

Functionally graded concrete: porosity gradation to enhance durability under carbonation

Concreto com gradação funcional: gradação na porosidade para o aumento da durabilidade frente à carbonatação

Daniel Veras Ribeiro 
Adriana dos Santos Silva 
Cléber Marcos Ribeiro Dias 

Abstract

The present paper evaluated the potential application of the functionally graded material (FGM) concept to develop more durable concrete to carbonation, one of the main degradation mechanisms of reinforced concrete structures. Accelerated carbonation tests with controlled temperature (27 ± 2 °C), CO₂ concentration ($3 \pm 0.5\%$) and humidity ($65 \pm 5\%$) were carried out in homogeneous concretes and with functional gradation in which the porosity of the material was varied across the slices. For the manufacture of graded concrete specimens, concretes with water/cement ratios equal to 0.35, 0.45, and 0.55 were produced, with lower porosity ($w/c = 0.35$) close to the surface of the specimen. The advance of the carbonation front was evaluated after 8, 9, 10, 14, and 24 weeks of accelerated exposure, using the chemical indicator phenolphthalein. The results show that the functionally graded concrete had a carbonation coefficient (K) slightly higher than that of the concrete with a w/c ratio equal to 0.35 (1.71 and 1.54 mm.week^{-0.5}, respectively) and much lower than concrete with water-cement ratio equal to 0.45 (2.31 mm.week^{-0.5}) and 0.55 (3.78 mm.week^{-0.5}). This demonstrates that functional grading can be an efficient method to increase the durability of concrete elements subject to carbonation.

Keywords: Concrete. Functional gradation. Carbonation. Porosity. Performance.

Resumo

O presente artigo avalia o potencial de aplicação do conceito de material com gradação funcional (MGF) ao desenvolvimento de concretos mais duráveis frente à carbonatação, um dos principais mecanismos de degradação das estruturas de concreto armado. Ensaios acelerados de carbonatação com temperatura (27 ± 2 °C), concentração de CO₂ ($3 \pm 0,5\%$) e umidade ($65 \pm 5\%$) controladas foram realizados em concretos homogêneos e com gradação funcional em que a porosidade do material foi variada ao longo dos corpos de prova. Para a confecção dos corpos de prova de concreto com gradação funcional foram utilizados concretos com relações água/cimento iguais a 0,35, 0,45 e 0,55 de forma que tivéssemos uma redução de porosidade à medida que nos aproximássemos da superfície da peça. O avanço da frente de carbonatação foi avaliado após 8, 9, 10, 14 e 24 semanas de exposição acelerada, empregando-se o indicador químico fenolftaleína. A partir dos resultados obtidos observou-se que os concretos com gradação funcional apresentaram coeficiente de carbonatação (K) ligeiramente superior ao do concreto com relação a/c igual a 0,35 (1,71 e 1,54 mm.semana^{-0,5}, respectivamente) e bastante inferior aos concretos com relação água cimento iguais a 0,45 (2,31 mm.semana^{-0,5}) e 0,55 (3,78 mm.semana^{-0,5}), evidenciando que a gradação funcional pode ser um método eficiente para proporcionar o aumento da durabilidade de elementos de concreto sujeitos à carbonatação.

Palavras-chave: Concreto. Gradação funcional. Carbonatação. Porosidade. Desempenho.

¹Daniel Veras Ribeiro
¹Universidade Federal da Bahia
Salvador - BA - Brasil

²Adriana dos Santos Silva
²Universidade Federal da Bahia
Salvador - BA - Brasil

³Cléber Marcos Ribeiro Dias
³Universidade Federal da Bahia
Salvador - BA - Brasil

Recebido em 21/08/23
Aceito em 10/10/23

Introduction

About 50 years ago, Bever and Duwez (1972) recognized that synthetic composites could be designed and produced by locally varying the characteristics (or volumetric fraction) of the dispersed phases, the composition, and the microstructure of the matrix to obtain more efficiency in various applications and properties of materials. Because of the numerous advantages that these provide over homogeneous materials (e.g. higher mechanical strength and elasticity and lower density), functionally graded materials (FGM) have been employed in several branches of science and technology including cement-based composites (Neubrand; Rödel, 1997; Amada; Terauchi; Hasegawa, 1999; Geng *et al.*, 2022; Mak, Lees, 2022).

Functionally graded materials (FGM) are different from conventional materials in that the presence of a composition and/or structure variation, progressively throughout their volume, results in corresponding alterations to their material properties (Nogata, 1999; Kieback; Neubrand; Riedel, 2003; Ramu; Mohanty, 2014).

Bamboo is a classic example of a biomaterial with functional gradation, It is structured by natural processes in such a way that the fiber content varies throughout the thickness of its wall, conferring high mechanical resistance in the regions most required by tensions under the action of the wind (Amada *et al.*, 1997; Tan *et al.*, 2011), as shown in Figure 1.

Conventional materials present a homogeneous composition, with properties constant throughout the volume of the material. In a material with functional gradation, properties can vary in a uni, bi or tridirectional way, as shown in Figure 2, resulting in a desired performance (Dias; Savastano Junior; John, 2010). The gradation can be continuous, without an abrupt change in the composition and/or structure of the materials, or discontinuous, with a sudden change, depending on the way a particular property is arranged throughout the volume of the material, as shown in Figure 3 (Dias; Savastano Junior; John, 2010; Dias, 2011; Koizumi, 1997).

