimens of 40×40×160 mm and 70×70×280 mm dimensions were used to realize the durability tests: resistance to acid attack (H_2SO_4) and accelerated carbonation [25], [26], respectively. All specimens were cast in one layer without any compaction. After 24 h, specimens were demolded and stored in water at 21±2°C for 28 days.

2.4 Test Methods

2.4.1 Degradation Test in Sulfuric Acid

To assess the resistance to acid attack (H_2SO_4), prismatic specimens (40×40×160 mm) were immersed in an aggressive sulfuric acid solution with an initial concentration of 3% for 336 days (Fig. 4). the solution was renewed every 15 days with a new solution with the same initial concentration (3%). Samples were extracted weekly, rinsed three times with tap water to remove the bulk reaction products and left to dry for 30 min before weighing and visual inspection. The resistance to acid sulfuric attack was evaluated physically by measuring the variation in mass (eq. 3). The mineralogical compositions of the samples before and after acid sulfuric attack was studied by X-Ray diffraction analysis (XRD). In addition, visual inspection of the exterior surface appearances was performed on selected specimens.

$$\text{Mass variation } (\%) = \frac{M_1 - M_i}{M_1} \times 100 \quad (3)$$

where M_1 is the average mass of specimens before exposure to acid solution (g) and M_i is the average mass of specimens after i duration of exposure to acid solution (g).

Fig. 4 *Immersion of specimens in sulfuric acid solution*

2.4.2 Accelerated Carbonation Test

The accelerated carbonation test was carried out in a carbonation chamber using prismatic specimens of 70×70×280 mm in sizes. After 28 days of curing in water, the specimens were dried in an oven, and then their surfaces were protected by an adhesive aluminum paper in order to have only two surfaces in contact with the chamber conditions and ensure unidirectional CO_2 diffusion (Fig. 5) [26]. The carbonation chamber conditions were kept as following: the CO_2 concentration was maintained at 50% with relative humidity of 65% and temperature at 20± 2°C (Fig. 6). Three samples for each mixture were used to measure the evolution of the mass during the test and the carbonation depth at 7, 14, 28, 42 and 56 days. The carbonation depth was determined by the phenolphthalein method, in which the specimens are divided in two and the fractured surfaces are sprayed with phenolphthalein solution (Fig. 7). Carbonation depth is the distance between the exterior surface of the concrete and the end of the colored zone. In addition, an X-ray diffraction analysis was carried out to examine possible modification in the mineralogical composition of selected specimens before and after carbonation.

Fig. 5 *Preparation of specimens for accelerated carbonation test*

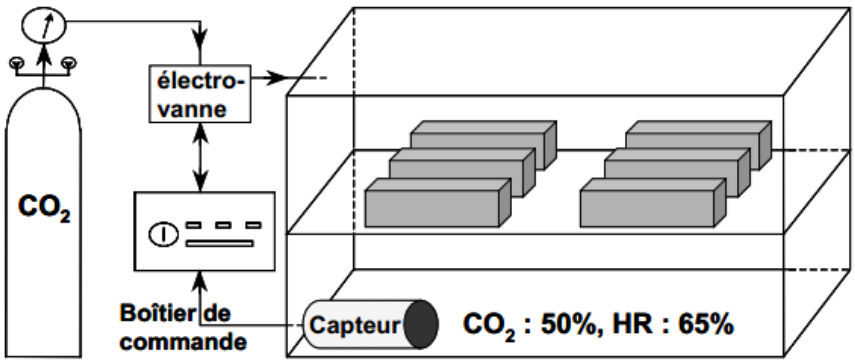

Fig. 6 *Test conditions in carbonation chambre*

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Fig. 7 *Evaluation of carbonation depth*

