Influence of Fly Ash as Cement Substitution on Accelerated and Natural Carbonation of Concrete

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Abstract. The use of fly ash (FA) for the production of new concrete seems to be a promising solution for "greening" of construction industry. However, replacement of cement with supplementary cementitious materials such as fly ash influences concrete performance especially in terms of durability. One of major durability problems worldwide is carbonation-induced corrosion, given that a large number of infrastructural objects are exposed to a CO2-rich environment. The objective of this research was to analyse the influence of cement substitution level on accelerated and natural carbonation of concrete. Experimental program considered testing of 10 concrete mixtures selected in two groups – with water to binder ratio of 0,5 (400 kg/m^3 of binders) and 0,6 (300 kg/m^3 of binders), while in each group the cement substitution ratio was varied from 0% to 50%. Carbonation depth was measured after 14, 21 and 28 days of exposure to 2% CO2 in carbonation chamber, while the twin samples were exposed to natural carbonation. It was shown that accelerated carbonation depths were similar in both groups for mixtures up to 30% of FA, but they were doubled and tripled for larger replacement levels. Using the previously modified fib carbonation model for service life design, the prediction of natural carbonation was made. A reliability of proposed modification was assessed by comparison between predicted and measured values of natural carbonation after 19 and 34 months of exposure.

Keywords: Fly ash concrete · Carbonation · Accelerated test · Prediction model

1 Introduction

In order to preserve natural resources and lower cement consumption, different supplementary cementitious materials (SCMs) like fly ash (FA), blast furnace slag and silica fume are used in concrete production. Supplementary cementitious materials are usually by-products obtained from different industries and they possess pozzolanic activity potential. Fly ash is the most widely used SCMs in the region of Western Balkan, where the energy mostly comes from the coal burning power plants and consequently large quantities of FA are deposited in the landfills. However, the use of FA affects physical, mechanical and durability properties of concrete. Although concrete is usually considered as a building material that possesses good durability properties, cases of insufficient

durability, i.e. duration of service life, are not uncommon. One of the key reasons for deterioration of concrete structures in continental climate is carbonation induced corrosion of embedded steel reinforcement. Reaction between penetrated CO_2 from the atmosphere and alkalis in the concrete matrix $(Ca(OH)_2)$, decreases the concrete pH value, changing the chemistry balance between pore solution and hydrates and initiates corrosion process.

The results from literature show opposing conclusions regarding carbonation resistance of concrete made with FA as a cement replacement. This is expected having in mind the opposing effects of FA on carbonation – denser concrete structure and lower amount of $Ca(OH)_2$ [1]. The increase of FA amount (up to 60%) leads to the increase in carbonation depth [2–8]. Some experimental results showed that concrete made with 50% of cement replacement had a lower or equal degree of carbonation compared with ordinary Portland cement concrete, while concrete with 70% of cement replacement showed a greater degree of carbonation [9]. Some research has shown that, for the same concrete strengths, concrete with high amounts of FA (50% or more) exhibited almost the same carbonation resistance compared with ordinary Portland cement concrete [6].

Significant contribution to the analysis of resistance to accelerated carbonation of Portland, Portland-fly ash and blast-furnace blended cements was provided by a round robin test conducted as the mutual effort of RILEM TC 281-CCC members [10]. Apart from analysis of SCMs effect on carbonation resistance of mortar and concrete which is generally in line with the abovementioned (CEM I > CEM II > CEM III), the influence of curing time and regime is also emphasized. When water curing is reduced from 28 to 3 or 7 days, this results in higher carbonation depths.

Many factors affect the carbonation process such as binder type and content, concentration of CO_2, humidity, temperature, curing conditions and transport properties of concrete. It is even more complicated in case of real concrete structures exposed to CO_2 in natural environment where the influence of weather conditions (rain, wind) have also to be taken into account. The time required to determine the carbonation depth under natural conditions is measured in years–CO_2 concentration is approximately 0.03% in rural and 0.3% in urban areas [11]. Having this in mind, the usual quantification of concrete carbonation resistance is done through the accelerated carbonation tests. The acceleration of the carbonation process is achieved primarily by increasing the CO_2 concentration. In different standards and technical recommendations a great variety of CO_2 concentrations are prescribed, ranging from 1% to 50% [12]. However, typically it is 1–4%, which can be found in the most prominent (pre)standards and guidelines [13–15]. Although there is a wide range of models for carbonation depth predictions in ordinary Portland cement concrete [13, 16–18], there is still a lack of widely accepted models for practical prediction of natural carbonation in concrete with significant volume of FA. Existing models either require values for sets of parameters that are usually not available during the service life design [19–21], either still not validated through the comparison between predicted values and measured carbonation depth in natural conditions after certain period time [22].

