

Comparative Analysis of Different Concentrations of CO₂ in Accelerated Carbonation Tests

Análise Comparativa de Diferentes Percentuais de CO₂ em Ensaios Acelerados de Carbonatação

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Abstract

Carbonation tests with carbon dioxide (CO₂) concentrations in excess of those found in nature have become common practice because they reduce test times. However, the lack of standards to guide the procedures and factors involved in these tests allow researchers to use different procedures, which makes comparisons between studies difficult or sometimes impossible. The present study aims to investigate the influence of the concentration of carbon dioxide on the carbonation depth of mortar samples subjected to accelerated tests. The tests were performed on concrete blocks measuring 40 x 40 x 160 mm that were cast with conventional Portland cement and pozzolanic Portland cement using water/cement ratios (w/c) of 0.40, 0.55 and 0.70. The samples were wet cured for 7 and 28 days in accordance with the procedures recommended in RILEM TC 116-PCD [1]. The tests were carried out at $20\pm1^{\circ}$ C and RH $70\pm5^{\circ}$. Two carbonation chambers were used, one with 6% CO₂ and the other with saturated CO₂¹. Additional tests included compressive strength at 7, 28 and 63 days to check the quality of the mortars. The test results indicated that the samples tested with CO₂ 6% have greater carbonation depths than those tested in the saturated carbonation chamber. The analysis of variance at 28 days shows that the concentration of CO₂ is a statistically significant factor in the carbonation depth of the mortars tested.

Keyords: accelerated carbonation, CO₂ concentrations, mortars.

Resumo

Os ensaios de carbonatação com concentrações de dióxido de carbono (CO₂) acima daquelas encontradas na natureza se tornaram uma prática comum. Isto é devido à redução do tempo necessário para o ensaio. No entanto, a falta de padronização dos procedimentos e fatores que envolvem estes ensaios permite que cada pesquisador adote procedimentos distintos para a sua realização, o que dificulta e, às vezes, até impossibilita a comparação entre as diversas pesquisas. Neste sentido, o presente trabalho tem por objetivo a verificação da influência do percentual de dióxido de carbono CO₂ na profundidade de carbonatação de argamassas submetidas a ensaios acelerados. Foram utilizados corpos-de-prova prismáticos, com dimensões de 40 x 40 x 160 mm, moldados com cimento Portland comum (CPI) e cimento Portland pozolânico (CPIV), nas relações água/cimento (a/c) de 0,40, 0,55 e 0,70. As amostras permaneceram em cura submersa por 7 e 28 dias e então foram sazonadas conforme os procedimentos da RILEM TC 116-PCD [1]. Os ensaios foram realizados a uma temperatura de 20±1°C e umidade relativa de 70±5%. Foram utilizadas duas câmaras de carbonatação, com concentrações de CO₂ de 6% e saturada¹. Foram ainda realizados ensaios de resistência à compressão axial aos 7, 28 e 63 dias, a fim de controlar a qualidade de execução das argamassas. Os resultados indicam que as amostras ensaiadas a 6% de CO₂ apresentam profundidades de carbonatação superiores às ensaiadas em câmara saturada. A análise de variância aos 28 dias de ensaio acelerado aponta que o percentual de CO₂ é estatisticamente significativo na profundidade de carbonatação das argamassas ensaiadas.

Palavras-chave: carbonatação acelerada, concentrações de CO₂, argamassas.

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1 Introduction

Carbonation is a complex physical/chemical phenomenon where cement components (hydrated or not) react with

other substances (mainly carbon dioxide) and form carbonates that lower the pH of the concrete.

