Impact of Concrete´s Curing Process on its Biocorrossive Resistance

V. Ondrejka Harbulakova, A. Estokova, A. Luptakova and M. Smolakova

***Abstract*—**The properties of the concrete are influenced by many factors. Selection of the proper curing of the concrete and cement mortars belong to the very important determinative factor of the required characteristics of the construction material. The air-treated concrete samples (XCA) and in water-treated concrete samples (XCW) were exposed to the concentrated and diluted bacterial environment. The comparative study proceeded also with control media without bacteria. Both sets of samples were immersed in three types of liquid media (medium with sulfur-oxidizing bacteria *Acidithiobacillus thiooxidans*, diluted bacterial medium and control medium).

Experiment run in laboratory condition during 270 days when a set of concrete samples were exposed to the different impact of bacterial aggressive media and evaluation of its properties at the end of the experiment were assessed in accordance to curing condition of the sets of samples. The results indicated a better resistance of the air-treated concrete samples compared to the in water-treated concrete samples to the concentrated bacterial environment. Any significant difference between the variable treated samples was observed in case of samples in control media (without bacteria). The findings are linked to the well-known positive concrete process of hydration proceeded in concrete matrix exposed to the liquid environments.

***Keywords*—**Biodeterioration, Correlation analysis, Dissolution, Leaching.

I. INTRODUCTION

THERE are many studies concerned on the investigation of biocorrosion processes as a factor of deterioration of concrete [1], [2], [3], [4]. While some of the studies deal with economy aspects [5], some deals with new concretes contain different supplementary material [6], [7] or geopolymers [8] some of them deals with microbial corrosion regarding the health risk for the worker of water system operation [9], [10]. The properties of the concrete are influenced by many factors. One of the important attribute among increasing the durability, resistance against aggressive environments or abrasion processes, increasing the strength or improve the resistance to freeze is proper curing of the concrete and cement mortars which is presented in the papers [11], [12], [13], [14], [15]. The results in a paper by Tan and Gjorv [16] pointed to the fact that penetration of aggressive environment and the compressive strength are attributes which are affected by curing temperature. On the other hand, it has no impact on water penetration of concrete. As it is applied in [15], [17] there is minimal curing period reaching up to 70% of the specified compressive strength stated in the American Concrete Institute (ACI) Committee 301.

There are many papers dealing with curing effectiveness and investigations on how it affects different concrete properties. Nahata et al. [15] compared the selected properties of the tested cement mortars prepared in accordance with ASTM [17] and mortars prepared by regular Indian field material. Based on the results it was concluded that this raw material (Indian sand) is not suitable for testing of curing compound. Importance of curing condition of high strength concrete is also presented by Paulik [11]. One of the goals of the study was to characterize unsuitable curing of the concrete on the construction site during the construction.

There are many different approaches to the investigation of deterioration of building material based on mathematical principles which are often used in civil engineering practice, as a mathematical assessment of failures, statistical evaluation, simulation, modelling and determination of prediction of the trend of deleterious processes of studied material [2], [18], [19].

Many methods used for curing of concrete depending on which properties of material/construction which need to be achieved. Concrete becomes stronger, more impermeable or more resistant to abrasion when the proper curing is applied [20]. To keep a uniform temperature in concrete and also to preventing loss of the moisture from the concrete the method called ponding is widely used. The most effective method of curing with water is the total immersion of the concrete samples, which is often used in the laboratory´s tests. The water used for test has to be without any compounds which lead to the formation of stains and discolor of the concrete/mortars samples [20], [21].

Wet covering with burlap, cotton mats or other moisture-retaining fabrics is also very often used for curing of concrete [20].

The investigation of the different cured concrete samples exposed to the microbially influenced corrosion during 9 months in three different environments is presented in the paper. Correlation analysis and X-ray fluorescence analysis

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(XRF) were used to gain the data which point to the trend of the deleterious process. The concentration of calcium and silicon into the liquid environment was measured during the whole tested period and based on its comparison the leaching trend of Ca or Si each other and well as its comparison based of leaching trend of Ca/Si in the same liquid environment the conclusion about the biocorrosion processes were formulated.

II. Material and methods

Investigation of a potential different predisposition of concrete samples to biocorrosion was researched in relation to the different 28-day curing. Two different processes of concrete´s curing were considered: (1) in tap water, and (2) under air. The samples treated under air (XCA samples) were covered by polyethylene foil to protect them against excessive carbonation and kept for 28 days at laboratory conditions at constant temperature 22 °C. The samples cured in a water environment (XCW samples) were immersed in tap water in water tanks over the same period of 28 days. The set of three samples of the same concrete mixture, with a density of concrete 2390 $kg.m^{-3}$, was investigated for each curing process in consequent bio-corrosion experiments.