Figure 1 - Structure with functional gradation of bamboo - (a) cross section and (b) fiber distribution in bamboo (Tan *et al.*, 2011)

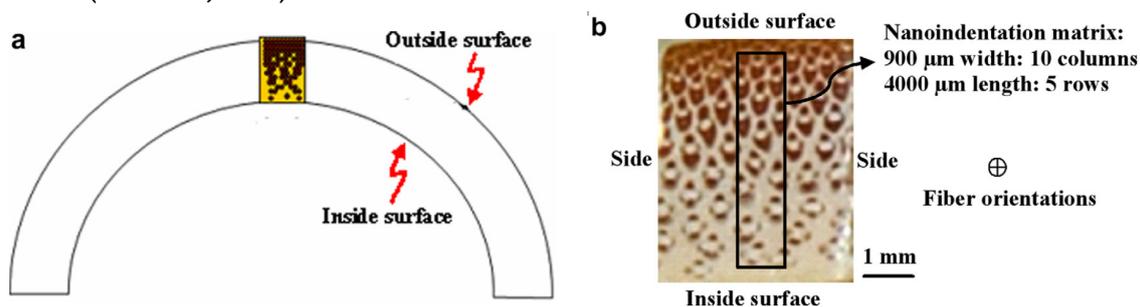


Figure 2 - Basic concept of a material with functional grading (Dias, 2011)

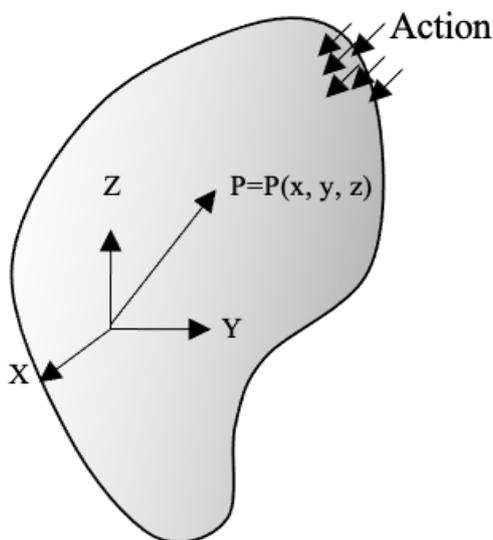
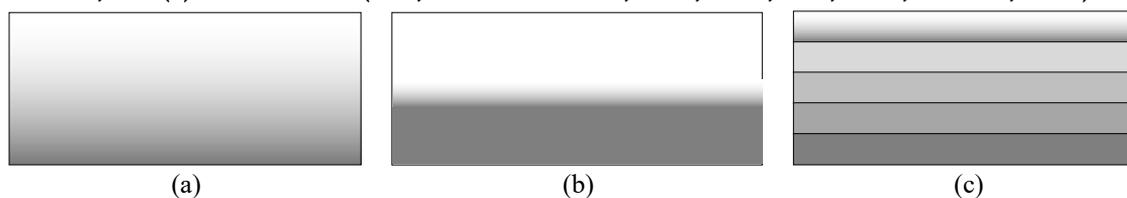


Figure 3 - Examples of gradation: (a) continuous; (b) continuous at the interface between two materials; and (c) discontinuous (Dias; Savastano Junior; John, 2010; Dias, 2011; Koizumi, 1997)

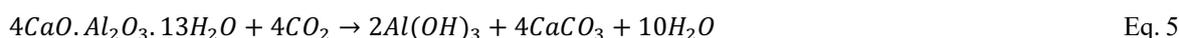


Gradation has been used incipiently in developing cementitious composites (Geng *et al.*, 2022; Mak; Lees, 2022; Dias; Savastano Junior; John, 2010; Sisomphon, Franke, 2007). However, the concept of FGM has yet to be applied to obtain durable concrete (Wen; Tu; Gan, 2013). Carbonation is one of the main degradation mechanisms of reinforced concrete structures. The high alkalinity of concrete promoted by the presence of calcium hydroxide $[Ca(OH)_2]$ and other alkalis present in the cement is responsible for the formation of a passivating film that surrounds reinforcements, protecting them (Sisomphon; Franke, 2007). Carbonation is a process in which carbon dioxide (CO_2) penetrates the interior of the concrete by diffusion, reacting with the water present in the pores, forming carbonic acid (Equation 1). The OH^- and Ca^{2+} ions are made available in the cement pore solution, mainly from the dissolution of calcium hydroxide or portlandite (compound formed during cement hydration), according to Equation 2, and then CO_3^{2-} reacts with Ca^{2+} , forming calcium carbonate, according to Equation 3.



Calcium carbonate does not cause concrete deterioration. However, its formation is characterized by the reduction of alkalinity due to the decrease in the concentration of hydroxyls in the interstitial solution, leading to a reduction in the pH to values close to 8 (Ribeiro *et al.*, 2018). The Pourbaix diagram (Figure 4) presents the immunity, passivation and corrosion situations for steel in an iron-water system at 25 °C (without chlorides). As can be seen in this diagram, the reduction of the pH of the medium to values close to 8, for the same corrosion potential, takes the system from the passivation zone to the corrosion zone. This pH reduction is responsible for destabilizing the passivating film, which protects the reinforcement against corrosion, thus initiating the corrosive process.