Carbonation phenomena have been studied for approximately 50 years and it is safe to say that this phenome-

Table 1 – Lack of standardization of factors involved in accelerated carbonation fests							
Researcher	Year	Type of material	RH (%)	T (°C)	CO ₂ (%)		
Tuutti (5)	1982	concrete	80	-	1		
Ying-Yu and Qui-Dong (6)	1987	mortar	52	20	20		
Ho and Lewis (7)	1987	concrete	50	23	4		
Andrade (8)	1988	mortar	50 to 70	20	100		
Fattuhi (9)	1988	concrete	50 to 70	20 to 26	100		
Dhir et al. (10)	1989	concrete	50	20	4		
Ohga and Nagataki (11)	1989	concrete	50	40	7		
Kobayashi and Uno (12)	1989	mortar	60	20	10		
Papadakis et al. (13)	1991	concrete	-	-	50		
Branca et al. (14)	1992	concrete	75	20	30		
Nepomuceno (15)	1992	concrete	50 to 70	20	100		
Isaia (16)	1995	concrete	-	-	10		
Kazmierczak (17)	1995	mortar	-	-	100		
John (18)	1995	mortar	-	21.5	5		
Bauer (19)	1995	mortar	65 to 70	-	50		
Monteiro (20)	1996	mortar	50 to 70	24	100		
Seidler (21)	1999	mortar	68	21	5		
Roy et al. (2)	1999	concrete	52, 64, 75, 84 and 92	-	6		
Vaghetti (22)	1999	concrete	50 to 80	23	10		
Lopes (23)	1999	concrete	55	27	100		
Papadakis (24)	2000	mortar	61	25	3		
Alves (25)	2000	concrete	80	24	5		
Jiang et al. (26)	2000	concrete	70	20	20		
Cunha and Helene (27)	2001	concrete	65	23,5	100		
Johannesson and Utgenannt (28)	2001	mortar	65	20	1		
Sanjuán and Olmo (3)	2001	concrete	70	-	5, 20 and 100		
Venquiaruto (29)	2002	concrete	75	23	5		
Kulakowski (30)	2002	concrete mortar	70	25	5		
Lo and Lee (31)	2002	concrete	-	21	2		
Coelho et al. (32)	2002	concrete	60 to 70	26	100		
Kirchheim (33)	2003	concrete	70	25	100		
Van Gerven et al. (34)	2004	mortar	>90	37	5 and 20		
Gervais et al. (35)	2004	mortar	23, 48 and 98	3 -	100		
Abreu (36)	2004	concrete	70	25	5 and >50		

non is not fully understood. The results of different studies have yet to be compared, but it must be remembered that in most cases the conditions of the studies are different, which makes a reliable comparison of results difficult.

There are no standards to be followed for carbonation tests and each researcher is free to choose a specific test method. It is widely accepted that the factors involved in these tests affect their results.

This lack of standards affects several key variables involved in carbonation tests such as the type and length of cure, type and length of preconditioning², concentrations of carbon dioxide (CO₂), air temperature and relative humidity, etc. This study investigates aspects related to the accelerated carbonation of mortars exposed to a range of CO₂ concentrations in order to contribute to the standard-ization of these tests.

2 CO₂ Concentrations

Carbon dioxide concentrations in excess of what is found in the environment (where the range from 0.03 to 1%) have often been used in tests to investigate carbonation phenomena. These lab tests, known otherwise as 'accelerated tests', allow for greater control of the procedures and exposure conditions used in the process and results can be obtained in a much shorter interval (days or weeks) than what is required when the process is allowed to develop naturally (usually years). However, some authors ([2]; [3]; and others) warn that care must be exercised when running accelerated tests because the increased concentration of CO_2 can result in distortions of the phenomena, i.e. the chemical reactions involved in the process and the porosity of the resulting products may be affected.

Parrot [4] explains that all hydrated cement compounds can react with CO_2 under normal atmospheric conditions (approximately 0.03% by vol). However, when high concentrations of CO_2 are used, non-hydrate cement compounds may also react, which means that natural and accelerated tests are diverse.

In spite of the differences between natural and accelerated tests, it is believed that the latter are useful

carbonation predictors. However, in order to be able to compare the results of a series of studies in a reliable way, it is necessary to standardize the factors involved in the tests, which may otherwise affect results. The lack of standardization in the tests is shown in table [1], which lists a series of accelerated carbonation tests in concrete and mortars with a wide range of CO_2 concentrations and other differing variables.

The figures in the row showing CO_2 concentrations in table [1] reveal that the concentrations used in accelerated tests range form 1 to 100% CO_2 . This wide range makes comparing results difficult because the carbonation behavior is different for each concentration of CO_2 .

