The Silesian University of Technology



APPLICATION OF POTENTIOSTATIC MEASUREMENTS ACCORDING TO PN-EN 480-14 IN ASSESSMENT OF THE EFFICIENCY OF REINFORCEMENT PROTECTION AGAINST CORROSION BY CONCRETE WITH ADDITION OF FLY ASHES

FNVIRONMENT

Katarzyna DOMAGAŁA a*, Andrzej ŚLIWKA a

^a PhD; Faculty of Civil Engineering, Silesian University of Technology, Akademicka 5, 44-100 Gliwice, Poland *E-mail address: *katarzyna.domagala@polsl.pl*

Received: 25.06.2017; Revised: 10.08.2017; Accepted: 14.09.2017

Abstract

Replacing part of the cement in the concrete with fly ash – products of solid fuel burning, requires testing, including the degree of the reinforcement protection against corrosion provided by concrete. The paper presents a comparison of the results of the tests of the effectiveness of corrosion protection of reinforced concrete with the addition of calcareous fly ash. Tests were conducted by two electrochemical methods. Results obtained by potentiostatic measurement on reinforcement inserts deposited in cylindrical specimens in accordance with EN 480-14 standard were compared with studies investigating the progress of reinforcement corrosion caused by chlorides in loaded and cracked test elements made of these concretes. Tests of the cracked elements reflect the behaviour of reinforced concrete construction during operation in adverse environmental conditions, classified as standard XD and XS exposure classes. Comparison of the tests indicates the potential for potentiostatic measurement to be used to current assessment of protective properties of concrete with mineral additives.

Streszczenie

Zastąpienie części cementu w betonie popiołami lotnymi – produktami spalania paliw stałych, wymaga przeprowadzania badań, w tym stopnia ochrony zbrojenia przed korozją, którą powinien zapewnić beton. W artykule przedstawiono porównanie wyników badań skuteczności ochrony antykorozyjnej betonu z cementu z dodatkiem wapiennych popiołów lotnych względem stali zbrojeniowej. Badania przeprowadzono metodami elektrochemicznymi. Wyniki uzyskane za pomocą pomiaru potencjostatycznego na wkładkach zbrojeniowych umieszczonych w próbkach cylindrycznych zgodnie z normą EN 480-14 porównano z badaniami postępu korozji zbrojenia wywołanej chlorkami w obciążonych i zarysowanych elementach próbnych wykonanych z tych betonów. Badania elementów zarysowanych odzwierciedlają zachowanie konstrukcji żelbetowej podczas pracy w niekorzystnych warunkach środowiskowych, charskteryzowanych jako klasy ekspozycji XD i XS. Porównanie badań wskazuje na możliwość zastosowania pomiaru potencjostatycznego do bieżącej oceny właściwości ochronnych betonu z dodatkami mineralnymi.

Keywords: Electrochemical tests; High calcium fly ash; Potentiostatic measurements; Protective properties of concrete; Reinforcement corrosion.

1. INTRODUCTION

Manage of solid fuel burning products – mostly hard coal and brown coal – is an important economic and technical problem. One of the products of combustion are fly ashes, which can be a useful mineral additive to concrete. Ashes that do not meet the standard criteria are secondary materials but research is undertaken on the wider implementation of them for the production of concrete. Replacing part of the cement in the concrete with fly ash requires testing, including the degree of the reinforcement protection against corrosion provided by concrete. Determination of these characteristics allows for predicting the durability of reinforced concrete structures made from materials modified with coal combustion products. The results of published studies are not directly comparable due to the different types of ash used and the percentage of cement used as well as the different concrete recipes, curing conditions, and the way the experiment is conducted. Most of the results confirmed that the use of ashes in appropriate proportions does not impair the protective properties of concrete to reinforcing steel [1, 2, 3, 4, 5] and improves the tightness of concrete [4, 6]. In some studies conducted in the environment heavily contaminated with carbon dioxide and chlorides, accelerated corrosion of reinforcement by the introduction of silica fly ash replacing part of the cement was found [3, 7, 8]. In addition, the use of common building chemicals poses a risk of antagonistic action and, as a consequence, deterioration of protective properties to reinforcing steel. Composition of fly-ashes can also vary considerably depending on the source and fuel used. Due to this should be conducted current control of protective properties. In the case of control tests, the speed of obtaining results is important.