The bio-corrosion experiments were simulated in laboratory conditions using the sulfur-oxidizing bacteria *Acidithiobacillus thiooxidans*, which are bacteria responsible for the deterioration of building materials (in cooperation whith the other species of bacteria). The bacterial cultures of *A. thiooxidans* were provided by the Institute of Geotechnics of Slovak Academy of Science in Košice, Slovakia. The bacteria were isolated from the acid mine water runing from the former shaft at the Smolník deposit in the eastern of Slovakia.

The concrete prisms, after the 28-day treatment, were after removing of fine surface´s impurities immersed for 2 hours to distilled water, and then slightly dried by filter paper. Subsequently, they were immersed in the ethanol for another 24-hour period to ensure sterile conditions. The samples were then placed on sterile filter paper in an aseptic box. After 2 hours of drying at room temperature, they were dried to a constant weight at 105 °C in an oven. Dry concrete samples were consequently exposed to bacterial and control environments by immersing them to three liquid media I., II. (bacterial ones with different ratio of bacteria) and III. (control abiotic - without bacteria). The experiments performed for 270 days in glass containers with the liquid-to-solid ratio 10:1, under 28-30 °C to secure the optimal growth of bacteria. The aerobic conditions, necessary for the bacterial growth, were ensured in the experiments.

The concentrated bacterial medium I (with pH=4) consisted of the bacterial inoculum (*A. thiooxidans*) and of a cultivation medium in proportions 1:4. Cultivation medium by Waksman and Joffe was of the following composition: 0.25 g/L $CaCl_2.6H_2O$, 0.2 g/L $(NH_4)_2SO_4$, 3.0 g/L K_2HPO_4, 0.5 g/L $MgSO_4.7H_2O$, traces of $FeSO_4.7H_2O$, 10.0 g/L elementary sulphur and up to 1000 mL distilled water. Diluted bacterial medium II was prepared by diluting the concentrated one with distilled water 1:1. The control medium III contained only the cultivation medium without any bacterial culture and represented an abiotic environment in biocorrosion experiment. The activity of bacteria was checked periodically in the experiment.

The liquid media were analysed in terms of concentration of the dissolved Ca^{2+} and Si^{4+} ions periodically during the whole experiment. Chemical composition of the leachates was determined by X-ray fluorescence analysis (XRF). SPECTRO iQ II (Ametek, Germany) with SDD silicon drift detector with a resolution of 145 eV at 10 000 pulses.

The differences in leaching trends of Ca and Si after exposing the different cured concrete samples to the different types of aggressive liquid media were evalauated using Pearson R_{xy} correlation which is the most widely used correlation statistic to measure the degree of the relationship between linearly related variables [22]. Excel 2016 software by Microsoft was used for the calculation of correlation coefficients. The calculated R_{xy} values are from the interval <-1,1>. If $R_{xy} = 1$, the correlation is full linear (perfect positive), if $R_{xy} = -1$ (perfect negative), then the correlation is inversely linear and if $R_{xy} = 0$, the pairs of values are fully independent (no relationship between the variables at all). That degree of the correlative closeness is defined as: medium, if $0.3 \leq | R_{xy} | < 0.5$; significant, if $0.5 \leq | R_{xy} | < 0.7$; high, if $0.7 \leq | R_{xy} | < 0.9$; and very high, if $0.9 \leq | R_{xy} |$.

Investigation of the aspect of curing and its evaluation by statistical analysis to gain a better overview of this phenomenon is presented in this paper.

III. Results and discussion

The first three pictures (Figures 1 - 3) show the trend in leaching of calcium from the concrete prepared according to the same standard recipe but cured in different conditions after its preparation. The air-treated samples (XCA) and in water-treated samples (XCW) exposed to the concentrated bacterial environment I are compared in Figure 1.