2   Objectives and Methodology

Carbonation resistance of ordinary Portland cement concrete (OPC) and FA concrete was a topic of many previously published studies. However, there is still an inconsistency in the obtained conclusions regarding the impact of FA amount on carbonation process. In addition, the proposed model [22] for the application of accelerated test results in the practical prediction of carbonation depth in natural conditions for FA concrete have to be tested and validated. Therefore, the main objectives of this study were:

- to analyse the influence of cement substitution level on accelerated and natural carbonation of concrete with FA.
- to test the influence of curing regime, i.e. duration of water curing, on carbonation resistance
- to assess the reliability of previously modified *fib* carbonation model when applied to FA concrete

Effect of FA content on concrete resistance to carbonation was assessed by comparison of measured carbonation depth in samples after 14, 21 and 28 days of exposure to accelerated carbonation at 2% CO_2 according to fib recommendation [13]. Furthermore, the comparison of carbonation depths in samples of different concrete was done after exposure to natural carbonation for 19 months and 34 months.

Prediction of the carbonation depth under natural exposure conditions was evaluated using the inverse effective carbonation resistance (R^{-1}_{ACC}) and based on the fib Model Code 2010 [13]:

$$x_c(t) = \sqrt{2 \cdot k_e \cdot k_{cur} \cdot \left(k_t \cdot R^{-1}_{ACC} + \varepsilon_t \right) \cdot C_s \cdot t} \cdot W(t) \tag{1}$$

$x_c(t)$ – carbonation depth at the time t [mm], k_e – environmental function [−], k_{cur} – execution transfer parameter [−], k_t – regression parameter [−], average value: 1.25, R^{-1}_{ACC} – inverse effective carbonation resistance of concrete [(mm²/year)/(kg/m³)], ε_t – error term [(mm²/year)/(kg/m³)], average value: 315.5, C_s – CO_2 concentration [kg/m³], $W(t)$ – weather function [−].

Inverse effective carbonation resistance of concrete under accelerated conditions (R^{-1}_{ACC}) for all type of concrete was calculated according to Eq. (2) and measured carbonation depth after 28 days of exposure to 2% of CO_2 in carbonation chamber.

$$R^{-1}_{ACC} = \left(\frac{x_c}{\tau} \right)^2 \tag{2}$$

The natural inverse effective carbonation resistance of concrete (R^{-1}_{NAT}) can be obtained indirectly from the accelerated inverse effective carbonation resistance, R^{-1}_{ACC}, Eq. (3):

$$R^{-1}_{NAT} = k_t \cdot R^{-1}_{ACC} + \varepsilon_t \tag{3}$$

where the values of coefficients k_t and ε_t for concretes with different amounts of FA were used from previous investigations [22, 23] and summarized in the Table 1. Detailed

information about analysis, based on which modified coefficients k_t and ε_t were determined, are presented in [22, 23] and they are based on a large number of results found in literature for mixtures made with different class F fly ashes used as a replacement of 10-50% of cement.

This was a starting point for calculation of predicted carbonation depth based on Eq. (1).

Table 1. Values of coefficients kt and εt for concretes with different amounts of FA

FA amount	k_t	ε_t
0%	1,25	315,5
10-35%	1,25	8366
40-50%	3,05	16264

Calculations conducted within this research based on Eq. 1 were used the measured environmental data in laboratory: CO_2 concentration was 0,0471% i.e. $7,6773 \cdot 10^{-4}$ kg/m^3, and relative humidity needed for calculation of environmental function (k_e) was 52.5%.

3 Experimental Procedures

3.1 Materials and Mix Proportion

For the purpose of this research, two types of concrete mixtures were prepared and tested: with 300 kg/m^3 and 400 kg/m^3 of cement. In both types of mixtures, the amount of FA varied from 0% to 50% in relation to the mass of cement. A total of 10 concrete mixtures were prepared.

