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Original Research

Analyzing the Relationship between Chemical and Biological-Based Degradation of Concrete with Sulfate-Resisting Cement

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Abstract

External sulfate attack is detected on concrete construction exposed to soils, groundwater, seawater, or wastewater, and leads to serious damage to concrete elements. This paper describes an investigation of acidic corrosion caused by artificial sulphuric acid and biogenic sulphuric acid. The concrete prisms prepared from concrete-containing sulfate-resisting cement were exposed to an acid environment of different origin over a period of 3 months. The concentration of basic chemical elements such as calcium, silicon, iron, and aluminium were measured in liquid phases. Correlation analysis was used to evaluate dependencies and trends of leached-out amounts in these concentrations. Based on correlation coefficient, the intensity of dependency of the leaching trend was determined. A higher aggressiveness of sulfuric acid produced by sulfur-oxidizing bacteria was confirmed in terms of main concrete components' leaching.

Keywords: sulfate-resisting cement, sulfate attack, biocorrosion, chemical corrosion, leaching

Introduction

The origin of concrete deterioration caused by an aggressive environment is usually the result of a complex combination of dissolution and crystallization processes. Leaching in concrete is the process by which soluble material is extracted from the concrete by flowing medium, e.g., water or another solution. The water may flow over the concrete surface of percolate through cracks. It extracts sodium and potassium hydroxide from the concrete pore fluid, lowering the pH. Consequently, it leads to calcium hydroxide dissolving and to the decomposition of calcium silicate hydrate (C-S-H), ettringite, and AFm phases – a group of phases where monosulfoaluminate/monosulfate, monocarboaluminate/monocarbonate, hemicarboaluminate/hemicarbonate, and others – belong [1].

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Considering the crystallization of the cement material, the range of corrosion products forms during various stages of the corrosion processes such as, in the case of sulfate attack, alkaline and calcium sulfates or ferric hydroxide. Calcium sulfate reacts with tricalcium aluminate (C3A) to form ettringite and reacts also with sodium and potassium hydroxides [2]. It increases the volume of oxidation products relative to the original solids (increasing about 15%). Secondary expansion occurs due to ettringite formation in the paste [3]. Unlike other types of sulfate attack, chemical corrosion by sulfuric acid is not a pure sulfate attack, but a combined attack by the proton (an acid) and sulfate attack in which the acid components enhance dissolution and thereby play a significant role in the corrosion mechanisms [3]. The influence of sulfuric acid on concrete is well described in papers by Monteney et al. (2000) [4], Hewayde (2005) [5], O'Conell et al. (2010) [6], and others. Sulfuric acid appears in different aspects: from industrial wastewater and within sewage systems, e.g., water, wastewater, stormwater [7, 8] which can be contaminated with organic and inorganic compounds [9, 10], and also from groundwater. Sewage is a significant source of sulfuric acid; the effect of microorganisms on sulfates in the sewer system leads to the formation of sulfide, typically hydrogen sulfide, which is oxidized to sulfuric acid on the sewer walls. It further together with ferrous sulfate reacts with the alkaline constituents of the concrete's pore solution to form various sulfates [3].

The first laboratory and field assessments and evaluations revealing the effect of the microorganisms on corrosion of a sewer system started in Australia [11]. Islander et al. 1991 [12] defined the three-stage model of the corrosion processes of the concrete sewer system. In the beginning, calcium hydroxide in a concrete matrix dissolves in the concrete pore water (pH 12-13), which is too alkaline for colonisation of bacterial species. During some time period, acidic gases existing in the sewer system react with alkali species and lower the pH value below 9, which creates a suitable environment for colonisation of the surface by bacteria. When pH reaches a neutral and lower value, surface biological oxidation of sulfur species occurs and it lowers pH of the surface even further. From this point (pH about 4) it significantly increases the existence of sulfur-oxidising bacteria on concrete surfaces [13]. The most important degradative manifestations include extensive cracking, expansion, and loss of bond between the cement paste and aggregate. A field study is one way to investigate microbially induced corrosion (MIC) [14-18].