This study compares the carbonation depths obtained in accelerated tests with CO_2 6% and in a saturated chamber.

3 Experimental program

In the present study a choice was made to work with mortars to minimize the influence of coarse aggregates in the determination of the carbonation front. In addition, smaller test specimens can be used with this type of material. The mortars were mixed using a fixed concentration of water (H = 13.5%) and changing the ratio between binder and fine aggregate.

3.1 Materials

Two cement types were used: conventional Portland (Brazilian type CPI-S-32) and pozzolanic Portland (Brazilian type CPIV), whose physical and chemical properties are listed in tables [2] and [3], respectively.

The results of tables [2] and [3] meet the requirements of Brazilian standards for cement types CPI and CPIV, namely ABNT – NBR 5732 [37] and NBR 5736 [38]. These cement types are similar to cement types I and IP specified in ASTM – C 150 [39] and C 595 [40], respectively.

The fine aggregate was quartz sand with fineness modulus 2.72 and maximum characteristic diameter 2.40 mm. Its unit mass is 1.61 g/cm³ and its specific mass is 2.63 g/cm³.

Table 2 – Physical and chemical properties of cement types CPI-S and CPIV							
Properties		CPI-S	CPIV				
Mean particle size – laser (µm	1)	19.77	14.04				
Specific mass (g/cm ³)		3.11	2.75				
Blaine specific area (m³/kg)		388	450				
Consistency water – normal paste (g)		136	160				
Time of setting (min)	Initial set	170	200				
	Final set	260	300				
	3 days	24.7	17.4				
Compressive strength (MPa)	7 days	28.7	23.8				
	28 days	36.7	34.7				

Table 3 – Chemical properties of cement types used							
Chemical property	CPI -S (%)	CPIV (%)					
SiO ₂	18.74	29.04					
Al ₂ O ₃	4.31	11.37					
Fe ₂ O ₃	2.79	3.21					
MgO	4.03	3.87					
CaO	61,15	41.42					
Na ₂ O	0.07	0.08					
K ₂ O	0.78	0.85					
SO ₃	3.11	2.69					
Free lime	1.94	1.17					
Loss on ignition	4.37	3.91					
Insoluble residue	0.55	27.33					
Alkali equivalent	0.58	0.64					

3.2 Methods

Three water/cement ratios (w/c) were used: 0.40, 0.55 and 0.70 and the test specimens measuring 40 x 40 x 160mm were used in the accelerated carbonation tests and cylinders measuring 100 x 200mm were used in the compressive strength tests.

After casting, the test specimens were wet cured at $23\pm1^{\circ}$ C and RH 100% for 24 hours. They were then removed from the casts and immersed in a saturated lime solution in the curing room.

The submerged cure lasted for 7 and 28 days for the accelerated carbonation tests and 7, 28 and 63 days for the compressive strength tests.

Sample preparation for the accelerated carbonation test was carried out in accordance with the recommendations for tests of concrete permeability as indicators of concrete durability - RILEM TC 116-PCD [1]. Part 'A' of these recommendations provides guidelines on preconditioning concrete test specimens for gas permeability and water absorption tests. The recommendations state that it is necessary to pre-cast the mortars to establish the amount of water that will be lost after curing and before accelerated carbonation tests so that the optimal moisture content for this test is reached. This step is necessary to ensure that the same degree of hydration is found in the material used in these calculations and in the samples that were exposed to carbonation.

As described in the recommendation, after curing the test specimens were placed in an oven at $50\pm1^{\circ}$ C until the required loss of mass was reached. The samples were placed in air and water tight bags made up of layers of polyester + adhesive + aluminum foil + adhesive + PE film which were heat sealed and then placed again in the oven at $50\pm1^{\circ}$ C for additional 14 days so that their internal moisture reached a state of equilibrium.

After this period, the test specimens were taken to a room

with controlled temperature and RH ($20\pm1^{\circ}C$, RH 70 $\pm5^{\circ}$), where they remained in the sealed packets for another 24 hours so that their temperature would be the same as the room. They were then placed in the carbonation chambers, which were located in the same room.