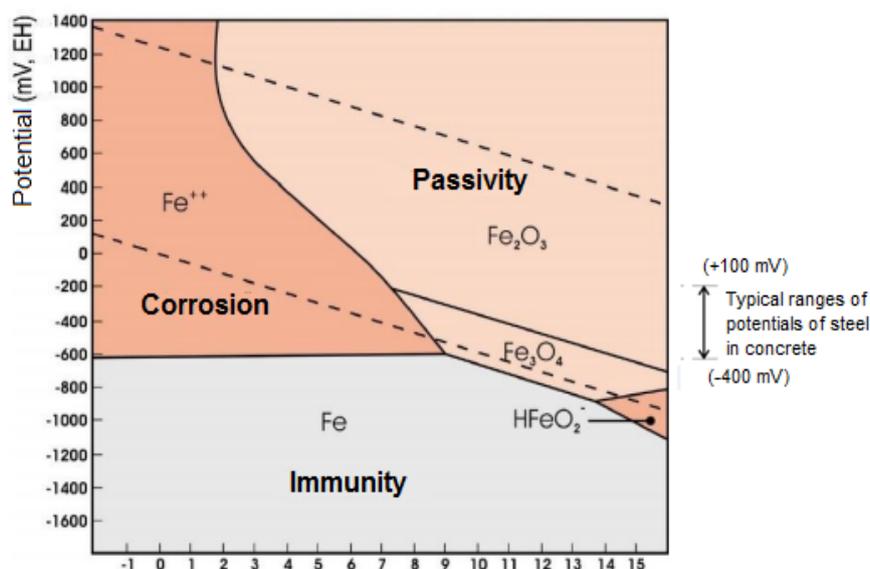
The carbonation reaction can also affect hydrated calcium silicate (C-S-H) and aluminate phases (as in C_4AH_x), according to Equations 4 and 5. The final product of the carbonation process is always calcium carbonate and water.



The thickness and quality of the concrete cover are of fundamental importance in the durability of concrete structures, as they are the main physical protection factors for reinforcement, making it difficult for aggressive agents to reach the reinforcement. In this context, functionally graded concrete emerges as an innovative alternative to increase the durability of reinforced concrete structures against the carbonation mechanism. Instead of developing concretes with high carbonation resistance and filling the entire element, it is possible to vary the properties of the specimens gradually and strategically to obtain an efficient barrier to the penetration of degradation agents, mainly in the cover thickness.

The present work therefore evaluates the potential application of the functionally graded materials (FGM) concept to develop more durable concretes to carbonation. Accelerated carbonation tests were carried out in homogeneous concretes (w/c equal to 0.35, 0.45, and 0.55) and concretes with functional gradation (in which the porosity of the material varied throughout the specimens so that there was a reduction of porosity close to surface). The advance of the carbonation front was evaluated after 8, 9, 10, 14, and 24 weeks of accelerated exposure using the chemical indicator phenolphthalein.

Figure 4 - Pourbaix diagram for the iron-water system at 25 °C and without chlorides (Ribeiro *et al.*, 2018)



Materials and methods

Materials

In the manufacture of both concretes (homogeneous and with functional gradation), Brazilian Portland cement with high initial resistance (CP V ARI), natural quartz sand, with maximum characteristic dimension (D_{max}) equal to 1.18 mm, basaltic gravel with D_{max} equal to 9.5 mm, polycarboxylate-based superplasticizer and potable water were used. Table 1 presents the physical properties of the materials used.

Methods

Preparation of test specimens

Three concrete mixtures were dosed to prepare homogeneous reference samples, with a slump equal to 150 ± 10 mm. Three water/cement ratios (0.35, 0.45, and 0.55) were used, with the workability being adjusted with superplasticizer. The weight-based unitary compositions of the concretes used were:

- composition 1 (w/c = 0.35): 1.00: 1.51: 2.01: 0.35 (cement: sand: gravel: water);
- composition 2 (w/c = 0.45): 1.00: 1.75: 2.49: 0.45 (cement: sand: gravel: water); and
- composition 3 (w/c = 0.55): 1.00: 1.99: 2.78: 0.55 (cement: sand: gravel: water).

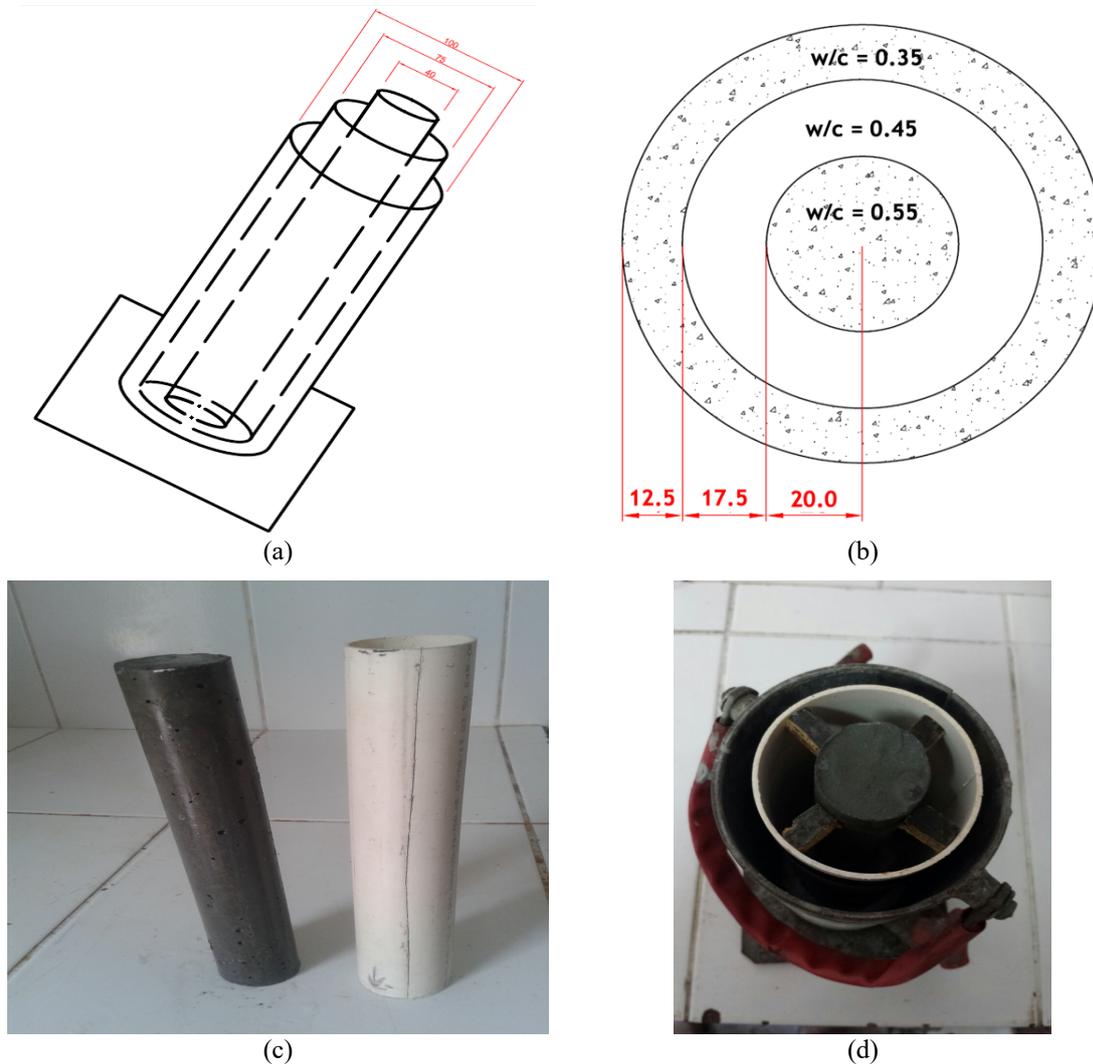
For the casting of each of the graded specimens, cylindrical molds with a diameter of 100 mm were used. Initially, the central core was molded, corresponding to a cylinder 40 mm in diameter, as shown in Figure 5c. Then, after curing, these concrete cylinders were inserted and centered in other tubes with a diameter of 75 mm, allowing the casting of the second layer, with a thickness of 17.5 mm. After the second layer was cured, the outer layer was cast with a thickness of 12.5 mm. The distribution of these layers is shown in Figures 5a and 5b.