However, laboratory testing is very helpful for studying MIC as a complex system. It needs not only knowledge of microbiology but also requires special safety concerns, mainly because H_2S is highly toxic even at low concentrations. A laboratory experiment is demanding on control of such factors as temperature, nutrients, humidity, and other parameters. The first controlled chamber of the investigation of MIC was created by Sand et al. (1984) [19], where used concrete specimens were inoculated with sulfur-oxidising bacteria (*Acitihiobacilus thiooxidans*) by a spray. They concluded that the rate of corrosion in the chamber was 8 times higher than those observed in the field (tested after 270 days, up to 9% mass loss). In 1999 Ehrich et al. [20] modified the chamber used by Sand with the aim of studying smaller mortar specimens of different composition. A simulated experiment of MIC investigation in chambers was also performed by [21-24]. To understand sulfate corrosion and predict the durability of concrete, many various approaches have been applied. A combination of mineralogical, hydro/geochemical, and biological approaches for determining exact mechanisms responsible for a high corrosion rate were investigated in [18]. The multi-proxy approach was also presented in [25] in relation to studying a biogenic sulfuric acid attack on concrete. An indication of intense colonization of different families of sulfate-reducing and sulfur-oxidizing bacteria was observed based on their research. In the field of civil engineering, also the cluster analysis (CA) method, neural modelling, the Kohonen neural network, multidimensional comparative analysis for structure protection, structure durability assessment, or failure prediction are used [26], as well as the calculation of Pearson's correlation coefficient in [24]. Authors used a mineral liberation analyzer for the first time to analyze the degree. The investigation of correlation between corrosion products and microstructure of concrete exposed to simulated sewer conditions with controlled H_2S concentration was published of sewer concrete affected by sulfide induced corrosion.

Sulfate-resisting cement is applied in concrete structures in order to increase the resistivity against the sulfate attack and durability of concrete. New types of concrete such as sulfur concrete or concrete with sulfate-resisting cement with the aim of increasing corrosion resistance were designed. However, there are only a few papers investigating the performance of concrete with sulfate-resisting cement under sulfate attack [27-29]. The biological sulfate attack on this concrete is also studied rarely. Neville (2004) [2] deals with the problem when it comes to sulfate resistance of concrete if it is more important to use a special type of cement (type V. sulfate-resisting concrete) or just to deal with w/c ratio (low w/c ratio could ensure higher resistance to sulfates). The mechanism of sulfate attack depends on the cation in the sulfate: sodium, calcium, or magnesium.

This paper aims to investigate the selected parameters in chemical and biological-based degradation of concrete with sulfate-resisting cement. The dissolved amounts of the most important elements (calcium, silicon, iron, and aluminum) have been evaluated using correlation analysis.

Material and Methods

Preparing Concrete Samples

Concrete samples based on a sulfate-resisting cement were employed for the experiment. The mixture composition per one cubic meter of fresh concrete was as follows: 360 kg of CEM I 42.5 N-SR 0 with a very low tricalcium aluminate (C_3A) content; 1800 kg of aggregates of three fractions: 0/4 mm (825 kg), 4/8 mm (235 kg), and 8/16 mm (740 kg); 170 L of water; and 3.1 L of polycarboxylate-based superplasticizer. The mixture proportions have proposed the concrete to have a water-to-cement ratio $w/c = 0.47$. Measured amounts of cement, aggregates, and superplasticizer were mixed with water in a concrete mixer and then shaped using a jolting table for compacting cement samples. Prepared concrete prisms with dimensions of 100 × 100 × 400 mm were cured for 28 days in an aqueous environment in order to support the hydration process. After the curing period, the concrete samples were cut into smaller prisms of dimensions about $50 \times 50 \times 20$ mm^3 and brushed to remove all mechanical impurities. Then the samples were washed with water. The samples aimed at bacterial exposure were, in addition, treated by ethanol for 24 hours to remove potential biological impurities. After that, these samples were kept in an aseptic box over a period of 2 hours at laboratory temperature. All concrete samples were consequently dried to a constant weight at 105ºC in a laboratory oven. Subsequently, the concrete samples were exposed to sulfate attack by immersion in bacterial and chemical solutions. Both experiments proceeded at the identical room temperature of 23ºC and 60% humidity.