Potentiostatic test according to PN-EN 480-14 allows for fast results, but test conditions deviate from the natural conditions in which the construction works. This can have an impact on the proper reasoning about proper protection in real construction. It should answer the question whether these tests are reliable and can be to draw conclusions about the protective properties of concrete containing fly ash cement.

Therefore, in order to verify the protective properties of concretes containing fly ash cements were examined [9]. Rapid selection tests were performed on standard test elements according to standard [10]. Then corrosion rate of reinforcing steel caused by the impact of a 3% solution of sodium chloride on reinforced beam elements was determined by linear polarization. Samples for both tests were made of concrete on cement with different contents of high calcium fly ash (Tab. 1). Concrete test elements made of Portland cement were also tested, which were reference level during the study.



Types of cement towards electrochemical measurements used in the experiments

| Series | Cement | High calcium fly ash [% by weight] |
|--------|-------------|---------------------------------------|
| Ι | CEM I 42.5R | - |
| II | CEM II/A-W | 15 |
| III | CEM II/B-W | 30 |
| IV | CEM IV/B-W+ | 50 |
| V | CEM IV/B-W | 50 |

2. CORROSION SUSCEPTIBILITY OF THE REINFORCEMENT IN CONCRETE

- POTENTIOSTATIC TESTS

2.1. Technique of potentiostatic tests

Specimens for potentiostatic tests were prepared in accordance with PN-EN 480-14 in the form of cylin-

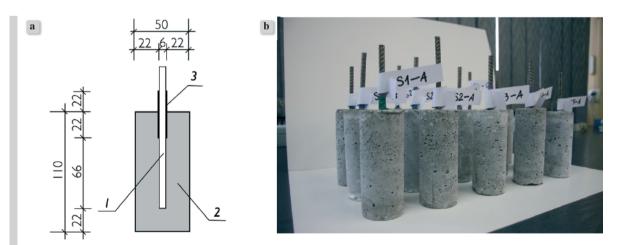


Figure 1.

Potentiostatic measurements: a) the test element scheme: 1 – working electrode (reinforcing bar), 2 – concrete, 3 – protective coating of plastic, b) photography of test elements

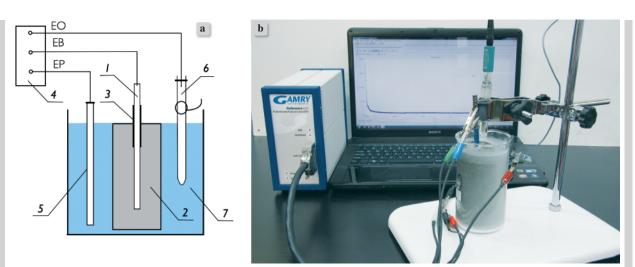
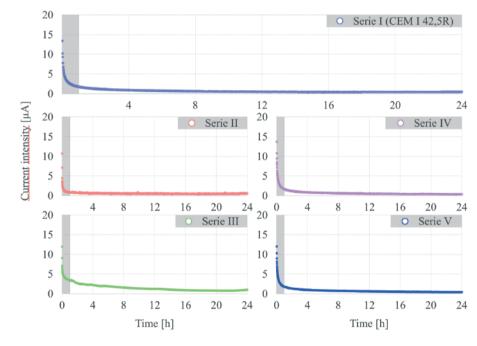


Figure 2.

