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EFFECT OF CURING REGIME ON POLYMER-CEMENT CONCRETE PROPERTIES

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Abstract: The general standards and guidelines recommendations for PCC suggest alternating conditions of curing: starting with wet conditions for effective hydration of Portland cement followed by air-dry conditions for polymer hardening. The often accepted curing regime of PCC covers 5 days of wet curing and then the air-dry curing but it is not the optimum one. The aim of the investigation was to find the best scenario for PCC with two types of polymer modifiers: two-component epoxy resin and water dispersion of polyacrylates. The following exploitation properties were accepted as the criteria of evaluation of PCC curing effectiveness: compressive strength, tensile splitting strength, surface tensile strength (by pull-off method), wear resistance, water penetration under pressure and resistance to carbonation. The optimum time of PCC wet curing is possibly between 7 and 14 days, however, it have to be verified experimentally for specific PCC composition.

Key words: Composites, Polymers, Polymer-cement concrete, Curing

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1. INTRODUCTION

Cement concrete is a dominating material in building structures due to the simple access to the constituent materials and relatively good technical properties at low costs. This probably will not change in the nearest future even taking into account ecological aspects (including high value of concrete carbon footprint). The development of sustainable concrete technology is focused not only on decreasing of ecological coefficients but also on designing concrete as a well-defined properties material i.e. material with properties precisely fitted to the exploitation conditions in designed structure and providing expected durability. Modern concrete modifications, among others, include the introduction of polymers into concrete a s a co-binder. The idea of polymer-cement concrete (PCC) is well described in the literature [5,17,19,25]. The studies in this field have brought different methods of polymer modifications, which are currently used in the various industry applications [5]. The effect of modification depends strongly on the mass proportions of cement and polymer. The content of polymer higher as 5% of cement mass allows to consider the polymer as a co-binder [17,19]. In such a case the binder in PCC consists of two phases: cement and polymer (introduced as polymer latexes or polymer dispersions, redispersible polymer powders, water-soluble polymers or liquid polymers [5]. The favorable influence of additives on concrete concerns mostly the transition zone between aggregate and cement paste, considered as a weakest zone which determined strength [42]. Cement paste in transition zone has usually higher porosity than that at a greater distance from coarse aggregate surface. In ordinary cement paste there are also more large crystals of calcium hydroxide $Ca(OH)_2$, which decrease mechanical properties of the material. The polymer phase in cement concrete increases the adhesion between paste and aggregate and make the microstructure more dense. The models for the microstructure formation of PCC and its use in composite designing were proposed by Ohama [25], Knapen [17] and others.

Relatively low attention in the published researches was payed to the problem of proper curing of polymer-cement concrete. This type of composite material contains two types of co-binder: hydraulic and organic. Optimum conditions for the setting and hardening of both of them are different. Cement hydration and hardening of the organic binder. The wet curing of the cement concrete fosters cement hydration while the organic resin hardening generally prefers dry conditions. Thus, the optimum conditions of PCC curing are the compromise between initial period of wet curing and subsequent period of dry hardening. Wet curing period recommended for cement composites varies from 1 to 21 days, depending on the cement type1. In the case of PCC extended time of maintaining high humidity may significantly worsen the effect of polymer hardening.

Hardening of PCC leads to the development of thin polymer film on the cement grains [18], which decreases the hydration degree. If the water access to the material in the first period of the process is not limited (due to the water curing) this film could be redissolved and redispersed in pores [39] giving opportunity to further cement hydration. After wet curing, in dry conditions polymer films are finally formed by coagulation on the cement and hydrates grains as an effect of water evaporation [21]. The type of polymer also is important, as the intensity of hardening disturbance by the high moisture environment varies for different types of organic binders. This effect is related to the different diffusivity of water through the continuous polymer phase. The influence of curing regime on polymer-cement concrete properties was discussed in the conference article of Lukowski, Woyciechowski and others [20] which contains also some own research results used in current paper. Disadvantageous effect of too short or too long period of wet curing of different polymer-cement composites, between them concretes and mortars, was observe not only on the mechanical properties but also on the properties related to the durability, for example such as water tightness, diffusivity for different media (chloride ions, carbon dioxide), frost resistance and resistance to abrasion [2,6,7,8,15].

2. RESEARCH SIGNIFICANCE

Standards, guidelines and recommendations show different requirements for the proper PCC curing regime. The literature references can be found for PCC on site curing [13,38] as well as for curing of PCC specimens in the laboratory [1,3,10,12,16,22,24,26,27,28,29,36] (Fig. 1). Recommended duration of wet curing of PCC is usually from one to seven days, with some guidelines recommending the higher humidity even after that time. It is hard to find in these documents any information about the quantitative impact of the course of curing on the technical characteristics of PCC [6,37].