3. Test Results and Discussion

3.1 Sulfuric Acid Attack (H_2SO_4)

3.1.1 Mass Variation

The mass variation of specimens immersed in H_2SO_4 is shown in Fig. 8. The results show a reduction in mass with increasing immersion time for all mixtures. The mass loss after 336 days of immersion is ranged from 31% to 47%. The acid resistance test revealed that, the maximum mass loss was observed in the control mixture at all ages. The mass loss of plain mixture cement after 336 days was 47%, however mixture including 20% of MP presented the lowest value of mass loss with 31%. It was observed that MP ratio of 5 and 10% showed a slight reduction in mass loss in comparison to corresponding plain cement mixture, while MP content of 15 and 20% developed better resistance than other mixtures. In fact, using MP with 5%, 10%, 15% and 20% reduced the mass loss by about 1.14%, 3.47%, 19.67% and 33.70, respectively. The high values of mass loss are due to the destructive mechanism of sulfuric acid which leads to the formation of voluminous and aggressive products (gypsum and secondary ettringite). Furthermore, the severe acid aggression on plain cement specimens is attributed to the high content of hydrated cement in calcium hydroxide, tricalcium aluminate, calcium sulfoaluminate and calcium silicate hydrate (C-S-H) which are very vulnerable to acid attack [27]. This results in an expansion of the specimens and causes cracking and spalling leading thereafter to a weak cement matrix. The introduction of MP reduced the content of cement hydration products and as consequent the acid aggression becomes quite weak.

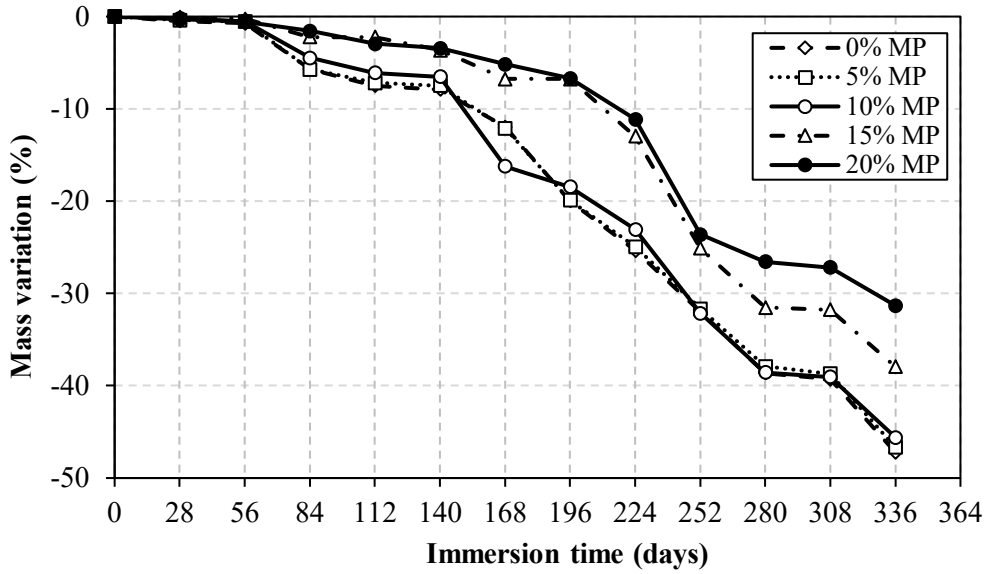

Fig. 8 *Mass variation of mortars exposed to H_2SO_4 solution*

3.1.2 Visual Inspection

Visual appearance of some selecting mortar specimens was carried out after 336 days of immersion in the sulfuric acid solution to evaluate the visible signs of softening, cracking and spalling in the mortar specimens, the results are shown in Fig. 9. A white color layer was noted to form on the surface of various mixture specimens. It is clearly shown in Fig. 8 that all mixture specimens showed a deterioration of the surface of mortar layer. The exterior specimens' surfaces become so soft especially for mixture without MP. This may be explained by the mechanism of degradation of sulfuric acid which takes place by leaching cement paste on exposed faces, since mixture without MP includes high content of portlandite [28]. The use of MP contributes to reduce the damages at the surface appearance. The reduction in deterioration for mixtures with MP is attributed to the decrease in portlandite content since the cement is partially substituted by MP.