Natural aggregate (NA) used in this study was commercially available river aggregate obtained from the Danube River in Serbia divided in three fractions: I (0/4 mm), II (4/8 mm) and III (8/16 mm). An oven-dry density and water absorption after 24 h are shown in Table 2.

Table 2. Oven-dry density and water absorption after 24 h

Fraction size	Oven-dry density [kg/m^3]	Water absorption [%]
I (0/4 mm)	2510	0.40
II (4/8 mm)	2550	0.99
III (8/16 mm)	2630	0.87

Portland cement CEM I 42.5R with a specific gravity of 3100 kg/m^3 was used for all types of concrete. FA used in this study was a by-product from the combustion of coal

in thermal power plant "Nikola Tesla B" in Obrenovac, Serbia. The FA was obtained by burning mostly lignite coal and it was collected from the power plant during pneumatic transport before storing in silos. Based on the chemical composition of FA given in [22], it can be classified as class F fly ash according to the ASTM C618 classification [24]. The specific gravity of FA was 2075 kg/m^3, and the mean particle size was 8.53 µm.

3.2 Casting, Curing and Testing of Specimens

The proportioning of the concrete mixtures was based on the absolute volume method. Concrete mix designs are given in Table 3. The mixtures were designed to have water to binder ratio $\omega = 0.6$ for mixtures with 300 kg/m^3 of binder, and $\omega = 0.5$ for mixtures with 400 kg/m^3 of binder. Within both groups of mixtures, the percentage of cement replacement with FA was 0%, 20%, 30%, 40% and 50%.

Table 3. Mixing proportions of tested concretes

Concrete*	m_c [kg/m^3]	m_{fa} [kg/m^3]	m_w [kg/m^3]	m_w	Aggregate		
				$(m_c + m_{fa})$ [–]	m_I [kg/m^3]	m_{II} [kg/m^3]	m_{III} [kg/m^3]
300-0	300	0	180	0.6	742	649	464
300-20	240	60	180	0.6	730	638	456
300-30	210	90	180	0.6	726	635	454
300-40	180	120	180	0.6	717	628	448
300-50	150	150	180	0.6	714	625	446
400-0	400	0	200	0.5	680	595	425
400-20	320	80	200	0.5	666	583	416
400-30	280	120	200	0.5	661	579	413
400-40	240	160	200	0.5	652	570	407
400-50	200	200	200	0.5	647	566	404

* mass of cement-mass of FA

All specimens were cast in steel moulds, and the concrete was compacted using a vibrating table. For the compressive strength testing at all ages, 150 mm cubic concrete samples were prepared. Carbonation resistance was tested on 120 × 120 × 360 mm concrete prisms. After casting, the specimens were covered with wet fabric and stored in the casting room at 20 ± 2 °C. They were demoulded after 24 h and the concrete cubes were kept in a water tank until testing while the concrete prisms were covered with a wet fabric and kept in the casting room for an additional 7 and 28 days. The aim of different curing period was to test the influence of curing regime on concrete carbonation resistance. The values of both mechanical and durability properties reported in this paper represent mean values of three measurements.

At the age of 28 days, prism samples were divided into two groups: one for the accelerated carbonation tests and the other for the natural carbonation test. Both tests were performed in the Laboratory for Materials at the Faculty of Civil Engineering, University of Belgrade. Accelerated carbonation tests were performed during 28 days

at a relative humidity (RH) of $65 \pm 5\%$ and a temperature of 20 ± 2 °C [13]. Samples were exposed to 2% CO_2 concentration in the carbonation chamber Memmert ICH 260C. The samples used for natural carbonation testing were placed in the laboratory under controlled conditions at an RH of 60% and a temperature of 21.0 °C [25] during 19 months for samples cured 28 days, and 34 months for samples cured 7 days. An average CO_2 concentration during that period was 0.0471% ($0.7677 \cdot 103$ kg/m^3) with a coefficient of variation of 10.9%. Carbonation depth measurements were performed with a phenolphthalein solution sprayed on the freshly broken concrete surface according to the European standard EN 14630 [26]. The carbonation depth was measured on five points per side, resulting in 20 measurements for each specimen and age.