Also, the guidelines for the handling of samples prepared in the laboratory and for concrete in the structure differ significantly. In the latter case, the key is technical simplicity of on-site processes which leads even to the complete withdrawal of the humid conditions according to guidelines [12]. In the laboratory conditions, it is essential to unify the procedures in different laboratories and optimize conditions for the co-binder in order to take advantage of the potential of both binders. Technical complexity of the curing process is less important here, as the size of the specimens is usually small.

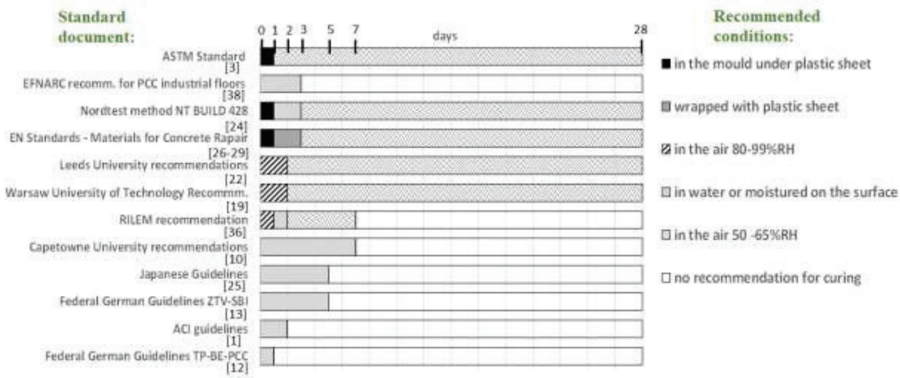

Figure 1. Time of PCC curing in humid conditions by various recommendations assuming 28 days as the time of total maturing.

The short analysis of the literature shown above leads to the formulation of the research problem, which includes the determination of the optimum water curing period for polymer-cement concrete from the point of view of most important exploitation properties, having regard to the type and content of the polymer binder.

3. INVESTIGATION PROGRAM

3.1 MATERIALS

The subject of the research program were the polymer-cement concretes modified with pre-mix and post-mix polymer binders, with polymer content 7% and 15% of the cement mass. Portland cement CEM I 42.5R was used as mineral binder. Natural river sand and gravel were used as aggregates. Cement concrete without any polymer was used as a reference material (OPC in Tab. 1). The binder content in OPC as high as 367 kg/m^3 was chosen as a typical medium content for structural cement and polymer-cement concrete. Modification of OPC with polymers was done by partial replacement of all components (constant mix volume) and correction of consistence by superplasticizer to the constant class S3 (acc. to EN 12350-2 [30]) As the pre-mix modifier aqueous dispersion of polyacrylic esters (PAE) was used. The content of the polymer in PAE was tested and obtained value 64% was adopted for the calculation of total water content in each compositions. The density of the dispersion at 23°C was 1.25 kg/dm^3. The post-mix binder used in research was a two-component (A-epoxy resin, B- hardener; mixing ratio A : B = 6 : 1 parts by weight) epoxy polymer.

Table 1. Compositions of concrete mixes [kg/m^3]

Mix symbol	Mix composition, kg/m^3							
	Water	Cement CEM I 42.5	Sand 0/2 mm	Gravel 2/8mm	PAE	EP		Superplasticizer
OPC	147	367	972	972	-	-		7.3
PAE 7%	130	360	953	953	40	-		4.5
PAE15%	110	349	924	924	81	-		1.8
EP 7%	145	362	958	958	-	Comp.A	21.7	2.5
						Comp.B	3.6	
EP 15%	142	356	942	942	-	Comp.A	45.7	2.5
						Comp.B	7.6	

3.2 CURING SCENARIOS

The primary aim of the study was to determine the effects of different curing conditions of PCC on the chosen properties of the composite. Six scenarios of curing in constant temperature were adopted (Figure 2), including 1-day in the form under the plastic sheet, and then - after demolding - in water at 20°C temperature (according to the recommendations of EN 12390-2 [31], concerning samples of cement concrete) or in air conditions (RH approximately 60%, temperature approx. 20°C - in line with European Standards [26-29], for materials with a polymer-cement binder for repairs). As extreme variants (1 and 6 in Fig. 2), the curing conditions preferred for only one of the co-binder components, ie. 27 days in water after demoulding as optimal conditions for cement (scenario 1) or 27 days in air after demolding as optimal conditions for polymer (scenario 6) are chosen. The intermediate variants covered the alternating conditions, i.e. the initial curing in water (for 2, 5, 7 or 14 days) and then in air-dry conditions until 28 day, i.e. the time of testing of the concrete properties.