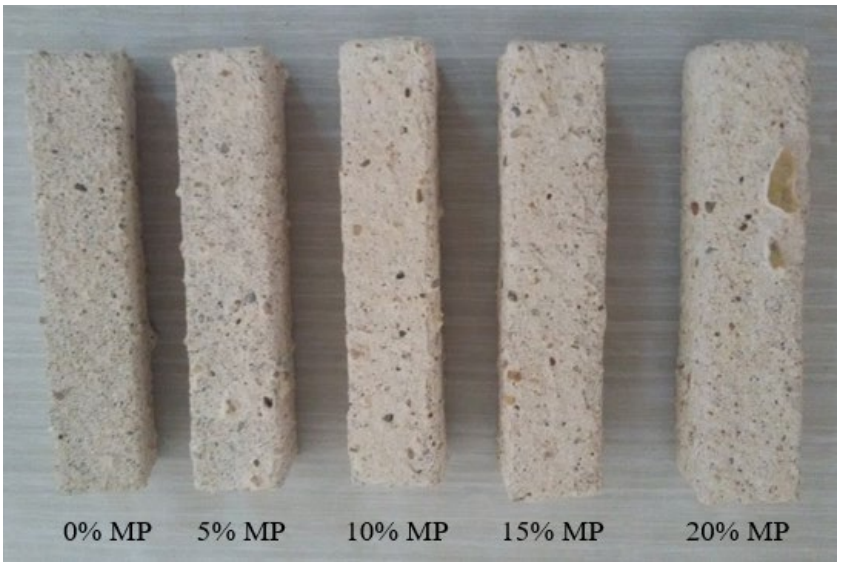

Fig. 9 *Visual control of specimens after 336 days of immersion in H_2SO_4 solution*

3.1.3 X-ray Diffraction Analysis:

For this study, small bits were extracted from the samples surface that were in direct contact with the H_2SO_4 solution during 336 days and were milled until obtaining fine powder (<80 μm). The latter was exposed to X-rays to get an idea about new formed products by comparing the results with corresponding specimens only cured in

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water for 28 days. The various mineral phases found in the powder of mortar specimens kept in both solutions are given in Fig. 10 and 11. As It's seen in Fig. 8, the primary ettringite and portlandite are presents in all mortars and this is the result of the cement hydration. By comparing the results of the two figures, it can be seen the formation of new products (gypsum and secondary ettringite) in specimens exposed to acid sulfuric solution. It was observed a decrease of the amount of the portlandite associated to the apparition of gypsum and secondary ettringite, this may be due to the partial consummation of portlandite to form expansive and aggressive products (gypsum and secondary ettringite).