4 Experimental Results and Discussions

4.1 Physical and Mechanical Properties

Results of tested physical and mechanical properties in fresh and hardened state are summary presented in Table 4. Workability of all tested concretes is in the range of class S1 and S2, according to [27]. Addition of FA in even small amounts of 20% increased the slump value for 4 times the value obtained for OPC concrete, while the further increase of FA volume did not significantly change workability of FA concrete.

Table 4. Mean values of physical and mechanical properties of tested concretes

Concrete	$\gamma_{c,fresh}$ [kg/m^3]	$\gamma_{c,hardened}$ [kg/m^3]	Slump [mm]	$f_{c,28}$ [MPa]	$f_{c,90}$ [MPa]
300-0	2348	2346	11	41.2	48.4
300-20	2342	2340	45	50.3	57.1
300-30	2341	2326	42	46.1	55.9
300-40	2337	2324	48	44.1	56.5
300-50	2334	2315	44	37.4	47.0
400-0	2352	2348	12	53.6	59.5
400-20	2325	2324	52	59.1	63.8
400-30	2321	2317	42	60.0	67.3
400-40	2319	2300	45	55.9	67.4
400-50	2299	2296	30	50.1	54.8

Densities of all concrete type both in fresh and hardened state are in expected range between 2300 kg/m^3 and 2350 kg/m^3, with lower values related to concrete with higher amounts of FA.

All concrete reached at least 45 MPa at 90–day compressive strength. Strength increase from 28 to 90 days is more pronounced in the group of concrete with 300 kg of binder (cement + FA) where the average increase was 21% compared to 12% increase in the group of concrete with 400 kg of binder. Addition of FA in both groups of mixtures

led to increase in compressive strength but both concrete with the highest percentage of replacement (50%) obtained lower compressive strength compared to the reference concrete. The increase is not linear but more random function with FA content, between 4% and 22%, with somewhat higher FA contributions to this increase in the group of concrete with 300 kg of binder. However, the highest strength in both groups reached concretes with 40% of FA as cement replacement.

4.2 Carbonation Resistance

Relationship between measured carbonation depth and square root of time for all concrete type is given in Fig. 1. It is mostly linear, as expected, but in several cases there were signs of carbonation reaction slowing down after 21 days. Due to the higher amount of cement and therefore higher alkaline capacity, all mixes in the group of 400 kg/m^3 of binder showed lower values of carbonation depth compared with the companion concrete from the group of 300 kg/m^3 of binder and the same percentage of cement replacement. The influence of FA content, i.e. cement percentage replacement is more pronounced within the group of 300 kg/m^3 of binder. There was a slight increase in carbonation depth up to 30% of cement replacement followed by significant rise of about 2,5 times in case of concrete with 40% of cement replacement with FA, Fig. 1. In the group of concrete with 400 kg/m^3 of binder, similar rise was notice in case FA content was increased from 40% to 50%. Obviously, in case of concrete with FA to binder ratio of 40% to 50% there is a lack of Ca(OH)$_2$ as it was used up in pozzolan reaction with FA. That led to faster penetration of CO$_2$ into the concrete, reaction with alkalis and consequently higher carbonation depth.
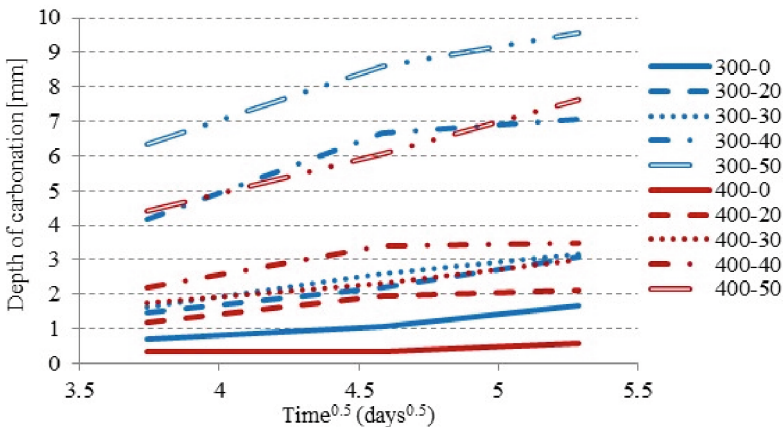

Fig. 1. Accelerated carbonation depth over time.