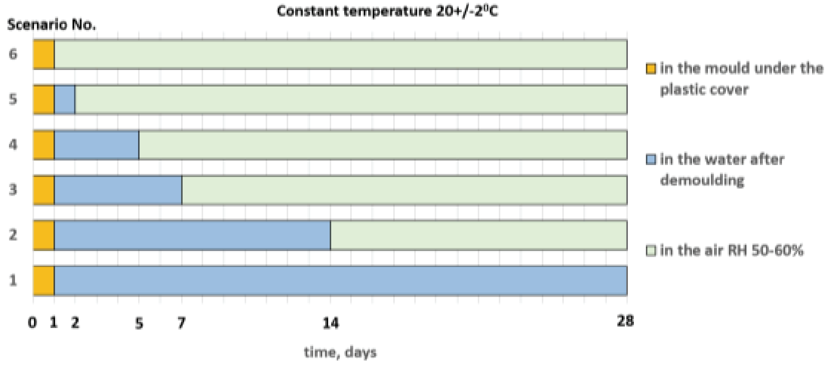

Figure 2. Scenarios of PCC curing adopted in the research program.

3.3 TEST METHODS

The research program included testing of selected concrete properties (tab. 2), which are considered as particularly sensitive to the curing procedure. In all cases the number of samples in tested series was 6, except of carbonation tests where the number of samples was 3. All the test procedures were beginning when the specimens were 28-days old. The conducted research allowed for assessment of the possibility of using these properties as the curing effectiveness criteria, as well as identifying of the preferred curing scenarios for polymer-cement composites.

Table 2. Concrete testing program

Property tested for 28-days old concrete	Method of testing acc. to standard	Dimensions of specimens [mm]
Compressive strength	EN 12390-2 [31]	150x150x150
Tensile splitting strength	EN 12390-6 [32]	150x150x150
Surface tensile strength by pull-off	EN 1542[26]	300x300x50
Water penetration under pressure	EN 12390-8 [33]	150x150x150
Wear resistance - Böhme method	EN 13892-3 [34]	70x70x70
Carbonation resistance in 4% CO_2	CEN/TS 12390-12 [35]	100x100x500

4. RESULTS AND DISCUSSION

The analysis of the effect of curing regime on the particular concrete properties was conducted with respect to both types of the polymers used. For all the series of results (in the paper the mean values are shown) the relative standard deviation (RSD, equal to standard deviation divided by mean value) was calculated to express the precision and repeatability of an assay. The range of RSD values for all tested characteristics are shown in table 3. The values are typical for cement and polymer-cement concrete and confirm the soundness of the investigation.

Table 3 Range of relative standard deviations for all tested properties in all series

Property tested for 28-days old concrete	Range of measured values for series	Range of calculated standard deviation for series	Range of calculated RSD for series, %
Compressive strength, MPa	36.1 - 54.3	4.1 - 5.5	8 - 10
Tensile splitting strength, MPa	2.1 - 3.5	0.22 - 0.39	10 - 13
Surface tensile strength by pull-off, MPa	1.9 - 3.5	0.21 - 0.36	11 - 12
Water penetration under pressure, mm	8 - 78	1 - 9	11 - 12
Wear resistance, $cm^3/50cm^2$	11 - 21	1.5 - 3.1	14 - 15
Depth of carbonation, mm	0 - 20	2 - 3	15 - 20

The results of investigation have confirmed the correctness of the generally accepted regularity that extending of the curing time of the cement concrete beyond seven days does not cause any further increase in compressive strength. However, within these 7 days the positive influence is clear (an increase of nearly 20%). In the case of concretes containing polymer, extension of curing time for more than 7 days can lead to a reduction in strength, especially if the polymer content is high (Fig. 3). Epoxy polymer EP addition resulted in a slight decreasing of the compressive strength, while this effect was not observed when using PAE.

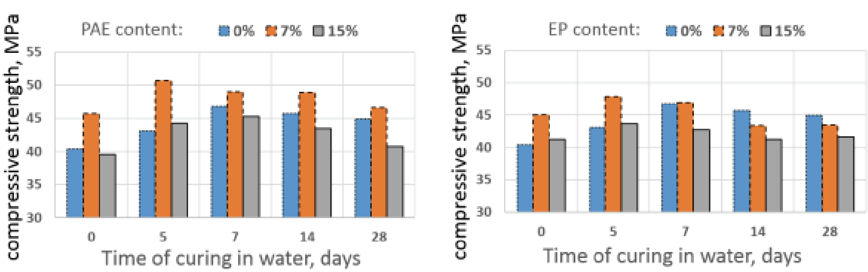

Figure 3. Influence of water curing time on the compressive strength of concrete. Test performed for 28 days concrete

Extending of the water curing time improves also the tensile splitting strength of the cement concrete. The addition of the polymer does not improve this property, regardless of the type and dosage of the polymer (fig. 4). In contrast, decrease of this property in case of a longtime water curing is clearly visible for PCC with PAE (fig. 5) regardless of the polymer content. This effect is not observed for PCC containing PE (fig. 6).