Potentiostatic measurements: a) diagram of the measuring system: 1 -working electrode (reinforcing bar), 2 -concrete, 3 -protective coating of plastic, 4 - potentiostat, 5 - counter electrode, 6 -reference electrode, 7 - electrolyte (saturated solution of calcium hydroxide), b) view of the test stand



The current intensity changes in representative element of all series

ders 110 mm in height and 50 mm in diameter, centrally reinforced with a ribbed rod of 6 mm diameter class B steel – Fig. 1. Research began 88 days after sample preparation – after maturation and completion of hydration of cement [9].

Potentiostatic studies were carried out in accordance with standard [10] – Fig. 2. The reference electrode was a silver chloride electrode and an counter electrode made of stainless steel. Based on the measurements, a maximum current value *I* between 1 and 24

hours was determined. Taking into account the active area of the test electrode, the current density i was calculated.

2.2. Results of potentiostatic testing of corrosion susceptibility

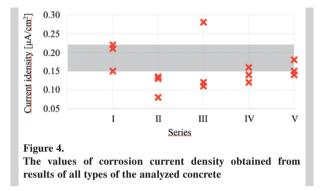
The results of potentiostatic measurements are current intensity changes, which are shown in Fig. 3 for elements of series $I \div V$.

Figure 3.

ENGINEERIN

CIVIL

The values of current density for the specimens of series $II \div V$ were compared with the results for the reference specimens of series I. The overall results from the potentiostatic testing of corrosion susceptibility are presented in Fig. 4.



The average values of current density for the specimens of series $II \div V$, made of concrete with cement containing high calcium fly ash were found to be lower than the average values of current density for the specimens of the reference series I referred to as the comparative level.

After the measurements were completed, the test elements were split. The surface of the reinforcement in each case showed no signs of corrosion. Photos of representative splited elements are shown in Fig. 5.

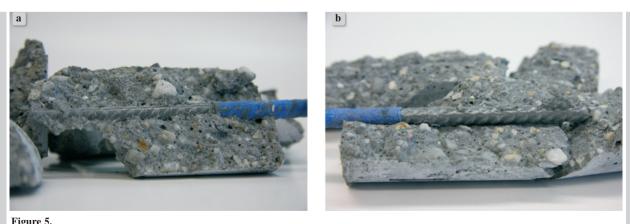
It was observed that using high calcium fly ash of different content (series $II \div V$) in cement did not result in reduction of the protective properties of concrete to the reinforcement.

3. TESTS OF PROGRESS OF REIN-FORCEMENT CORROSION CAUSED BY CHLORIDES

3.1. Test elements and the course of measurements of reinforcement corrosion rate

The development of reinforcement corrosion was determined in loaded and cracked test elements. Test elements were made of concrete containing high calcium fly ash. To induce corrosion of reinforcement to determine the rate of corrosion processes the elements were periodically wetted with 3% solution of sodium chloride. The measurements were performed under conditions similar to the original conditions existing in the structure exposed to chlorides. Beams of 50 x 100 x 600 mm dimensions were prepared to determine the rate of corrosion processes. They were reinforced with ribbed bar of 6 mm diameter of steel class B. Concrete made of multi-component cement containing high calcium fly ash in 3 following proportions - 15% (series II), 30% (series III) and 50% (series IV and V) by mass of cement were used in the test elements. The test results were compared to the values of measurements of test elements of reference concrete made of Portland cement (series I). Platinised titanium rod of 1.6 mm diameter was embedded in each test element as the counter electrode.

The measurements were carried out using the polarization method. The corrosion rate was determined as the corrosion current density. The corrosion density was measured every few weeks in the crack and in the selected uncracked points along the reinforcement axis. The measuring points were so located along the beams to consider the differences in the migration of aggressive substance through the cover between cracked tension zone and loaded and unloaded zones



Photos of representatives elements after tests: a) series I, b) series III

of elements – Fig. 6. Cracks were obtained by loading the simply supported beam elements with a concentrated force. The elements were loaded using the individually prepared tie-beam system [11]. The load was increasing until the crack from 0.05 to 0.1 mm in width appeared on the element surface.