The superficial (external) layer was made with concrete with w/c ratio equal to 0.35. In contrast, the intermediate and inner layers were produced with w/c ratios equal to 0.45 and 0.55, respectively, so that the best quality concrete simulated the cover. The interval between casting each layer was 24 hours. A vibrating table was used to compact the concrete. All specimens were demolded 24 hours after the last layer was cast, and they were cured in water saturated with lime for 28 days. These specimens were used for porosity/density, mechanical strength, and carbonation tests.

Table 1 - Physical characterization of the raw materials used

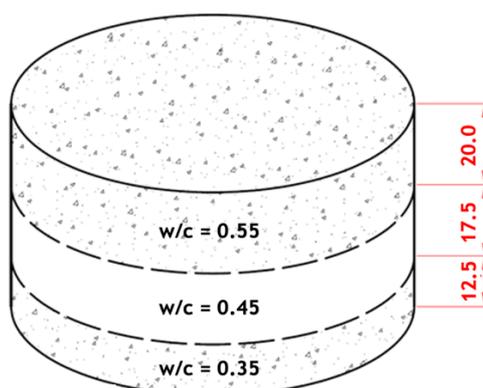
Property	Unit	Material		
		Sand	Gravel	Cement
Density NBR NM 52 (ABNT, 2009a)	g/cm ³	2.66	2.87	3.14
Specific Surface Area (Blaine method)	cm ² /g	-	-	4,120
Unit Weight NBR NM 45 (ABNT, 2006)	g/cm ³	1.46	1.40	-
Fineness Modulus NBR 7211 (ABNT, 2009b)	-	1.32	5.72	-
Powdery Material, < 75 μm NBR NM 46 (ABNT, 2003)	%	0.53	0.41	-

Figure 5 - (a) Scheme of the procedure for molding the concrete layers with functional grading (b) representation of the final configuration of the graded concrete section (mm) (c) central core, and (d) general distribution of layers in the graded specimen



For the capillary absorption (sorptivity) tests, the test specimens were made with the layers arranged in parallel, maintaining the thickness of the layers of the other test specimens and with the best quality concrete being positioned in the lower portion, in direct contact with the water, as shown in Figure 6.

Figure 6 - Scheme of concrete layers with functional grading for specimens used for the capillary absorption (sorptivity) tests



Apparent porosity and density

The method used to determine the apparent porosity of concrete is based on the Archimedes principle, according to the Brazilian standard NBR 9779 (ABNT, 2012). Three specimens were used for each water/cement ratio (homogenous and graded concretes), as shown in Figure 5. After 28 days of curing, the specimens were dried in an oven for 24 h at a temperature of 80 °C. After that, their dry masses (M_d) were measured. The specimens were immersed in water at a temperature of 22 °C and after 72 hours, the saturated (M_{sat}) and immersed (M_i) masses of each one were measured. Apparent porosity (P_a) and apparent density (D_a) were calculated using Equations 6 and 7, where ρ is the density of the liquid used (for water, equal to 1.0 g/cm³).

$$\%P_a = 100 \times \frac{M_{sat} - M_d}{M_{sat} - M_i} \quad \text{Eq. 6}$$

$$D_a = \rho \times \frac{M_d}{M_{sat} - M_i} \quad \text{Eq. 7}$$

Sorptivity

The Brazilian standard NBR 9779 (ABNT, 2012) prescribes the method for determining the water absorption through capillary rise for mortars and hardened concrete. The excessive absorption of water indicates a greater diffusion of elements and solutions into the mixture, increasing the chances of the occurrence of hydraulic and osmotic pressures.