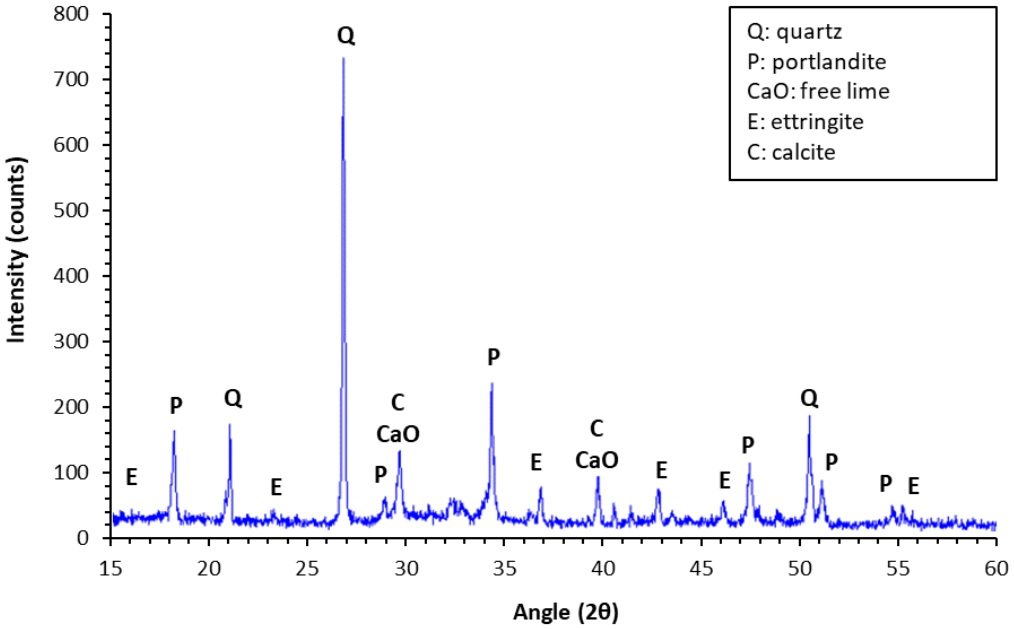

Fig. 10 *X-ray diffraction of specimens conserved in water for 28 days*

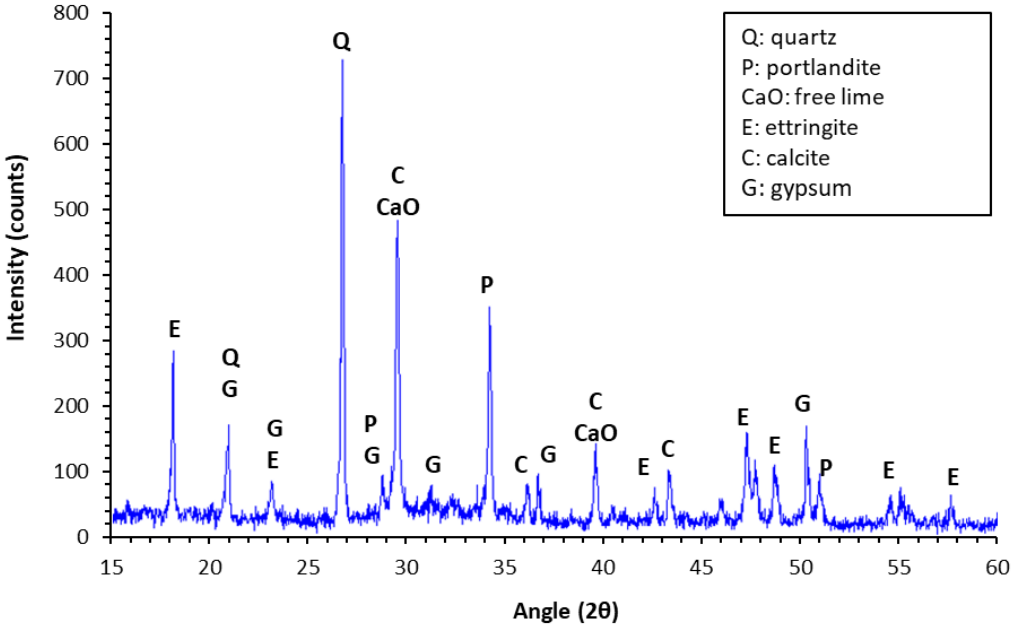

Fig. 11 *X-ray diffraction of specimens exposed to H_2SO_4 solution for 336 days*

3.2 Accelerated Carbonation

3.2.1 Carbonation Depth

Fig. 12 presents the evolution of carbonation depth as function of time for all mixtures. According to Fig. 12 the substitution of the cement by the MP increases the depth of carbonation. It was noted that the kinetic of carbonation in the first 7 days was higher for mixtures made with MP, whereas mixture with only cement showed no kinetic of carbonation. After that, the kinetic of carbonation considerably increased for all mixtures. The values of carbonation depth at 56 days are ranged between 8 and 15 mm. We can see that the reference mixture has the lowest carbonation depth value, which may be due to their dense structure which delayed or stopped the progression of carbonation from exterior faces. However, adding MP with 5%, 10%, 15% and 20% increased the carbonation depth by about 25%, 50%, 68.8% and 87.5%. According to previous work of Boukhelkhal et al. [29], it was reported that water capillary absorption and water absorption by immersion increased when cement is partially replaced by MP, this confirms our results because the increase in water absorption reflects the presence of voids in concrete microstructure. Leemann and Moro [30] reported that the resistance to carbonation of concretes including various types of mineral additions (limestone, ground granulated blast-furnace slag, fly ash and microsilica) was lower in comparison to Portland cement concrete. Similar findings were reported by Ikotun et al. [31].