Chemical and Bacterial Solutions

The bacterial solution consisted of sulfur-oxidizing bacteria *A. thiooxidans* and cultivation medium in proportions of inoculum:medium = 1:4. Used bacteria were isolated from the mixed culture originating from mine water (Pech shaft in Smolník, eastern Slovakia) and treated by selective cultivation medium (medium S) according to Waksman [30] at 28-30ºC and pH of 4 – optimal to bacteria growth. The activity of bacterial culture was monitored at periodical intervals during the experiments while keeping the optimal conditions at a constant level. *A. thiooxidans* produced the biological sulfuric acid as a result of an oxidation of the elemental sulfur originating from cultivation medium S.

Chemical sulfate attack was simulated by a solution of sulfuric acid (H_2SO_4) with the same pH value as that kept in bacterial solutions (pH = 4). The pH was periodically measured and adjusted to 4 because, due to concrete's leaching, it tends permanently to be increased.

Both bacterial and chemical solutions' influence on concrete samples was investigated continually over a period of 3 months from December to February 2017.

Chemical and Mineralogical Analysis of Specimens

The prepared concrete samples and liquid phases were analyzed by x-ray fluorescence (XRF) spectrometer SPECTRO iQ II (Ametek, Germany) for 300 s using an air-cooled low-power Pd end window x-ray tube (25 kV and 50 kV at 1 and 0.5 mA, respectively) combined with an HOPG crystal for monochromatization and polarization of the primary tube spectrum.

The surface compounds were observed by electron scanning microscopy (SEM) using a Jeol JSM-35CF (Japan). The elemental EDX analysis was carried out on a LINK AN 10 000 micro-analytical system operating in secondary mode at a potential 25 kV and at extension 90-5500.

Mineralogical composition of samples and surface precipitants was analyzed using a D2 PHASER x-ray powder diffraction meter (Bruker, Germany) using Cu K alfa radiation generated at 10 mA and 30 kV. Scan conditions were identical for all samples with the step size of 0.02º over the range 2 theta from 10 to 70º.

Correlation Parameters

A degradation of the concretes was manifested in our study by dissolving the main concrete components. Leached quantities of calcium, silicon, iron, and aluminium ions were analyzed in regular intervals, once a week, by a SPECTRO iQ II x-ray fluorescence (XRF) spectrometer (Ametek, Germany) under an inert atmosphere. The intensities for all elements were corrected automatically for line interference and absorption effects due to all the other elements using the fundamental parameter method.

The leached-out amounts of Ca, Si, Fe, and Al represented correlation parameters and have been used in the correlation analysis of the concrete's deterioration to find a potential dependence between bacterial and chemical degradation. The statistical method was also used for better interpretation of the leaching trends.

Correlation Analysis

Correlation coefficients are used in statistics to measure how strong a relationship is between two variables. There are several types of the correlation coefficient, e.g., Pearson's correlation is a correlation coefficient commonly used in linear regression. One of the most commonly used formulas in stats is Pearson's correlation coefficient formula (1) [31]:

Table 1. Chemical composition of concrete with sulfate-resisting cement.