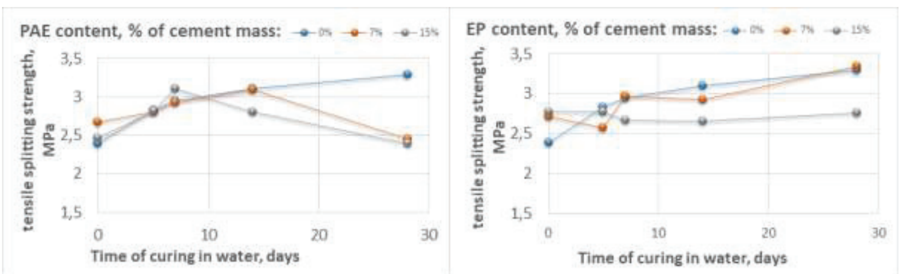

Figure 4. Effect of water curing time on tensile splitting strength of PCC with PAE (a) and EP (b). Test performed for 28-days old concrete

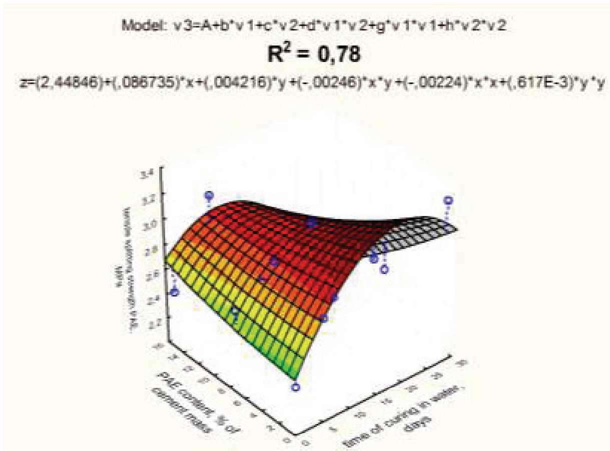

Figure 5. Tensile splitting strength for 28-days old PCC with PAE as a function of PAE content and water curing time

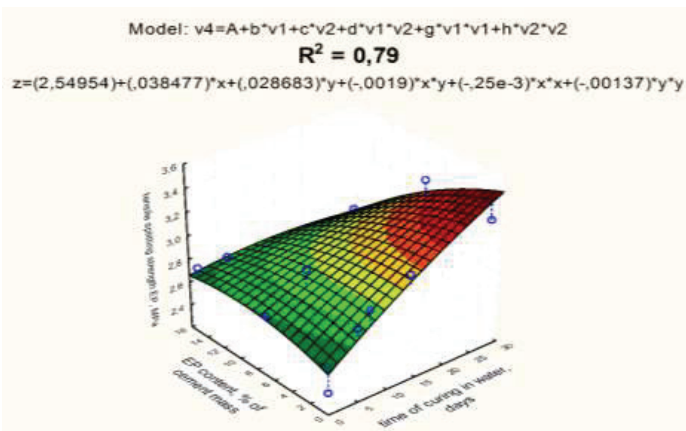

Figure 6. Tensile splitting strength for 28-days old PCC with PE as a function of PE content and water curing time

Relation of surface tensile strength tested by a pull-off to a time of water curing is similar. Also in this case, prolonging of water curing of PCC for over 5-7 days leads to a decrease of this property (Fig. 7). Only exception is the concrete with the addition of 7% of PAE, for which the reduction of surface tensile strength is observed after 27-day curing in water.

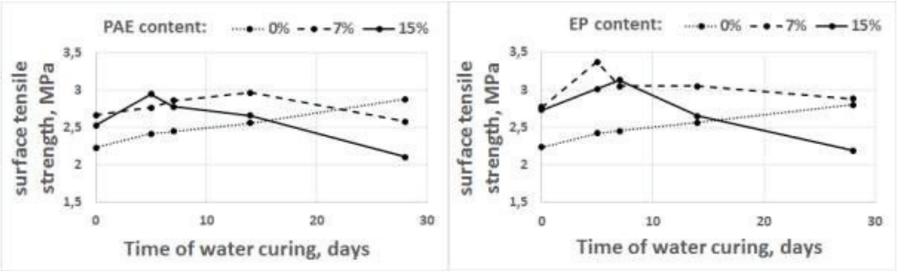

Figure 7. Effect of water curing time on surface tensile strength of a PCC with PAE (a) and EP (b). Test performed for 28-days old concrete

The polymer addition, as expected, tightens the concrete structure, which is reflected in the results of testing of the water penetration under pressure. In the case of concrete with EP, the sensitivity of this property to the course of curing was not stated, which may be due to an "autogenic curing", associated with the reduction of water loss from the concrete by the sealing film formed of a polymer introduced as a resin. In the case of concrete with PAE, worse results in the absence of curing (40 mm - Figure 8) can be associated with an increased permeability of the polymer film introduced to concrete as an aqueous dispersion. The conducted analyses enable the estimation of the sensitivity of PCC properties to curing regime. The relative difference between maximum and minimum values of the property, tested after 28 days, obtained after various water curing time (Tab. 3) has been proposed as the basis for the estimation. Water penetration under pressure appeared to be the most dependent on the curing way, while the compressive strength was the least sensitive, however, its sensitivity was still at a level allowing for using this property as a criterion for optimization of PCC curing method. It should also be stressed out that the addition of EP in the amount of 15% (by mass of cement) reduces the influence of curing on the results of compressive strength and tensile splitting strength testing. The results of testing the wear resistance (fig. 9) are not clear. The prolonged wet curing slightly worsening wear resistance of ordinary concrete which was unexpected and is probably related with wet conditions of specimens during wearing procedure. The effect for PCC concretes is opposite and it is connected with more watertight structure of modified concrete, which prevents the water penetration which leads to the effect of specimens softening, depending on materials susceptibility for softening coefficient.