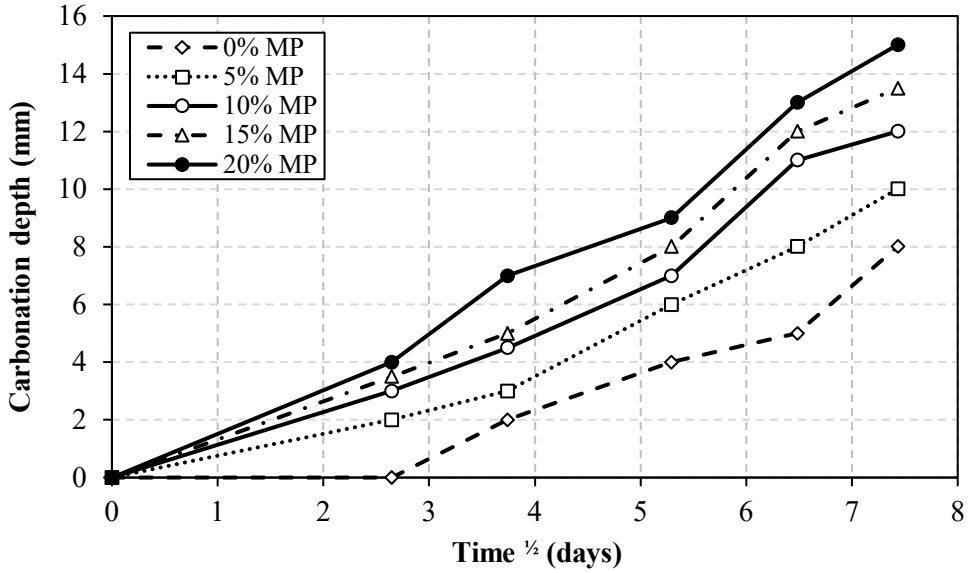

Fig. 12 *Evolution of carbonation depth for different mixture specimens*

3.2.2 Mass Variation

Fig. 13 depicts the variation in mass of mixtures as function of the age. It was observed an increase in mass for all mixture specimens. The mass gain increased by increasing the time of exposure in the accelerated carbonation chamber. Plain cement mixture showed the lowest value of mass gain, however, the mass gain increased by adding MP and increasing its percentage. The mass gain for control mixture at 56 days was 1.5%, while mixtures with 5%, 10%, 15% and 20% showed mass gain of 2.4%, 2.97%, 3.7% and 4.0%, respectively. These indicated that mass gain for mixtures including 5%, 10%, 15% and 20% of MP was higher by 1.6, 2, 2.5 and 2.6 in comparison to reference mixture. This mass gain may be due to the formation of new product: calcium carbonate ($CaCO_3$) because of the carbonation reaction between portlandite (C-H) or calcium silicate hydrate (C-S-H) and CO_2 as given by eqs. (4) and (5). The formation of calcium carbonate filled the pores and micropores and densified the internal structure of cement matrix and participated therefore to the mass gain.

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O \quad (4)$$

$$(CaO)_x(SiO_2)(H_2O)_z + xCO_2 \rightarrow xCaCO_3 + (SiO_2)(H_2O)_z \quad (5)$$

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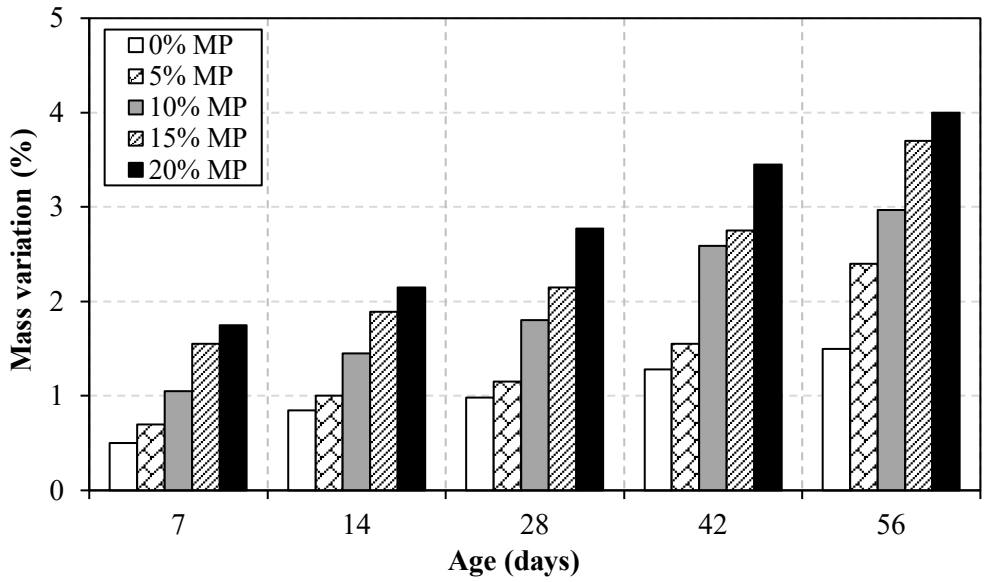