One of the carbonation chambers uses a continuous feed of CO_2 with a concentration of 6%. The other chamber is equipped with a closed CO_2 system, and the system is only vented when the gas is fed into it. After filling the system with 100% CO_2 , the in and out valves are closed. The concentration inside the chamber could not be determined because there was no device available to gauge this. It was agreed that this chamber would be labeled 'saturated' because the volume of carbon dioxide substituted for air was always equal to or greater than 60% of the volume of the empty chamber.

Carbonation depths were measured at 7, 14, 21 and 28 days of exposure to CO_2 . The test specimen blocks were broken across their width with the aid of a press and a chemical indicator solution (phenolphthalein 1%) was sprayed over the freshly exposed surface, as recommended by RILEM CPC-18 [41].

Compressive strength was measured at 7, 28 and 63 days.

4 Results and discussion

4.1 Carbonation depth

Table [4] shows the mean carbonation depths obtained from six results as 3 test specimens were used, each one of which had two surfaces tested, as figure [1] shows. The edges of the samples, where CO_2 can penetrate from two directions, were disregarded. The carbonated area was measured on each surface and this value was divided by the length of lateral surface, resulting in the mean carbonation depth. The upper and lower surfaces of the

Table 4 – Mean carbonation depths (mm)								
Sample ID	Carbonation (days)				Carbonation (days)			
$Cement = W/C = \%CO_2$	7	14	21	28	7	14	21	28
CPI - 0.40 - 6	2.0	3.5	4.9	5.3	2.1	3.0	3.6	4.5
CPI – 0.40 – saturated	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
CPIV - 0.40 - 6	2.5	4.6	5.9	6.4	3.2	3.8	5.5	5.8
CPIV – 0.40 – saturated	0.0	0.0	0.0	2.1	0.9	3.0	3.3	4.1
CPI – 0.55 – 6	2.9	4.9	6.9	7.6	3.7	5.7	7.8	7.2
CPI – 0.55 – saturated	0.0	0.0	0.0	0.0	0.0	2.4	2.8	4.4
CPIV – 0.55 – 6	4.1	7.5	10.2	11.1	4.4	6.0	7.9	8.9
CPIV – 0.55 – saturated	0.0	3.4	4.8	5.1	1.1	4.6	6.1	7.0
CPI – 0.70 – 6	4.4	8.4	12.7	13.3	5.3	8.1	10.9	13.1
CPI – 0.70 – saturated	1.4	2.6	3.7	4.8	0.2	5.6	6.6	8.2
CPIV - 0.70 - 6	6.1	11.6	18.5	24.4	7.1	10.1	13.3	15.0
CPIV – 0.70 – saturated	6.5	7.2	8.5	8.8	2.4	6.4	8.3	9.3

test specimens were discarded because the results here showed high dispersion. Measurements were taken using the Carl Zeiss Vision software package (*Realise* 3.0) from digital images of freshly broken samples that were then sprayed with phenolphtalein. The effect of the concentration of CO_2 on the carbonation depth was checked using an analysis of variance (ANOVA) of the data obtained at 28 days of the accelerated test. The confidence level was

at 62 days of exposure using 5% $\rm CO_2$ and nearly saturated $\rm CO_2$ and found that the carbonation was more intense when concentrations of 5% carbon dioxide were used.

Saetta and Vitaliani [42] claim that one of the most important factors affecting carbonation rates is the concentration of CO_2 . Saetta and Vitaliani [42] and Abreu [36] believe that if the reaction is fast, the production of $CaCO_3$ in a given time interval is accompanied by the release of a

95% and results are shown in table [5].

Table [5] shows that the main effects of each factor were significant for carbonation depth. Apart from the interaction between the curing period and the w/c ratio, all remaining interactions were statistically significant. Figure [2] illustrates the isolated effect of the concentration of CO_2 on the carbonation depth.

Figure [2] shows that the mean carbonation depth in the test specimens stored in the saturated chamber is 4.56 mm, while in those kept in a chamber with 6% CO₂, this depth is 9.53 mm, a value approximately 2.1 times higher. In other words, the carbonation depth in the saturated chamber amounts to 48% of the carbonation depth in the chamber with 6% CO₂.