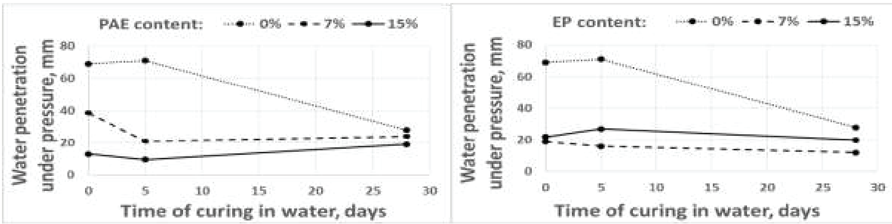

Figure 8. Wet curing time vs water penetration under pressure of PCC with PAE (a) and EP (b). Test performed for 28-days old concrete

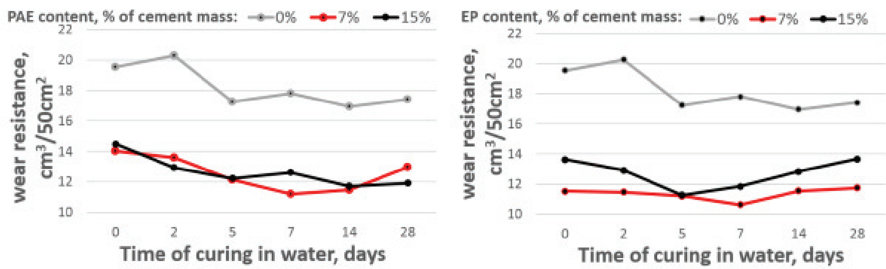

Figure 9. The influence of water curing time on wear resistance of a PCC with PAE (a) and EP (b). Test performed for 28-days old concrete

Table. 4 Relative differences between maximum and minimum values of concrete property tested after 28 days, obtained after various water curing time

Property	Relative difference between maximum and minimum values of property tested after 28 days, obtained after various water curing time, %				
	PAE		EP		OPC
	7% of cement mass	15% of cement mass	7% of cement mass	15% of cement mass	
Compressive strength	10.9	14.1	10.4	5.8	15.8
Wear resistance	25.7	23.7	10.6	21.3	19.5
Pull-off strength	15.1	40.5	21.7	42.9	29.1
Tensile splitting strength	26.1	30.1	30.0	4.1	37.3
Water penetration under pressure	83.3	100.0	58.3	35.0	153.6

The analysis presented on the Fig. 10 confirms the existence of the optimum time of the humid curing of PCC. For each of the five tested properties the highest value was obtained when the alternating conditions of curing were employed, regardless of the polymer content in concrete.

Figure 11 shows the results of testing at the age of 28 days, regarding to compressive strength, wear resistance and surface tensile strength, respectively, obtained in the absence of wet curing (red bars) and in the optimum curing conditions (i.e. in the conditions in which the value of the given property was the highest - yellowy bars) in relation to the result of the tests after 28 days of curing in water. The highest value of each of the tested characteristics, regardless of the type of polymer and its content, was obtained after the curing in water from 5 to 14 days. This finding confirms that the time of water curing, optimum from the point of view of the aforementioned characteristics, exists in this range. It should also be noted, that in the case of the compressive strength of all the tested concretes and wear resistance of concrete with PAE - apart of the optimum curing – the storing for 28 days in water is a better scenario of curing than no humid curing at all.

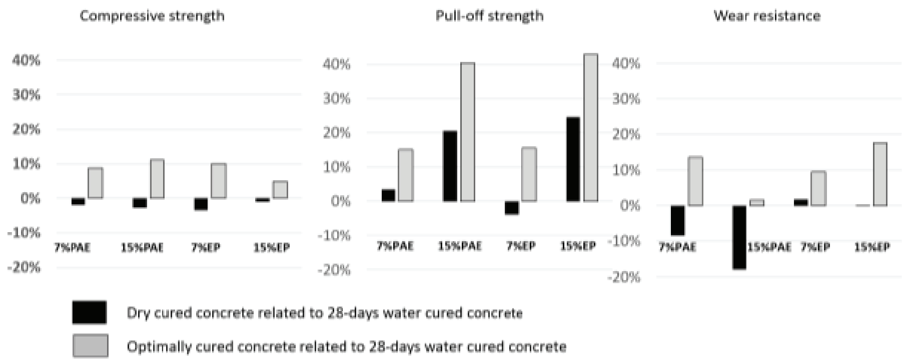

Figure 10. Results of testing at the age of 28 days: compressive strength, wear resistance and surface tensile strength