Both diffusion and sorptivity increase with increased porosity, capillary rise is more dependent on the pore size than diffusion (diffusion is governed by concentration gradient, and capillary rise is influenced by fluid-pore wall interactions and pore size). Despite the driving mechanisms of both processes being different, diffusion becomes easier when the interconnection between the pores is greater, since ion exchange takes place through the interstitial solution. The wetting angle (fluid-pore wall interactions) is a determining factor if a water-repellent additive is used, but this was not the case in the present work.

Three specimens were used for each homogenous and graded concretes, as shown in Figure 6, aged 28 days. The specimens were dried in an oven for 24 hours and then cooled to room temperature and the mass of the dry specimens was determined. Then, specimens were positioned on supports and the test container was filled with water so that the water level remained constant and equal to 5±1 mm above its face bottom, avoiding wetting other surfaces. During the test, the mass of the specimens is determined at certain time intervals. The capillary absorption coefficient, or sorptivity (S), represents the mass of water absorbed per square meter of concrete, as a function of the square root of the elapsed time, according to Equation 8.

$$i = S \times \sqrt{t} \quad \text{Eq. 8}$$

Mechanical strength

The values of axial compression strength correspond to the average of three values for each homogeneous and graded concrete. Results were obtained at 3, 7, and 28 days after casting, with a displacement rate of 1.5 mm/min. The results that presented an error of more than 5% were excluded and replaced with others. The

limit of axial compression strength (R_c) is given by the ratio between the maximum load (P) supported by the test specimen and the area of its original cross-section (A), according to Equation 9.

$$R_c = \frac{P}{A} \quad \text{Eq. 9}$$

Carbonation test

To determine the carbonation depth, the specimens cured for 28 days were cut into slices, 50 mm thick. Their upper and lower bases were then sealed with paraffin, making the flow in this direction impossible.

Slices of homogeneous concrete specimens and concretes with functional grades were placed in a micro-processed carbonation chamber, with temperature (27 ± 2 °C), CO_2 concentration ($3 \pm 0.5\%$), and humidity ($65 \pm 5\%$) controlled, according to ISO 1920-12 (ISO, 2015) and IBRACON recommended practices (Ribeiro *et al.*, 2021). The progress of the carbonation front was evaluated after 8, 9, 10, 14, and 24 weeks of accelerated exposure, using a digital caliper and image treatment to measure the depth of carbonation after spraying an aqueous-alcoholic solution containing 1% phenolphthalein on the surface of the fractured specimens.

Due to the number of variables that influence carbonation, several mathematical models have been developed to estimate carbonation depth over time (Papadakis; Vayenas; Fardis, 1991; Lu; Liu, 2009; Taffese *et al.*, 2015; Possan, *et al.*, 2021; Felix; Carrazedo; Possan, 2021; Felix *et al.*, 2023). Due to the particularities of each developed model, the model derived from Fick's 1st Law of Diffusion is the most usual (Ribeiro *et al.*, 2023). Under stable hygrometric conditions, the carbonation depth is proportional to the square root of the exposure time, according to Equation 10, where K_a is the accelerated carbonation coefficient ($\text{mm/year}^{0.5}$ or $\text{mm/week}^{0.5}$); x is the carbonation depth (mm) and; t is the exposure time (years or weeks).

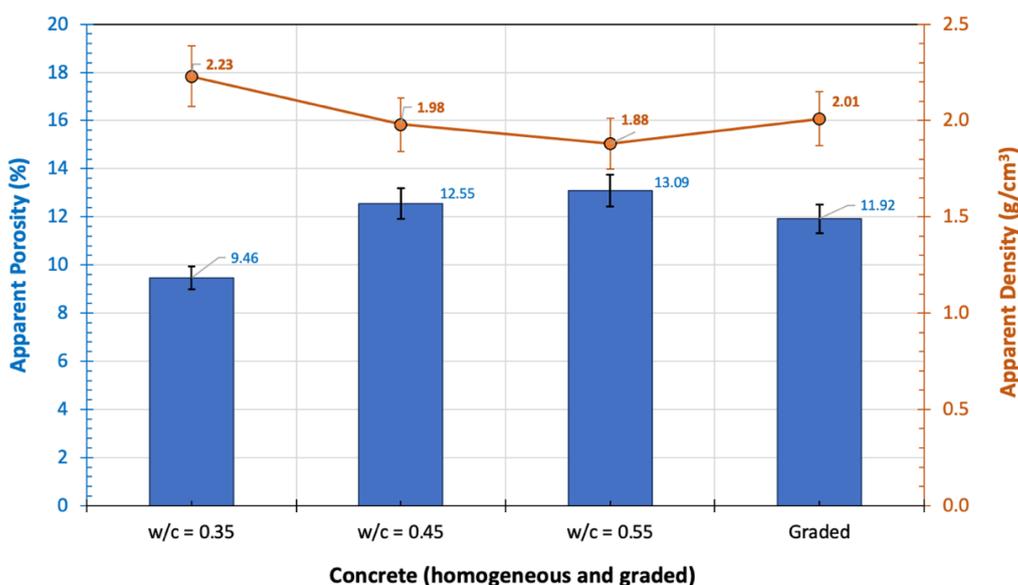
$$x = K_a \cdot \sqrt{t} \quad \text{Eq. 10}$$

Results and discussion

Apparent porosity/density and sorptivity

Figure 7 shows the apparent porosity and density values of the homogeneous and graded concrete specimens at 28 days. As expected, the increase in the w/c ratio led to an increase in the apparent porosity and a reduction in the apparent density.