	CaO	SiO_2	Fe_2O_3	Al_2O_3	MgO	P_2O_5	SO_3	K_2O	TiO_2	MnO	Other
[%, by mass]	14.49	62.81	5.51	6.94	1.61	0.06	0.16	1.1	0.33	0.08	6.91

$$R_{xy} = \frac{n\sum_{i=1}^{n} x_i y_i - \left(\sum_{i=1}^{n} x_i\right)\left(\sum_{i=1}^{n} y_i\right)}{\sqrt{\left[n\sum_{i=1}^{n} x_i^2 - \left(\sum_{i=1}^{n} x_i\right)^2\right]\left[n\sum_{i=1}^{n} y_i^2 - \left(\sum_{i=1}^{n} y_i\right)^2\right]}} \tag{1}$$

The formulas (1) return a value between -1 and 1, where 1 indicates a strong positive relationship, -1 indicates a strong negative relationship, and a result of zero indicates no relationship at all. Other values point to medium closeness (if $0.3 \leq |R_{xy}| < 0.5$); significant if $0.5 \leq |R_{xy}| < 0.7$; high (if $0.7 \leq |R_{xy}| < 0.9$) or very high (if $0.9 \leq |R_{xy}|$) closeness.

Correlation coefficients were used for assessing investigation relation of the leached-out amount of basic chemical elements from the concrete matrix.

Results and Discussion

The chemical analysis of studied concrete samples, measured by XRF analysis, is given in Table 1. The chemical composition of the concrete based on the sulfate-resistant cement differed slightly from the concrete based on the ordinary Portland cement (OPC) only [32].

The higher content of iron and silicon oxides has been likely linked to the variability in silicon and iron amounts in OPC and sulfate-resisting cement, respectively. Even though C_3A content in mentioned binders differs significantly, almost the same percentage of aluminum oxide (about 7 wt. %) was observed in studied concrete samples as well as in OPC-based concretes.

The deterioration process of concrete was manifested by dissolution of the main cement matrix components as well as by the formation new sulfate-based compounds.

Dissolved Quantities of Ca, Si, Fe, and Al

Measured amounts of dissolved calcium silicon, iron, and aluminum ions are presented in Figs 1 and 2. As for bacterial exposure, the cumulative leached amounts of calcium ions ranged 71.9 to 527.4 mg/L, while of silicon from 206.8 to 651.5 mg/L. Dissolved iron was in a close interval, ranging from minimum 7.1 to maximum 18.2 mg/L, whereas Al ranged from 0 to 131.6 mg/L. The cumulative leached amounts of calcium ions,

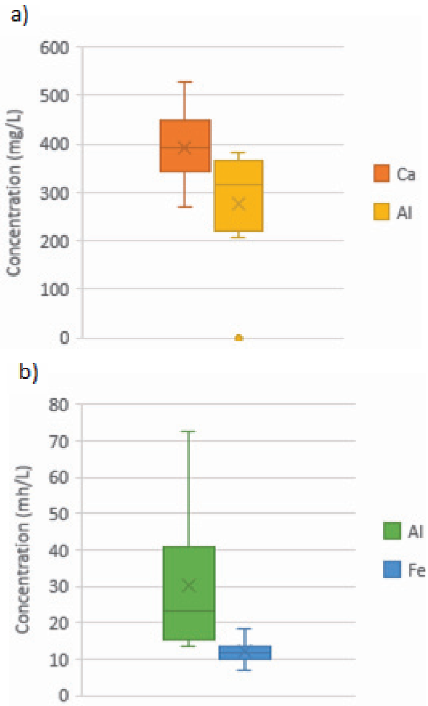

Fig. 1. Leached-out quantities of a) Ca and Si, and b) Al and Fe during bacterial exposure.

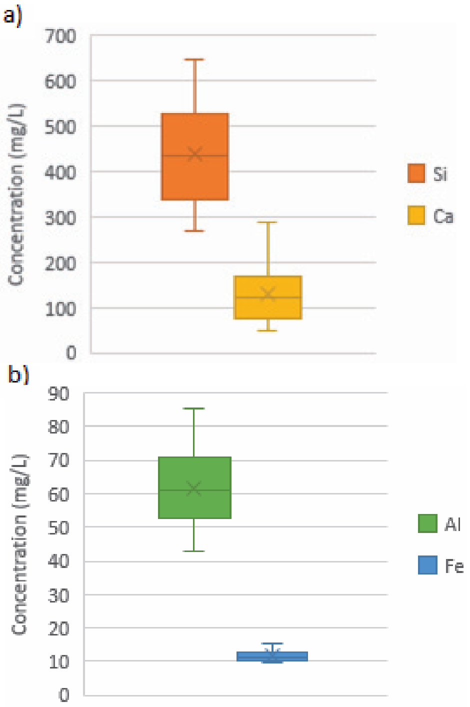

Fig. 2. Leached-out quantities of a) Ca and Si, and b) Al and Fe during chemical exposure.