On every test element were located 5 measurement points – one in the crack – most exposed to the effort

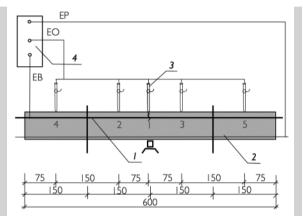


Figure 6.

Diagram of polarization measurement performed in selected points of the test element: 1 – working electrode (reinforcing bar), 2 – counter electrode (titanium rod covered with platinum), 3 – reference electrode (Ag/AgCl), 4 – potentiostat

Table 2.

| The fundamental results of the electrochemical measurements |
|---|
|---|

and to aggressive substances section, two on both sides of the crack – zone, in which reinforcement is protected by stretched but uncracked concrete, and two in no loaded zone.

The full way of inducing corrosion and measuring is described in [9, 11, 12]. The wetting was conducted till the corrosion current density reached the value of $i_{cor} 2 \div 3 \,\mu\text{A/cm}^2$ in the crack of test elements made of reference concrete of series I with Portland cement. This value indicated the moderate level of the reinforcement corrosion according to the paper [4]. From that moment, only the electrochemical measurements were conducted under conditions of developing corrosion of the reinforcement without the impact of the aggressive solution. The tests were conducted for 40 weeks.

3.2. Test results of reinforcement corrosion rate

The results of each electrochemical measurement were obtained in the form of computer printouts of cathodic and anodic polarization, as well as the values of corrosion potential E_{cor} and corrosion current density i_{cor} determined in Echem Analyst software, using the method of polarization resistance. The main results are presented in Table 2.