Abreu [36] studied the carbonation of concrete samples Figure 1 – Test specimen after analysis of carbonation depth larger amount of water than the pore network in the matrix can release in the same interval, and when equilibrium is reached, the progress of the carbonation front is slowed down. Saetta and Vitaliani [42] also mention that the amount of CO₂ taken up by the reaction may be greater than the diffusion rate of the gas in the concrete in the same interval of time, which would also slow down the reaction as there would not be enough carbon dioxide available to react. In other words, Saetta and Vitaliani [42] and Abreu [36] believe that when very high concentrations of CO₂ (values close to saturation) are used, the carbonation speed is reduced.

These conditions could explain why the carbonation depth in the saturated CO_2 chamber is lower than in 6% CO_2 is used. However,

Table 5 – Analysis of variance of the accelerated carbonation results at 28 days								
Factors	SQ	GDL	MQ	F-Test	p-value	Significance		
Curing period	19.83	1	19,83	16,75	0.0001	S		
Type of cement	248,96	1	248,96	210.23	0.0000	S		
w/c ratio	1287.51	2	643,76	543.62	0.0000	S		
% CO ₂	899.53	1	889.53	751.16	0.0000	S		
Interaction cure x cement	23.31	1	23.31	19.69	0.0000	S		
Interaction cure x a/c	0.11	2	0.056	0.05	0.9537	NS		
Interaction cure x $\%$ CO ₂	121.92	1	121.92	102.95	0.0000	S		
Interaction cement x w/c	19.89	2	9,94	8,40	0.0004	S		
Interaction cement x % CO ₂	5.14	1	5.14	4,34	0.0392	S		
Interaction a/c x % CO ₂	88.54	2	44.27	37.39	0.0000	S		
Error	152.76	129	1.18					
Total	2857.51	143						

p-value = probability, Student's t-distribution; significance: S = significant and NS = non-significant.

these assumptions still need to be confirmed by means of microstructural analysis before their validity is asserted.

Sanjuán and Olmo [3] carried out tests using concentrations of 5, 20 and 100% CO₂, and in their tests the carbonation depth was greater in test specimens carbonated with 100%. These researchers calculated the coefficients of diffusion for CO₂ and found unusual values for 100% CO₂. In another study, Kazmierczak and Lindenmeyer [43] compared the loss of mass in fully carbonated pastes with 100% CO₂ and atmospheric CO₂ and thermo-gravimetric (TG) tests showed that the loss is greater in pastes exposed to accelerated carbonation. This may suggest that when higher concentrations of $\mathrm{CO}_{\!_2}$ are used, a greater amount of carbonation products is generated or that the crystals formed in this condition are denser.

In this study, the carbonation front of the samples exposed

to 6% CO₂ was usually better-defined, i.e. it was easier to determine the carbonation front when compared with the samples testes in the saturated CO₂ chamber. The same phenomenon was reported by Sanjuán and Olmo [3] and Abreu [36], who point out that in accelerated tests, the carbonation front in lower CO₂ concentrations (20 and 5%) is better defined than when high concentrations are used (100% CO_2 and close to saturation).

Figure [3] shows the interaction between the curing time and the type of cement on carbonation depths.



Figure [3] indicates that the carbonation was affected by the initial curing period and this influence is greater when the test specimens are cured for 7 days.

Ohga and Nagataki [11] found that the initial cure affects more the coefficient of carbonation in cement with fly ash than when the pozzolan is not used. Balayssac et al. [44] studied curing times of 1, 3 and 28 days. When the curing period was extended from 1 to 28 days, the carbonation depth was halved after 18 months of exposure. However, the effect of the curing period was related to the type of cement.

At first sight, the behavior of cement CPI-S seems illogical. However, a strong influence of the test specimens subjected to carbonation in the saturated CO₂ chamber on this behavior is observed, as figure [4] shows. This illustrates the relationship between curing times and the percentage of CO₂ on carbonation depth.