Figure 7 - Apparent (a) porosity and (b) density of concretes with different water/cement ratios (homogeneous) and graded, at 28 days



The graded concrete is expected to present porosity and density associated with its volumetric fractions and the properties of the layers that constitute it. Thus, considering that the volumetric fractions of the layers are 43.75% ($w/c = 0.35$), 40.25% ($w/c = 0.45$), and 16.00% ($w/c = 0.55$), it is estimated that the theoretical apparent porosity and density of graded concrete would be equal to 11.29% and 2.07 g/cm^3 . The values obtained were equal to 11.92% (5.6% higher) and 2.01 g/cm^3 (2.9% lower), respectively.

Herrmann and Sobek (2016) used the concept of porosity gradation to produce a 4-meter-long concrete beam, with the aim of reducing its weight. The optimal spatial variation in porosity was determined through a numerical optimization procedure based on interactive finite element analyses. The results showed that the practical porosity was around 8% higher than the theoretical one suggested through numerical simulation due to interface problems. Furthermore, there was a weight reduction of up to 62% compared to a homogeneous structural component.

The values obtained experimentally in the present work are coherent, and this slight difference is believed to be associated with defects in the interfaces between the layers that constitute the graded concrete. Sequential casting, that is, after the preceding layer had hardened, caused the appearance of interfaces and possible adhesion problems between subsequent layers. An alternative to prevent the appearance of these defects is to carry out simultaneous molding of the layers still in the fresh state.

Figure 8 presents the capillary absorption coefficients (sorptivity) for both homogeneous and functionally graded concretes. As expected, it can be seen that the increase in the w/c ratio provided an increase in the capillary absorption coefficient, due to the increase in interconnectivity between the pores, with a stabilization between the values observed for concretes with the same w/c ratios at 0.45 and 0.55, which is associated with an increase in the number of pores. The graded concrete presented a satisfactory performance, close to that of the concrete with the lowest water/cement ratio, indicating the effectiveness of the grading.

Mechanical strength

Grading method is sometimes considered insufficient for the production of functionally graded materials (FGM) because it leads to the formation of sharp interfaces between layers causing the section fails by delamination instead of having a cohesive failure as occurs with sections built with the same material in its entirety (Ruys *et al.*, 2001).

The results obtained of the axial compressive strength of homogeneous and graded concretes are shown in Figure 9. Homogeneous concretes showed high strength, superior to 50 MPa ($w/c = 0.35$), 40 MPa ($w/c = 0.45$), and 30 MPa ($w/c = 0.55$). The graded concrete specimens presented axial compressive strength similar to homogeneous concrete with water/cement ratio equal 0.55. In directions perpendicular to the gradation, this effect should be even more pronounced, due to anisotropy introduced by gradation.

Figure 8 - Sorptivity of concretes with different water/cement ratios (homogeneous) and graded, at 28 days

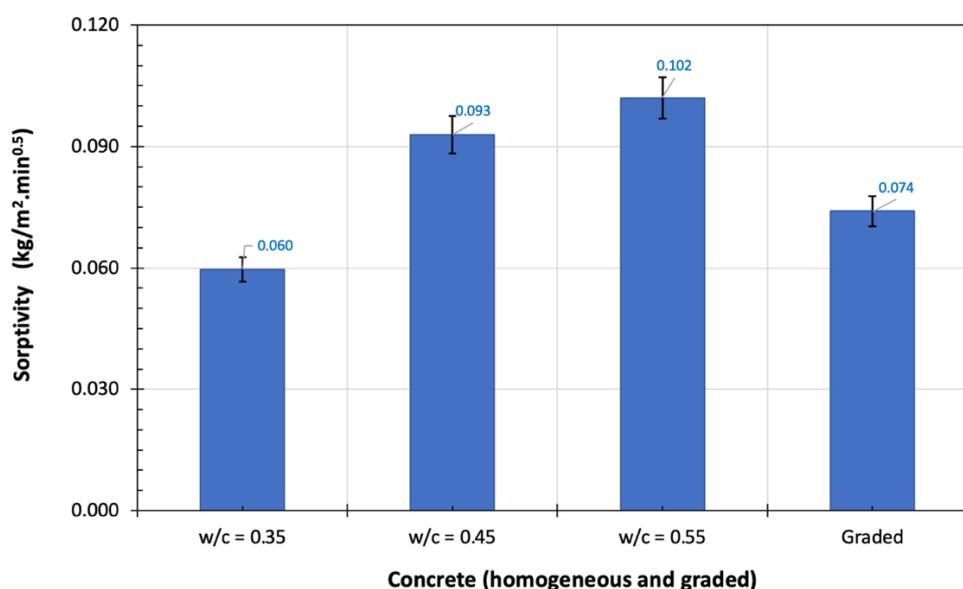
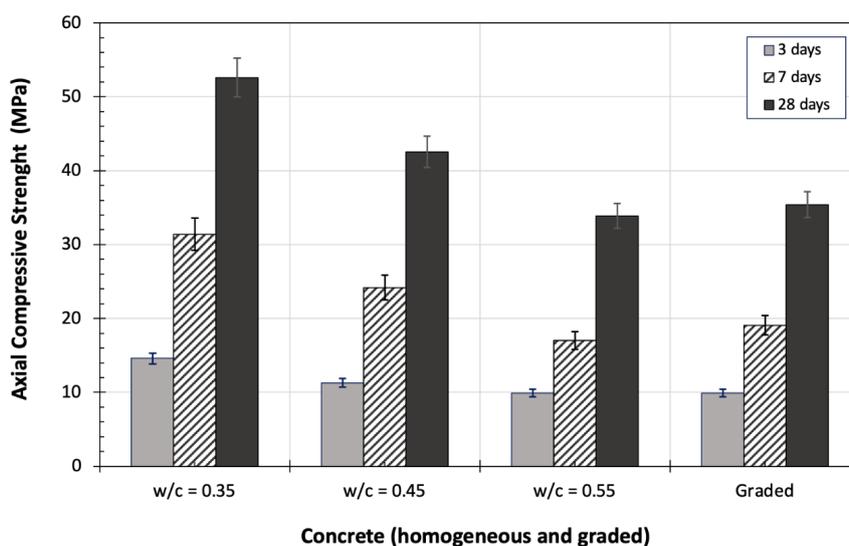