| | Stage | Time [week] | i_{cor} [µA/cm ²] | | | | | | | | | | | | | | |
|--------|-------|----------------|---------------------------------|------|---------|------|------|---------|------|------|---------|------|------|---------|------|------|------|
| Series | | | Point 1 | | Point 2 | | | Point 3 | | | Point 4 | | | Point 5 | | | |
| | | | Elements | | | | | | | | | | | | | | |
| | | | 1 | 2 | 3 | 1 | 2 | 3 | 1 | 2 | 3 | 1 | 2 | 3 | 1 | 2 | 3 |
| Ι | 0 | 1 | 0.01 | 0.06 | 0.03 | 0.46 | 0.06 | 0.35 | 0.38 | 0.01 | 0.52 | - | - | 0.00 | - | - | 0.00 |
| | Ι | 2 | 0.65 | - | 0.13 | 0.08 | 0.14 | 0.35 | 0.46 | 0.22 | 0.54 | 0.00 | 0.00 | 0.07 | 0.00 | - | 0.00 |
| | R | 23 | - | 2.03 | 3.06 | 2.12 | - | 3.18 | 1.44 | 2.10 | 2.89 | 2.01 | 2.11 | 2.05 | - | 2.05 | 2.79 |
| | | 40 | 1.46 | 1.60 | 1.16 | 0.84 | 1.73 | - | - | 2.01 | 2.71 | 0.73 | 0.98 | 0.47 | 2.07 | 2.05 | - |
| II | 0 | 1 | 0.02 | 0.03 | 0.01 | 0.01 | 0.03 | 0.02 | 0.03 | 0.06 | 0.03 | 0.01 | 0.01 | 0.01 | 0.05 | 0.05 | 0.02 |
| | Ι | 2 | 0.01 | - | 0.02 | - | - | 0.00 | 0.01 | 0.03 | 0.02 | 0.06 | - | 0.01 | - | - | - |
| | R | 23 | - | 0.10 | 0.01 | - | 0.06 | - | - | 0.03 | - | - | 0.01 | - | - | 0.03 | - |
| | | 40 | 0.07 | - | - | 0.17 | 0.05 | - | - | 0.05 | 0.05 | - | 0.04 | - | 0.03 | - | - |
| III | 0 | 1 | 0.04 | - | - | - | - | - | 0.03 | 0.01 | - | 0.05 | - | 0.01 | 0.02 | 0.03 | - |
| | Ι | 2 | 0.01 | 0.01 | 0.01 | 0.04 | - | 0.03 | 0.03 | 0.02 | 0.01 | - | 0.01 | - | 0.01 | 0.05 | 0.01 |
| | R | 23 | 0.24 | - | 0.02 | 0.13 | 0.25 | - | 0.17 | - | - | 0.04 | 0.06 | - | 0.02 | - | - |
| | | 40 | 0.05 | - | 0.26 | 0.09 | - | - | 0.14 | 0.08 | 0.19 | 0.01 | - | - | 0.06 | - | - |
| | 0 | 1 | 0.01 | 0.09 | 0.01 | 0.01 | 0.02 | 0.03 | 0.01 | 0.02 | 0.06 | - | 0.00 | 0.05 | 0.02 | 0.01 | 0.02 |
| IV | Ι | 2 | 0.07 | 0.05 | 0.04 | 0.00 | 0.01 | - | 0.00 | 0.01 | 0.05 | 0.21 | 0.01 | 0.03 | 0.11 | - | - |
| | R | 23 | 0.13 | 0.09 | 0.01 | - | 0.05 | - | - | 0.05 | - | 0.06 | 0.04 | - | - | 0.08 | - |
| | | 40 | 0.04 | 0.06 | 0.08 | 0.10 | - | - | 0.13 | 0.03 | - | 0.04 | 0.09 | - | - | - | 0.02 |
| v | 0 | 1 | 0.01 | 0.02 | 0.04 | 0.05 | 0.03 | 0.02 | 0.09 | 0.04 | 0.09 | - | _ | 0.11 | 0.18 | - | 0.16 |
| | Ι | 2 | 0.06 | - | 0.07 | 0.10 | 0.02 | - | - | 0.05 | - | 0.05 | 0.01 | - | 0.28 | - | - |
| | R | 23 | 0.02 | 0.02 | 0.10 | 0.02 | - | 0.08 | - | - | 0.05 | 0.15 | - | 0.01 | 0.02 | 0.34 | 0.09 |
| | | 40 | 0.03 | - | 0.09 | 0.03 | 0.36 | 0.20 | 0.04 | - | 0.23 | 0.04 | _ | 0.02 | 0.34 | - | 0.08 |

The results obtained in series $II \div V$ were referred to the values estimated on reference series I - made of Portland cement. For cracked elements, the obtained values of corrosion current density in the crack (Tab. 2) indicate that concrete of series II÷V maintained its protective properties with respect to reinforcing steel. The measurement results of points 2 and 3 confirmed as well the good protective properties of concrete with cement containing high calcium fly ash. The developed corrosion was found in the reinforcement inserts embedded in reference concrete of series I, while the reinforcement present in concrete of series II ÷ V made of cement containing high calcium fly ash was well protected. The values of corrosion current density usually did not exceed the assumed threshold value 0.20 µA/cm². Very good protective properties of the analyzed types of concrete were also confirmed by the results of points 4 and 5 in the specimens.

4. CONCLUSIONS

The efficiency of reinforcement protection against corrosion by concrete with addition of fly ashes were conducted using two individual methods: (i) the potentiostatic method for the cylindrical concrete specimens with the centrally located rebar and (ii) the method of polarization resistance conducted during 40 weeks of corrosion rate induced by chlorides on the loaded and cracked beam elements.

On the basis of the both conducted electrochemical tests and the analysis of obtained results it can be concluded that high calcium fly ash used as the component of cement didn't get worse the protective properties of concrete with respect to the reinforcement in comparison to reference concrete with Portland cement.