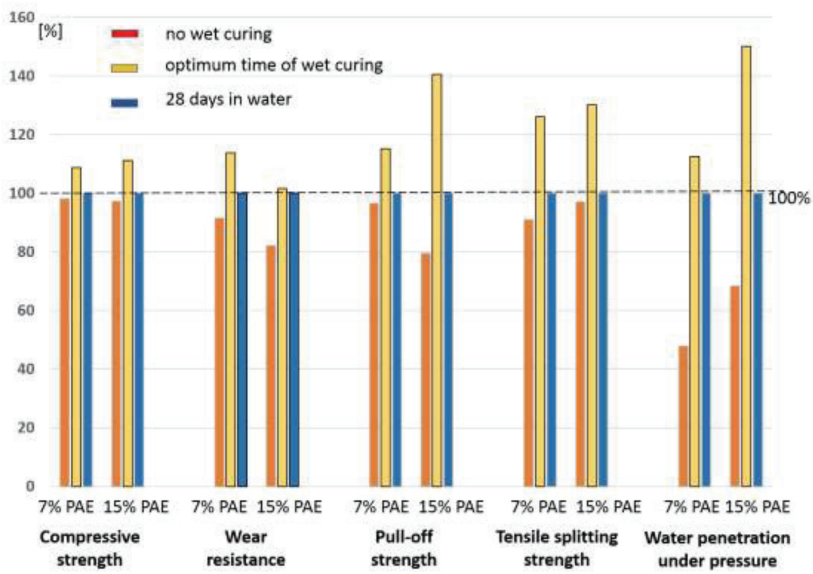

Figure 11. The influence of curing conditions on the properties of 28-days old PCC modified with PAE (red bars - no curing, yellow bars - optimum time of wet curing, blue bars - 28 days in water)

The optimum effect of mixed curing conditions is also visible in the microstructure of tested materials. An example of microstructure differences between specimens with 15% of EP cured in water during 14 days and then dry cured (fig. 12 a) and the same mix specimens cured in dry conditions all the time is visible on the SEM images 12 a and b. The microstructure on image a (curing conditions close to optimum) is more homogeneous with uniformly distributed CSH and polymer phase, and with lower content of smaller portlandite crystals. On the image b portlandite crystals are higher and not uniformly distributed and the microstructure could be interpreted as more porous. Those observations confirm the conclusion about the positive effect of longer wet curing on polymer cement concrete properties.

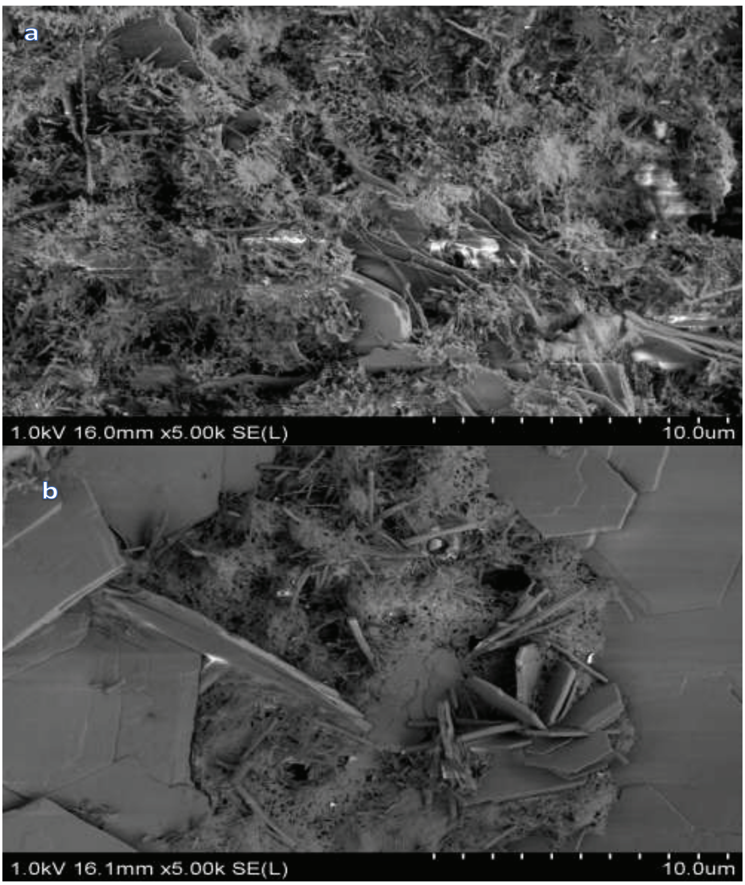

Figure 12 SEM analysis of microstructure of 28-days old polymer-cement concrete with 15% of EP after different regimes of curing: a) 14 days in wet conditions (water) and 14 days in dry conditions; b) 28 days in dry conditions

Broader research was carried out in the area of evaluation of carbonation resistance of PCC as a function of curing scenario. The results are shown in table 5.