Figure 9 - Variation in compressive strength of concretes with different water/cement ratios (homogeneous) and graded



The graded concrete behavior visualized in Figure 9 occurred because the zone of lower resistance, with the highest w/c ratio, was the weakest link in this chain. The appearance of the first cracks inside the more porous concrete facilitated their propagation to the interior of the specimens, showing that the transfer of stresses between the layers was not adequate enough to increase the resistance of the whole set. This aspect is evident in Figure 10, which shows a specimen with a rupture in the interfacial region between the layers.

Han, Gan and Pratama (2016) observed a decreasing of load-carrying capacity of graded concrete due to the initial cracking of the weaker strength concrete layer (20 MPa), creating a crack propagation pattern that deviated from the columnar crack mode observed in the homogeneous specimens. The gradation, however, had little effect on the Poisson's ratio of graded concrete.

The alternatives to reduce this problem would be to improve the interfacial zone by applying adhesive bridges or increasing the surface roughness of each layer. Use concretes with similar strengths, or performing a simultaneous casting of the layers could improve the effects of the interfacial zone too.

Rio, Nguyen and Nguyen (2015) observed that graded sample strengths were always closer to those of the individual material with higher strength, using self-compacting cement composites (SCCC). Bajaj, Shrivastava and Dhoke (2014) observed an increase in strength in some gradation configurations containing concrete with 35% fly ash replacing cement.

Carbonation test

Since the material's durability is directly related to the ease of penetration of aggressive agents, such as CO₂ and chlorides, graded concrete must have low penetrability, despite the interfaces resulting from the casting process.

The average results of the carbonation depth for concrete specimens with different w/c ratios (homogeneous) and with porosity gradation are presented in Figure 11. The accelerated carbonation coefficients (K_a , mm.week^{-0.5}) are presented in Table 2.

As expected, there was an increase in the carbonation coefficient due to the increase in the water/cement ratio and consequently the porosity. The specimen with radial porosity gradation showed an accelerated carbonation coefficient (K_a) which was slightly higher than that of concrete with a w/c ratio equal to 0.35 (1.71 and 1.54 mm.week^{-0.5}, respectively) and much lower than concretes with water/cement ratio equal to 0.45 (2.31 mm.week^{-0.5}) and 0.55 (3.78 mm.week^{-0.5}). This shows that functional gradation can be an efficient method to increase the durability of concrete elements subject to carbonation (Figure 12).

Rio, Nguyen and Nguyen (2015) found that the gradation of silica fume addition contents provided a significant reduction in chloride penetration and carbonation of specimens. Graded concrete containing high levels of silica fume on the surface presented twice the service life of homogeneous concrete with lower silica fume content.

The carbonation results of graded concrete are also positive from an economic point of view, with a significant reduction (10.3%) in cement consumption compared to homogeneous concrete with a w/c ratio equal to 0.35 (Table 3).

Figure 10 - Rupture of graded concrete in the transition zone between layers



Figure 11 - Carbonation depth of concrete with different water/cement ratios and for graded concrete as a function of the square root of the time of exposure to the carbonation chamber