Fig. 13 *Mass variation of various mixture specimens vs time of exposure in the accelerated carbonation chamber*

3.2.3 X-ray Diffraction Analysis

In this study, small bits were extracted from the surface of carbonated samples after 28 days of conservation in carbonation enceinte, and were milled until obtaining fine powder (<80 µm). The obtained powder was exposed to X-rays diffraction to get an idea about new formed products. The various mineral phases found in the powder of carbonated samples are given in Fig. 14 and 15. It was noted the formation of new product: calcite (or calcium carbonate $CaCO_3$). The calcium carbonate was formed by carbonation of the portlandite according to equation 6. By comparing the results of the Fig. 14 and 15 with Fig. 8 (before carbonation), it can be seen that the calcite is more present in 20% MP mixture than 5% MP mixture which means that 20 MP are more carbonated. This can be attributed to the increase in the mixture porosity with increasing MP replacement rate [29]. In other hand, mixture with blended cement showed low portlandite content, so a smaller quantity of CO_2 is sufficient to consume all of the portlandite and as consequent the pH decreased and the specimens carbonate much faster than plain cement mixture.

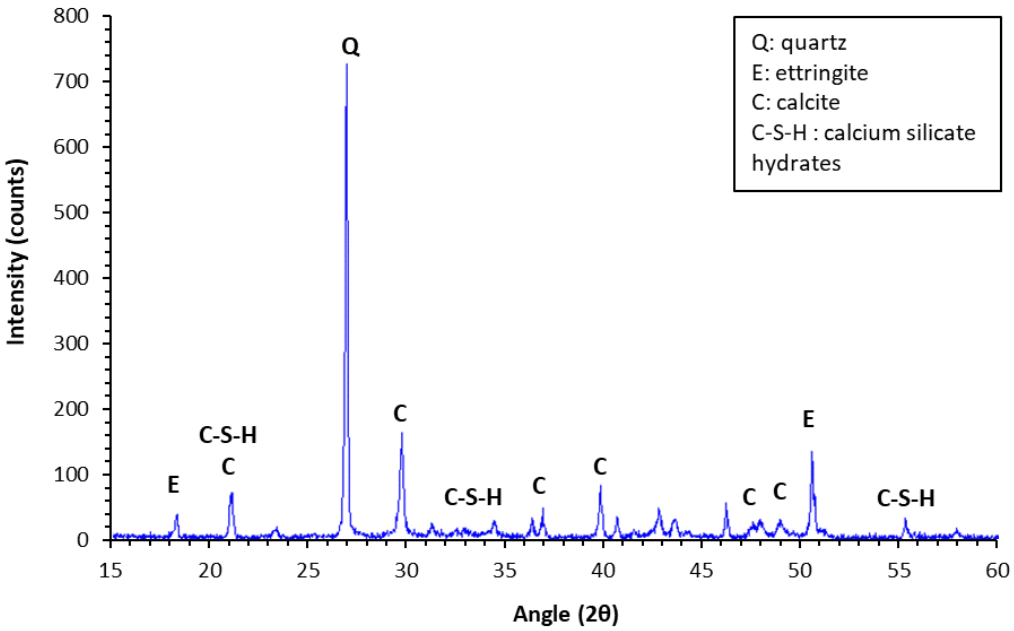

Fig. 14 *X-ray diffraction of carbonated specimens (5% MP)*

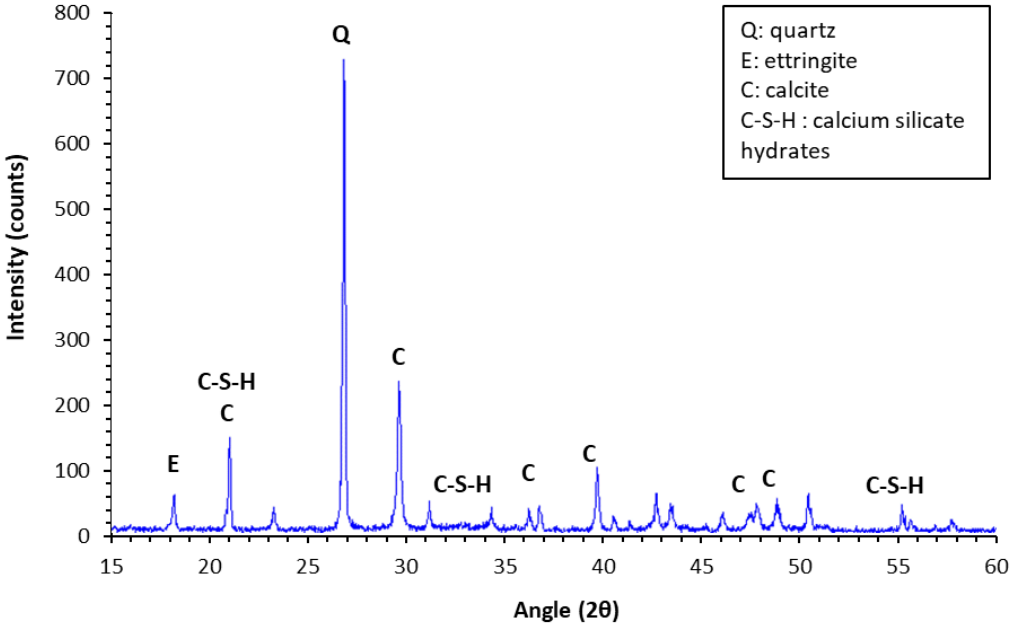

Fig. 15 *X-ray diffraction of carbonated specimens (20% MP)*

3.3 Development of Analytical Models for Durability Properties

3.3.1 Mass Variation vs Immersion Time in Sulfuric Acid Solution

Relationship between mass variation and immersion time in sulfuric acid solution in days (t) was established using regression analysis. Fig. 16 shows the obtained polynomial equations and coefficients of regression for various combinations of cement and MP. We note that the mass variation is increasing function of the immersion time. It can be deduced that the mass variation had an excellent correlation with the immersion time in sulfuric acid solution as the regression coefficients were to 1 for all the equations. These mathematical models can be used to estimate the mass variation at any given age, all other factors being equal.