due to the chemical sulfuric attack, ranged from 0 to 1546.2 mg/L, while of silicon from 0 to 963.4 mg/L. Dissolved iron was measured at a level from 9.5 to 15.3 mg/L with no significant peak, whereas Al ranged from 43.1 to 122.5 mg/L.

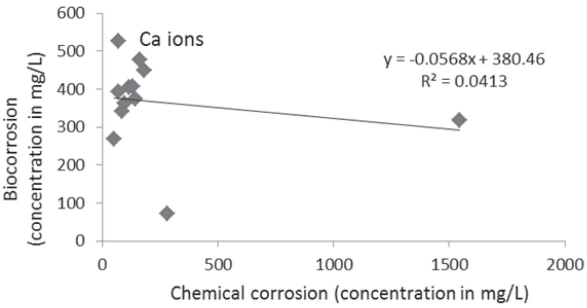

Fig. 3. Dependency of leached-out quantities of Ca ions due to chemical corrosion and biocorrosion, respectively.

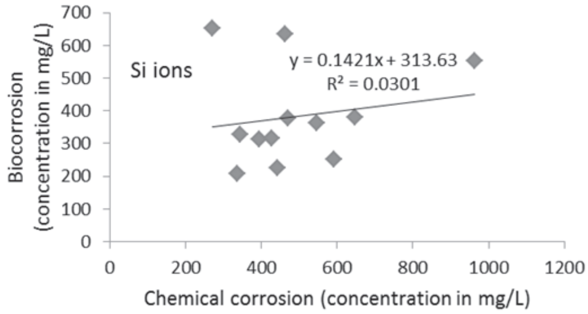

Fig. 4. Dependency of leached-out quantities of Si ions due to chemical corrosion and biocorrosion.

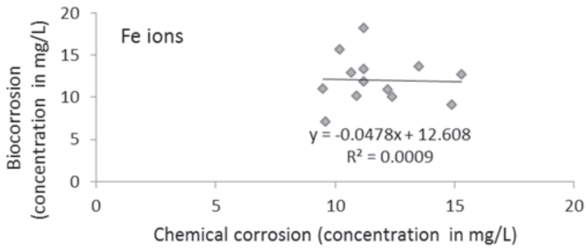

Fig. 5. Dependency of leached-out quantities of Fe ions due to chemical corrosion and biocorrosion.

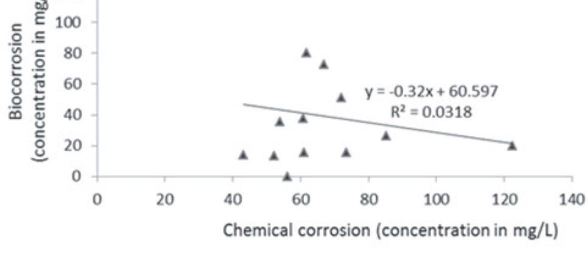

Fig. 6. Dependency of leached-out quantities of Al ions due to chemical corrosion and biocorrosion.

As seen in Figs 1 and 2, the most intensive leaching was observed for silicon followed by calcium ions. Massive leaching of calcium ions is in good agreement with the degradation process of concrete matrix. Hardened concrete includes hydration products such as portlandite ($Ca(OH)_2$), calcium silicate hydrates (C-S-H), ettringite (AFt), or monosulfate (AFm). When exposed to a water environment, portlandite dissolves first by leaching calcium ions. The dissolving of the iron compounds has been negligible. The leached quantities of cement components resulting from bacterial exposure have been more significant than those resulting from chemical exposure.