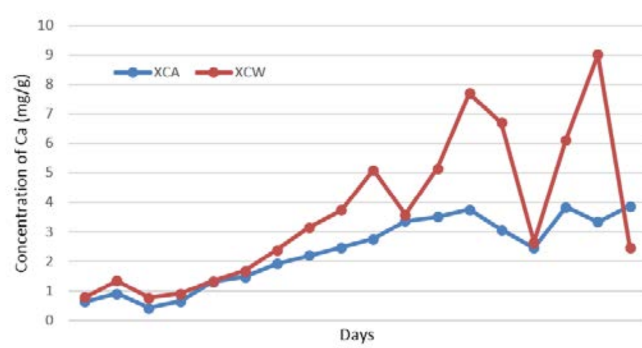

Fig. 1 Dissolving trend of calcium due to concentrated bacterial environment I

The dissolved amounts are presented in mg of the leached-out Ca^{2+} from 1 g of the concrete sample. As seen in Fig. 1, the amounts of Ca leached from the water-treated samples (XCW), due to the concentrated bacterial environment I, ranged in a wider interval and reached higher values than those from the under-air-treated samples (XCA). This was valid for all measurements during the experiment excepting from the final value. In the case of XCA, the trend of calcium leaching is more stable thus the samples showed greater stability in the concentrated bacterial environment I.

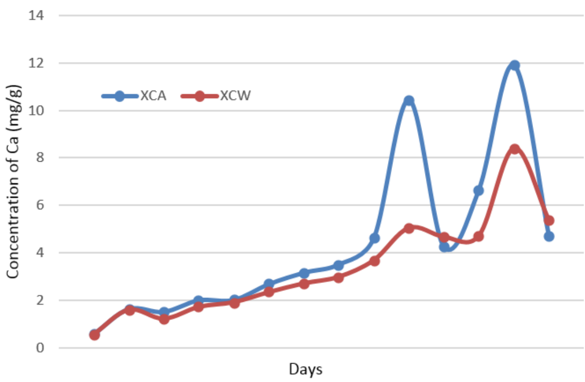

Fig. 2 Ca dissolving trend due to diluted bacterial environment II

The trend of calcium dissolution in the diluted bacterial medium II was similar for both sample types (Fig. 2). However, unlike the concentrated environment I, lower amounts of leached calcium were observed for XCW samples. Based on the dissolved amounts, the higher stability of the XCA samples was not confirmed.

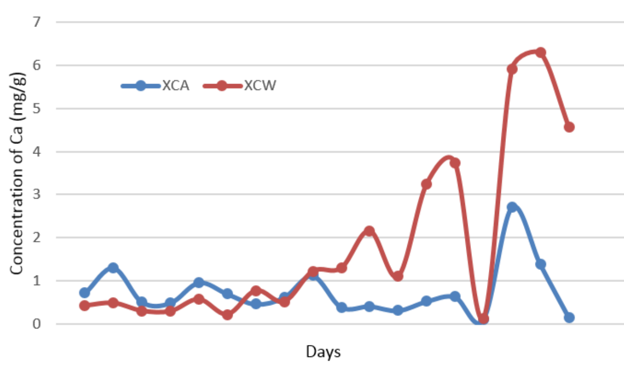

Fig. 3 Ca dissolving trend in control cultivation medium III

The lowest calcium concentrations from the liquid media analyzed were measured in a control medium without bacteria for both sample types, XCA and XCW. This has confirmed expectations of higher aggressiveness of both bacterial environments.The XCA sample again showed higher stability compared to the XCW sample as illustrated in Fig. 3.

The dissolution trend of silicone due to the concentrated bacterial environment I followed the trend of dissolution of calcium when the XCW sample reached higher concentrations of leached silicon (Fig. 4). This indicates a better resistance of the XCA sample compared to the XCW sample.

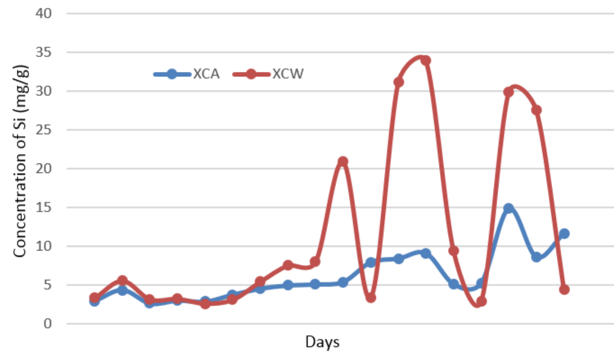

Fig. 4 Si dissolving trend due to concentrated bacterial environment I

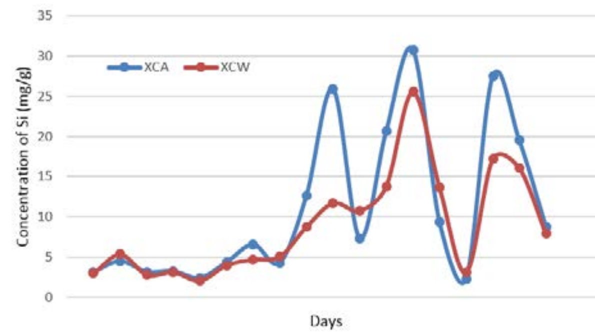

Fig. 5 Si dissolving trend due to diluted bacterial environment II

In the diluted bacterial medium II, similarly as in case of calcium, no significant difference was found regarding the Si^{4+} dissolved amounts in relation to the individual samples. The final silicon concentrations at the end of the experiment were almost identical as well as it can be seen in Fig. 5.