In case of tests carried out according to standard method, cements containing high calcium fly ash have been reported to improve the protective properties of concrete to reinforcing steel in comparison to Portland cement concrete. As the fly ash content increases, the protective properties slightly deteriorate but are not worse than in case of Portland cement. Corrosion-induced chloride rate measurements made on beam elements by linear polarization method also showed better protective properties of concrete on cement containing fly ash. Due to the very high variability of the linear polarization results, it is much more difficult to observe the effect of the ash content on the protective properties and thus the corrosion rate, but there is a tendency to increase the corrosion current density value in case of higher ash content in the cement.

The obtained results indicate the possibility of using a fast method, such as the standard method [10], for the current assessment of the protective properties of concrete. Both applied research methods allowed the same conclusions to be drawn, indicating the possibility of their replacement. Of course, the number of tested samples is not great and further studies are needed, taking into account other factors, to verify presented conclusions.

REFERENCES

- [1] Ampadu K.O., Torii K. (2002). Chloride ingress and steel corrosion in cement mortars incorporating low-quality fly ashes. *Cement and Concrete Research*, *32*, 893–901.
- [2] Saraswathy V., Muralidharan S., Thangavel K., Srinivasan S. (2003). Influence of activated fly ash on corrosion-resistance and strength of concrete. *Cement* and Concrete Composites, 25, 113–120.
- [3] Jiang L., Liu Z., Ye Y. (2004). Durability of concrete incorporating large volumes of low-quality fly ash. *Cement and Concrete Research*, *34*, 1411–1413.
- [4] Kayali O., Zhu B. (2005). Chloride inducted reinforcement corrosion in lightweight aggregate highstrength fly ash concrete. *Construction and Building Materials*, 19, 327–336.
- [5] Pacewska B., Bukowska M., Wilińska I., Swat M. (2002). Modification of the properties of concrete by a new pozzolan. A waste catalyst from the catalytic process in a fluidized bed. *Cement and Concrete Research*, *32*, 145–152.
- [6] Hossain K.M.A., Lachemi M. (2004). Corrosion resistance and chloride diffusivity of volcanic ash blended cement mortar. *Cement and Concrete Research*, 34, 695–702.
- [7] Montemor M.F., Simões A.M., Salta M.M. (2000). Effect of fly ash on concrete reinforcement corrosion studied by EIS. *Cement and Concrete Composites*, 22, 175–185.
- [8] Montemor M.F., Cunha M.P., Ferreira M.G., Simões A.M. (2002). Corrosion behaviour of rebars in fly ash mortar exposed to carbon dioxide and chlorides. *Cement and Concrete Composites*, 24, 45–53.
- [9] Śliwka A., Domagała K., Zybura A. (2013). Evaluation of protective properties of concretes made of cements with calcareous fly ash with respect to reinforcing steel. *Roads and Bridges – Drogi i Mosty*, 12(2), 237–250.

- [10] PN-EN 480-14: (2008). Domieszki do betonu, zaprawy i zaczynu. Metody badań. Część 14: Oznaczanie podatności korozyjnej stali zbrojeniowej w betonie za pomocą potencjostatycznego badania elektrochemicznego (Admixtures for concrete, mortar and grout. Test methods. Part 14: Determination of the effect on corrosion susceptibility of reinforcing steel by potentiostatic electro-chemical test).
- [11] Domagała K., Śliwka A., Zybura A. (2013, Sep. 4-6). Protective properties of reinforcing steel in concrete containing fly ash. Central European Congress on Concrete Engineering "Concrete Structures in Urban Areas", Wrocław, Poland, 292–295.
- [12] Domagała K., Śliwka A., Zybura A. (2014). Przebieg korozji zbrojenia chronionego betonem na cemencie zawierającym popiół wapienny (The process of corrosion of the reinforcement protected with concrete made of cement containing high calcium fly ash). *Ochrona przed Korozją*, 57(1), 21–27.