Carbonation depths in accelerated conditions in relation with concrete curing period are shown in Fig. 2. A period of 7 days of water curing was used because it represents the reference concrete curing period according to fib Model Code 2010 [13] and returns the unit value of execution transfer parameter, k_{cur}, Eq. (4). On the other hand, a period

of 28 days which represents a standard curing period for all physical and mechanical properties of concrete would produce 54% lower value of execution parameter and 32% lower value of calculated carbonation depth according to Eq. (1) if other parameters stay unchanged. It seems to be excessive so these two periods were chosen to test the influence of duration of curing time on both accelerated and natural carbonation depth.

$$k_{cur} = \left(\frac{t}{7}\right)^{-0.567} \tag{4}$$

It can be seen from the Fig. 2 that the curing period did not affect the carbonation depth as significant as it can be obtained through Eq. (4). There was no expected decrease in carbonation depth for samples cured 28 days compared to samples cured 7 days. Even more, except for referenced and concrete 300-40 all concrete mixtures had a slightly higher carbonation depth for samples cured 28 days compared to samples cured 7 days, although these differences are rather small, similar to the order of the measurements accuracy.

Based on the results it seems that the concrete curing period longer than 7 days did not affect the accelerated carbonation depth. However, it remains difficult to make conclusion as only two curing periods were analyzed. A specific experimental program dedicated to this important and still open question is urged.
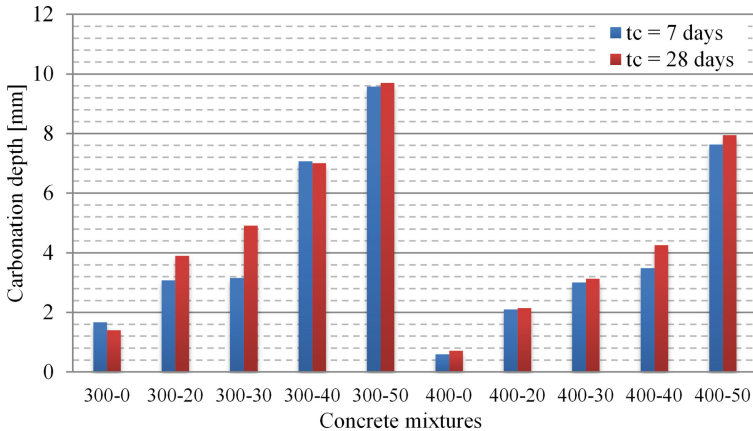

Fig. 2. Influence of curing conditions on accelerated carbonation depth

4.3 Carbonation Depth Prediction

The measured values of carbonation depths after 34 months in natural conditions for samples cured 7 days ($x_{c,NAT,meas.}$) are shown in Fig. 3. Two types of carbonation depth predictions are presented: 1) based on inverse accelerated carbonation resistance ($R^{-1}{}_{ACC}$) calculated with coefficients k_t and ε_t defined by fib Model code ($x_{c,NAT,cal.fib}$) [13] and 2) based on $R^{-1}{}_{ACC}$ calculated with modified coefficients k_t and ε_t for concretes with

different amounts of FA according to Table 1 ($x_{c,NAT,cal.modif.}$). In both cases, x_c was calculated according to Eq. 1.

It can be seen from the Fig. 3 that for reference OPC concrete, the measured carbonation depths was more than two times higher compared to the predicted depths. Similar level of (in)accuracy can be noticed for all other mixtures regardless of the cement amount and FA to binder ratio, except for mixes 300-40, 300-50 and 400-50 where the differences were about 25%, also not on the safe side.