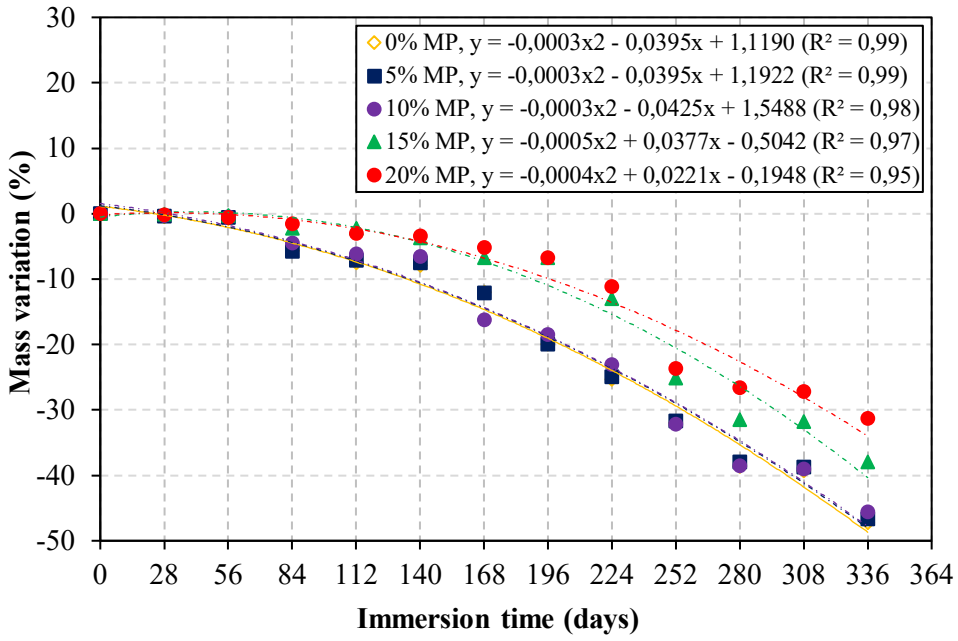

Fig. 16 *Relationships between mass variation and immersion time for various MP content exposed to sulfuric acid attack*

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3.3.2 Carbonation Depth vs Time

Numerous studies have aimed at developing analytical models for determining the carbonation depth of concrete. Most of these models are created after Fick's first law, in which the base model considers the carbonation depth to be a function of the squared root of time [29], [32]. In this study, a different analytical model was selected to determine the carbonation depth of the experimental conditions. Fig. 17 presents correlation between carbonation depth and the square root of exposure time of SCC mixtures subjected to carbonation conditions. It is seen that carbonation depth is increasing function of the of exposure time. The results indicate that there is a strong correlation between these parameters with high coefficients of regression.

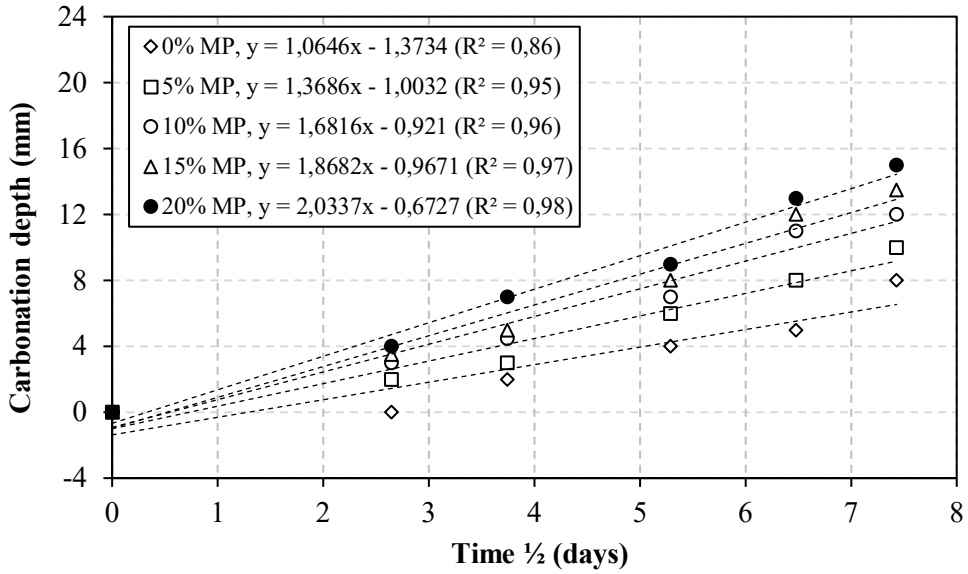

Fig. 17 *Relationships between carbonation depth and square root of exposure time for various MP content under accelerated carbonation*

4. Conclusion

Based on the obtained results, the following conclusions can be drawn:

- All mixtures showed progressive degradation with the immersion time in the acid sulfuric solution. Partial substitution of cement with marble powder reduced the mass loss and improves therefore the resistance to acid sulfuric attack.
- For accelerated carbonation test, adding marble powder led to mass gain at all age of concrete mixtures. The highest mass gain was observed in mixture with 20% of marble powder.
- Carbonation depth significantly increased by increasing marble powder content. It increased by 25%, 50%, for 68.8% and 87.5% for mixture with 5%, 10%, 15% and 20% of marble powder, respectively.
- Regression models established could be used to predict the mass variation of specimens subjected to sulfuric acid aggression based on the time of immersion for various combinations of cement and MP. A strong correlation was found between carbonation depth and square root of exposure time for all mixture specimens (depending on the MP content) under accelerated carbonation.

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