> Figure [4] indicates a significant interaction between the concentration of CO₂ and the time of submerged cure. When this is extended from 7 to 28 days, an average decrease of 11% is observed on carbonation depths with 6% CO₂ while in the saturated CO₂ chamber, this decrease amounts to 79%.

> The influence of the concentration of CO₂ is more evident when a curing period of 7 days is used. In that case, the difference between the two concentrations is approximately 6.81 mm, while in the cure of 28 days this difference is approximately 3.13 mm.



According to table [5], the value of the 'F-test' for the interaction between the submerged cure and the type of cement is 19.69 and for the interaction between the submerged cure and the concentration of CO_2 is 102.95. A comparison of these values shows that the interaction with the concentration of CO_2 is 5.2 times stronger, which indicates that the behavior of cement, particularly CPI-S, in figure [3] was masked by the percentage of CO_2 , as figure [5] shows. This figure illustrates the second order interaction between the time of submerged cure, type of cement and the concentration of CO_2 on carbonation depths.

Figure [5] confirms that the behavior of CPI-S with reference to the time of submerged cure was the opposite of what was expected because of the carbonation tests in the saturated CO_2 chamber. An analysis of the results of the saturated CO_2 chamber (figure [5] (b)) also reveals that for both types of cement, carbonation was higher when the curing period was 28 days. It can also be pointed out





that when the lower concentration of CO_2 was used (6%), the carbonation depth showed a decrease for both types of cement when the curing period was extended from 7 to 28 days, and CPIV suffered a stringer influence of this extended cure.

The interaction between the w/c ratio and the concentration of CO_2 on carbonation depth is shown in figure [6].

Figure [6] indicates that for all w/c ratios used, the carbonation depth with 6% CO₂ is greater than with the saturated CO₂ chamber. The distances between the different w/c ratios are greater for 6% CO₂, particularly when the w/c ratio is increased from 0.55 to 0.70.

The literature on studies relating accelerated carbonation tests with different concentrations of CO_2 is limited. Overall, there is no consensus on the behavior of carbonation in accelerated tests, which means that further studies are



Table 6 – Mean compressive strength (f _c)								
w/c ratio	7 d	7 days		days	63 c	63 days		
w/c rano	f _c (MPa)	CV (%)	f _c (MPa)	CV (%)	f _c (MPa)	CV (%)		
CPI – 0.40	32.5	5.7	37.9	8.3	43.5	4.9		
CPIV – 0.40	29.8	3.9	39.9	13.4	50.6	3.5		
CPI – 0.55	23.6	5.8	28.9	2.7	31.2	5.1		
CPIV – 0.55	19.1	3.0	28.8	5.8	34.9	7.2		
CPI – 0.70	15.6	4.5	19.0	3,5	20.0	3.5		
CPIV – 0.70	12.5	6.2	20.5	5.1	27.2	1.9		

required to establish the effect of different concentrations of $\mathrm{CO}_{2}.$

4.2 Compressive strength

Table [6] presents the mean results for compressive strength for each age of break as well as their coefficient of variation (CV). The mean value was obtained from six test specimens.

These compressive strength results were only used as a parameter of control to check whether the mixes had been prepared correctly. The analysis of results shows small coefficients of variation, thus attesting the quality of the casting.

5 Conclusions

The results obtained in this study show that the use of very high concentrations of CO_2 (saturated chamber) result in lower carbonation depths than those obtained with lower concentrations of CO_2 (in this case, 6% CO_2) in the conditions of this study.

The carbonation front for the lower concentration of CO_2 was better defined, i.e. it was easier to determine than that of the samples from the saturated chamber.

It should be pointed out that other concentrations of CO_2 must be studied to establish the most suitable concentration for carbonation tests. Microstructural analysis is also required to investigate why carbonation is reduced with higher concentrations of CO_2 in accelerated tests.

In addition, the results of accelerated carbonation tests must be correlated with natural carbonation tests so that the conditions (including the concentration of CO_2) needed to approximate the results of accelerated tests to what is seen in actual practice can be identified. Tests allowing comparisons between accelerated and natural carbonation tests are in progress by the authors of this paper.

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