It could be observed that for both types of polymers there is an optimum time of water curing from the point of view of resistance carbonation (fig. 12). Models shown on the figure indicates that even the quite long time of water curing (app. up to 15 days) is not harmful in view of carbonation depth. The results of carbonation measurement were used also for verification of hyperbolic carbonation model developed by author for different types of concrete. Traditional models treat the phenomenon of carbonation as process unlimited in concrete space and unlimited in time. It is assumed that the end of carbonation is related only to the exhaustion of available reagents. Important issue is the diffusion of CO_2 into the deeper layers of concrete which depends not only on the concentration gradient but also on the concrete microstructure.

Table 5 Depth of carbonation for OPC and PCC with both types of polymers after exposure in 4%CO_2 atmosphere up to 70 days. The age of specimens on the beginning of test was 28 days.

Polymer content, % of cement mass	**Time of water curing, days**	**Depth of carbonation, mm**											
		OPC				PAE concrete				EP concrete			
		time of exposition in 4% of CO_2, days											
		0	56	63	70	0	56	63	70	0	56	63	70
0	0	0	0.4	0	1.7								
0	5	0	5	6.5	7.4								
0	7	0	4.2	4	5.9								
0	27	0	6.7	9.6	10.8								
7	0					0	2	3.2	3.4	0	7.6	9.9	9.5
7	5					0	0	0.1	0.1	0	5.6	7.1	6.7
7	7					0	0	0.1	0.1	0	2.5	2	2.8
7	27					0	0	0.1	0.3	0	3.1	3	3.4
15	0					0	0	0.5	2.1	0	4	6	6
15	5					0	0	0	1.1	0	1.5	3.2	4.5
15	7					0	0	0	0.5	0	1	1.5	3
15	27					0	0	0	1.5	0	1.5	2.3	4.1

The models based on the Fick`s law assume that the diffusion takes place in concrete pores which will not change over time. This is a significant simplification of carbonation process description. The effect of carbonation is a decrease in capillarity, which leads to decrease of diffusivity over the time. This nature of the phenomenon was mentioned by Bakker in 1988 [4] and later by Hergenröder (1992) [14] Nilsson (1996) [23] and Fagerlund (1992) [11]. Such approach to the carbonation process was also developed and published by the author of this paper [7,8,9,40,41]. Abovementioned articles by Czarnecki, Woyciechowski and al. conclude that concrete carbonation can be described with a hyperbolic model of carbonation depth progress in time of exposure (reciprocal square root of time), which has asymptotic value parallel to the time axis. This asymptote is a limit of carbonation depth. Some other material and technological factors could be involved in the model. Such an approach was used for modeling results shown in the table 5, taking into consideration also the time of curing in water as a polynomial factor in model. The effect is shown on figure 13.

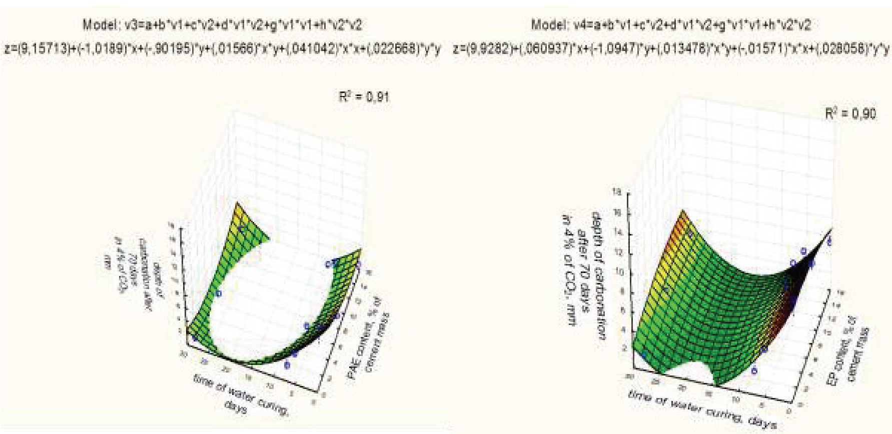

Figure 13. Depth of carbonation after 70 days of exposure in 4% CO_2 atmosphere as a function of time of water curing and polymer content