Use of modified coefficients k_t and ε_t defined in previous studies [22, 23] and given in Table 3 led to significantly better correlation between predicted and measured values in the group of four concrete with 20% to 30% of FA in total binder mass, Fig. 3. The differences in this group were up to 30%. For concrete mixture with 40% to 50% of FA differences were in a range of 30% to 40% but all on the safe side.
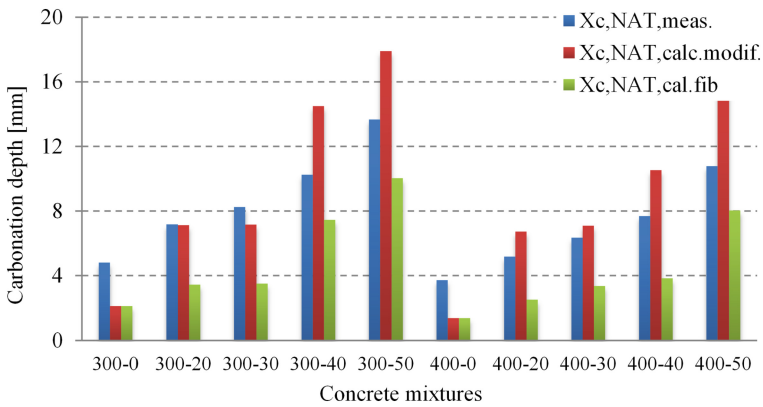

Fig. 3. Carbonation depth after 34 months in natural conditions for samples cured 7 days.

Carbonation depths after 19 months in natural conditions for samples cured 28 days are shown in Fig. 4. The different curing period was taken into account through the execution transfer parameter k_{cur}, Eq. (4).

The same trends can be observed as for the samples that were cured 7 days and tested after 34 months of natural carbonation. For all mixtures, regardless of the FA to binder radio, fib model did not return acceptable and reliable results when compared to the measured values. It is worth mentioning that the differences between measured and calculated carbonation depths are even the highest in case of two reference concretes without FA- more than double. In case when modified coefficients k_t and ε_t were used, improvements in predictions are noticeable in terms of safety and compared to the measured carbonation depths, showing the difference up to 30% for the group of four concrete mixes with 20% and 30% of FA in total binder content. For higher FA content (40% and 50%) differences are higher, but on the safe side.
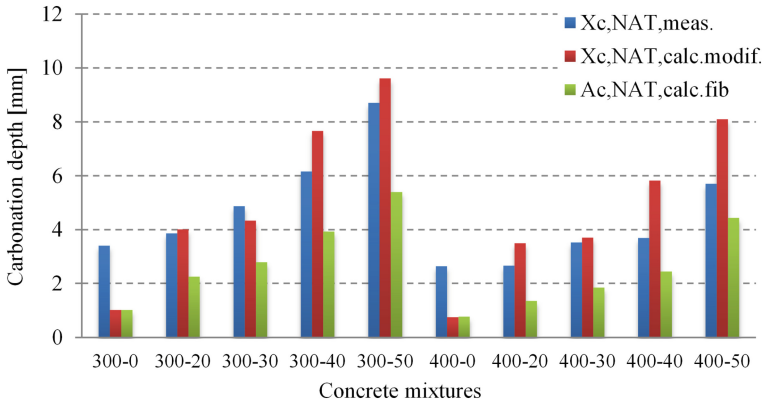
Fig. 4. Carbonation depth after 19 months in natural conditions for samples cured 28 days.

5 Conclusions

Based on the conducted measurements of the carbonation depth on concrete samples with and without fly ash (FA) and analysis performed using own experimental data and analytical models, the following conclusions can be made:

- even a small amount of FA of 20% as a cement replacement increased a workability of fresh concrete mixture; further increase in FA content did not affect concrete consistency,
- effect of 90-day compressive strength increase in FA concrete compared with the referenced OPC was the highest in case 40% of cement was replaced with FA,
- effect of FA on accelerated carbonation resistance was detrimental; significant drop in carbonation resistance occurred in concrete with content of FA of 40% and more in total binder content,
- 28-day water curing did not result in higher carbonation resistance of FA concrete exposed to accelerated test
- fib model for prediction of carbonation depth did not return reliable values if compared to the measured natural carbonation after 34 months, neither for FA concrete nor for the referenced OPC concrete; relationship between inverse carbonation resistance of OPC concrete in accelerated and natural condition needs adjustments,
- fib model for prediction of carbonation depth modified in previous studies for the use in case of FA concrete significantly improve correlation between measured and calculated carbonation depths, making the differences up to 30% in the group of concrete with maximally 30% of FA; database of experimental results on natural carbonation of FA concrete needs improvement to provide better prediction.

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