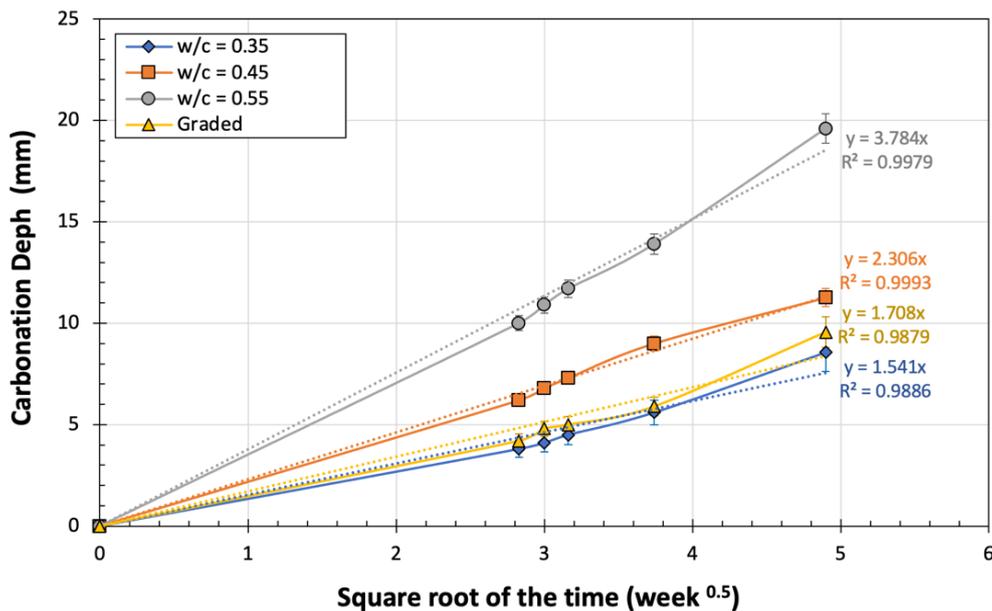
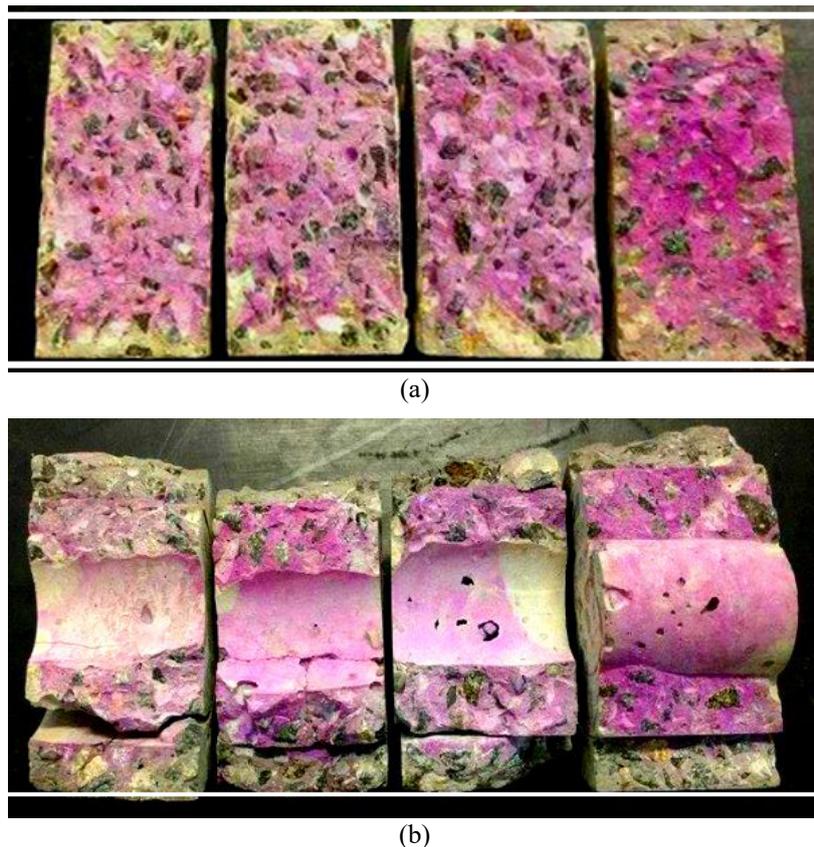


Table 2 - Accelerated carbonation coefficients (K_a , mm.week^{-0.5}) of concrete with different water/cement ratios and for concrete with porosity gradation

Accelerated carbonation coefficients (K_a , mm.week ^{-0.5})			
Homogeneous Concretes			Graded Concrete
w/c = 0.35	w/c = 0.45	w/c = 0.55	
1.54	2.31	3.78	1.71

Figure 12 - Carbonation of (a) homogeneous concrete (w/c = 0.35) and (b) graded concrete

Table 3 - Consumption of cement (kg/m^3) in concrete with different water/cement ratios and to graded concrete

Consumption of cement (kg/m^3)			
Homogeneous Concretes			Graded Concrete
w/c = 0.35	w/c = 0.45	w/c = 0.55	
516.40	435.93	386.81	463.27

Conclusions

This article evaluates the potential application of the functionally graded material (FGM) concept to develop concrete which is more durable to carbonation. The porosity of the concrete specimens was strategically varied throughout the layers to obtain higher performance against the actions of the environment to which the material is exposed. According to the results obtained in this research, it is possible to conclude that:

- the apparent porosity and density of concrete with porosity gradation presented intermediate values in relation to homogeneous concrete which were proportional to each of the constituent fractions;
- the graded concrete performed similar to that of the concrete with the lowest water/cement ratio (0.35), indicating the effectiveness of the grading;
- the concrete with porosity gradation presented axial compressive strength similar to that of the concrete with lower strength because the zone of lower resistance, with the highest w/c ratio (0.55), limited the graded concrete strength;
- the specimen with radial porosity gradation showed an accelerated carbonation coefficient (K_a) which was slightly higher than that of concrete with a lower w/c ratio (0.35) and much lower than that of concrete with a water-cement ratio equal to 0.45 and 0.55, showing that the functional grading can be an efficient method to increase the durability of concrete elements subject to carbonation;

- (e) the results presented for the graded concrete are relevant not only in terms of the durability of the material subjected to carbonation but also from an economic point of view since there was a considerable reduction in cement consumption per cubic meter for these concretes; and
- (f) although the results presented for the graded concrete have been positive, the performance optimization by applying this technique could be more significant if the grading were continuous, reducing the differences in the properties between adjacent layers.

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Acknowledgements

The authors thank the Research Support Foundation of the State of Bahia (FAPESB) for the scholarship granted to Adriana dos Santos Silva.

Daniel Veras Ribeiro

Conceptualization, Formal analysis, Funding acquisition, Methodology, Project administration, Resources, Supervision, Writing - review & editing.

Departamento de Ciência e Tecnologia dos Materiais | Universidade Federal da Bahia | Rua Aristides Novis, 2, Federação | Salvador - BA - Brasil | CEP 40210-630 | Tel.: (71) 3283-9852 | E-mail: verasribeiro@hotmail.com

Adriana dos Santos Silva

Data curation, Formal analysis, Investigation, Methodology, Validation, Writing - original draft.

Departamento de Ciência e Tecnologia dos Materiais | Universidade Federal da Bahia | E-mail: dricaa_ss@hotmail.com

Cléber Marcos Ribeiro Dias

Conceptualization, Formal analysis, Methodology, Supervision, Writing - review & editing.

Departamento de Ciência e Tecnologia dos Materiais | Universidade Federal da Bahia | E-mail: clebermrd@gmail.com

Editor: **Marcelo Henrique Farias de Medeiros**

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