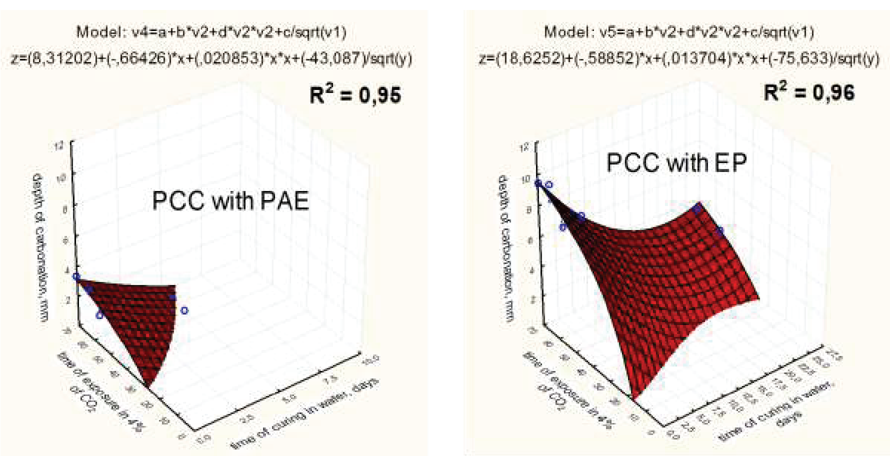

Figure 14. Models of carbonation progress in time for PCC concretes with PAE and EP polymers

The developed models fit to the experimental results with high determination coefficient (0.95 - 0.96). For both of tested polymer-cement concretes the hyperbolic model is adequate which indicates that the nature of the carbonation phenomena is similar in cement and polymer-cement concrete. The models confirm also the strong influence of wet curing on depth of carbonation of PCC, especially an unfavorable effect of too short wet curing period (fig. 14).

5. CONCLUSIONS

The conducted research allows to formulate the following conclusions:

• the optimum time of water curing of the investigated PCC ranged from 5 to 14 days, which is longer than recommended by most of the literature sources;

• the optimum time of curing depends on the polymer type and content;

• all tested PCC properties are sensitive to the way of curing, with the largest impact of this process on the results of water penetration under pressure test;

• each of the considered properties can be used as a measure for PCC curing effectiveness;

• the results confirmed the negative impact of the extension of wet curing of PCC beyond 14th day on its properties, which is probably due to the disturbance of polymer hardening process in water;

• the progress of polymer-cement concrete carbonation in time could be described by hyperbolic model similar to the models developed earlier by author for cement concrete.

The tests were carried out for two substantially different polymer binders (pre-mix, aqueous dispersion of acrylic polymer vs. post-mix, liquid epoxy resin), with low and medium content of the polymer in the binder. The results of evaluation of curing effectiveness are in all cases similar. The results shown in the paper do not allow for establishing of the very precise rules of PCC curing. The optimum time of water curing exceeds 5 days but the accurate value depends on type and content of polymer in the binder. Further studies, particularly on the intermediate curing scenarios, but also for the higher contents of polymers and other kinds of the polymer modifiers are necessary. Such comprehensive approach should give the bases for general guidelines for the selection of the optimum time of water curing for PCC, taking into account both material aspects and conditions of the works on site.

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There is no conflict of interest for this publication

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Figures:

Figure 13. Depth of carbonation after 70 days of exposure in 4% CO_2 atmosphere as a function of time of water curing and polymer content
Figure 14. Models of carbonation progress in time for PCC concretes with PAE and EP polymers

Tables

Table 1. Compositions of concrete mixes [kg/m^3]
Table 2. Concrete testing program
Table 3 Range of relative standard deviations for all tested properties in all series
Table. 4 Relative differences between maximum and minimum values of concrete property tested after 28 days, obtained after various water curing time
Table 5 Depth of carbonation for OPC and PCC with both types of polymers after exposure in 4%CO_2 atmosphere up to 70 days. The age of specimens on the beginning of test was 28 days

WPŁYW PRZEBIEGU PIELĘGNACJI NA WŁAŚCIWOŚCI BETONU POLIMEROWO-CEMENTOWEGO

Słowa kluczowe: Kompozyty, polimery, beton polimerowo-cementowy, pielęgnacja

STRESZCZENIE

Ogólne normy i zalecenia dotyczące PCC sugerują naprzemienne warunki pielęgnacji: począwszy od warunków mokrych dla skutecznej hydratacji cementu portlandzkiego, a następnie warunków suszenia na powietrzu korzystnych z uwagi na utwardzanie polimerów. Często akceptowany reżim pielęgnacji PCC obejmuje 5 dni w warunkach mokrych, a następnie suszenie na powietrzu, ale nie jest to optymalny sposób. Celem badania była optymalizacja scenariusza pielęgnacji dla PCC z dwoma rodzajami polimerów: dwuskładnikową żywicą epoksydową i wodną dyspersją poliakrylanów. Jako kryteria oceny skuteczności pielęgnacji PCC przyjęto następujące właściwości eksploatacyjne: wytrzymałość na ściskanie, wytrzymałość na rozciąganie przy rozłupywaniu, wytrzymałość na rozciąganie powierzchniowe (metodą pull-off), odporność na ścieranie, głębokość wnikania wody pod ciśnieniem i odporność na karbonatyzację. Ustalono w wyniku badań, że optymalny czas pielęgnacji PCC w wodzie wynosi od 7 do 14 dni, jednak wskazana jest weryfikacja eksperymentalna w przypadku konkretnego składu PCC.

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