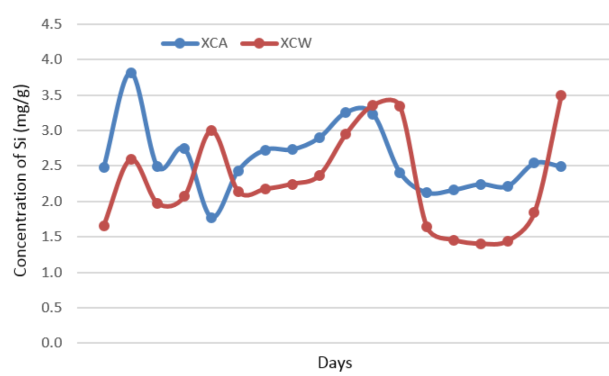

Fig. 6 Si dissolving trend in cultivation III

The cultivation medium was confirmed to be the least aggressive compared to both bacterial ones. The maximum measured concentration in the control medium was up to ten-time lower than that measured in the bacterial medium. Comparing the silicon leaching trends in the cultivation medium, it is not possible to unambiguously determine a more stable sample, although the final concentration in the solution, at the end of the experiment, was lower for the XCA.

Table 1 presents the calculated Pearson's correlation coefficients used to investigate the dependence among calcium and silicon leaching, respectively, in the individual aggressive media.

Table 1 Correlation between calcium and silicon leaching, respectively, in bacterial and control media

	R_{xy} (XCA/XCA)		
	I/II	I/III	II/III
Ca	0.73	0.11	0.16
Si	0.68	- 0.15	-0.08
	R_{xy} (XCW/XCW)		
	I/II	I/III	II/III
Ca	0.66	0.03	0.19
Si	0.88	-0.11	0.13

The calculated low correlation coefficients do not indicate any relationship between calcium leaching in the bacterial and control media, whether it is the concentrated or diluted bacterial environment (Table 1). The same finding was also found for silicon, as expected from the results presented in Fig. 1 - 6. Only the trend of leaching of calcium between the two bacterial environments in case of XCA samples and silicon in case of XCW samples could be considered important.

Table 2 Correlation between XCA and XCW samples leaching

	R_{xy} (XCA/XCW)		
	I	II	III
Ca	0.44	0.90	- 0.09
Si	0.65	0.90	- 0.56

To evaluate the behaviour of different cured samples, the smallest differences in the leaching of the specimens were recorded in the diluted bacterial medium, where high full linear correlations were found for the leaching of both ions (Table 2). On the contrary, an inverse correlation, even though low, was detected in case of the silicon leaching between XCA and XCW samples. In the concentrated bacterial medium I, the difference in the behaviour of the sample was significant. The different patterns of concrete specimen curing have been found in differences in the resistance of the samples to the concentrated bacterial medium, which has been demonstrated by the different trends in leaching of the monitored cement matrix elements.

IV. Conclusion

In the paper different cured concrete samples exposed to the microbially influenced corrosion during 270 days months in three different environments were tested and evaluated.

Experimental findings have clearly shown that less intense leaching of silicone and calcium from both types of concrete samples took place in the control medium containing only the cultivation solution. This confirmed the aggressiveness of the bacterial environment consisted of *Acidithiobacillus thiooxidans.* The biogenic sulfuric acid, produced by bacteria, was responsible for increased deterioration of the concrete samples under bacterial attack. This was manifested by dissolution of the main hydration products and consequently leaching or the dissolved products from the cement matrix. The results showed better resistance of air-treated concrete samples to concentrated bacterial environments compared to samples treated in water. That finding could be linked to the fact that among the positive concrete hydration processes running in water environment, the dissolving processes of hydration products can occur during this period as well. The behavior of the samples in the control medium without bacteria showed no significant difference between the samples treated in the variants. The further investigation is focused on the changes in mechanical properties of concretes such as absorbability and strength parameters.

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