Correlations between Chemical and Biological Corrosion

As mentioned, the measured dissolved quantities of the particular ions in bacterial and chemical environments were compared using correlation analysis. Figs 3-6 present the relationships of leaching the individual ions each other in bacterial and non-bacterial media, respectively.

Surprisingly, no significant correlation was found between the leached amounts resulting from the chemical and biological sulfuric acid exposures. This is contrary to our previous results evaluating the corrosion of concrete with ordinary Portland cement [33, 34]. The calculated correlation coefficients are given in Table 2. The findings point to the fact that there is a great difference in leaching behaviour of the main concrete components due to chemical and biological sulfuric acid attack when examining the concrete with sulfate-resisting cement.

Sulfate Content in Solutions

Besides the dissolved amount of the main components of the concrete matrix, the sulfate content was investigated in both solutions during the experiment. The courses of the sulfate concentrations in chemical and biological solutions are given in Fig. 7. The sulfate amounts originating from the cultivation medium S have been subtracted from the measured concentrations in biological solution. The input sulfate concentration in chemical solution corresponds to pH = 4.

Table 2. Leaching of elements due to chemical corrosion (CC) and biocorrosion (BC).

BC/CC	Correlation coefficient (R_{xy})
Ca	-0.2
Si	0.17
Fe	-0.17
Al	-0.03

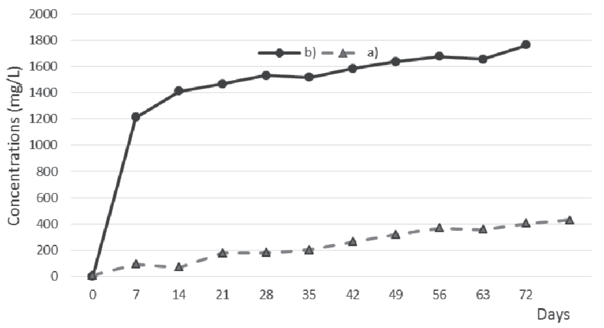

Fig. 7. Concentrations of sulfates in a) chemical and b) biological solutions.

As seen in Fig. 7, the concentrations of sulfates in biological solution were several times higher than in the chemical ones during the experiment and reached values of up to 1800 mg/L. As seen from Fig. 7b), the oxidation of elemental sulfur and production of biological sulfuric acid started after several days of bacteria inoculation that was manifested by the significant increase in sulfate anion concentrations to 1200 mg/L. Subsequently, the formation of biological sulfuric acid consistently continued until the end of the experiment. The presence of bacteria was periodically checked by microscopic observation of staining microscopic specimens. The bacteria-produced sulfuric acid was likely consumed in the deterioration process and by the formation new sulfate compounds.

Remarkably, the sulfate concentrations in chemical solution (Fig. 7a) increased significantly over the whole experimental period as well. The increase could be linked to the fact that once a week 4 ml of solution was removed to analyze the chemical composition of liquid and this procedure could cause the pre-concentration of the solution. Alike, the pH was adjusted periodically to 4 by adding the 0.1 M sulfuric acid. On the other hand, the stronger increase in sulfate amount could indicate that the sulfuric acid was not as involved in the deterioration process as in the biological corrosion.

Mineralogical Composition of Concretes

The diffractograms of the concrete samples after chemical and biological sulfate exposures, measured at Cu Kα radiation (λ = 1.5406 Å), are presented in Figure 7. Portlandite (18.0°, 28.7°, 36.4°, 47°), quartz (26.6°, 20.8°, 42.5°, 50.1°), slurry phases (29-30°, 31°), and sulfate compounds, e.g., plaster (12°, 20.7°, 29.1°, and 31.1°), magnesium sulphite monohydrate (13.8°, 27.9°), and aluminum hydrosulfate (20.8°, 18.8°, 25°, 2°) have been identified according to the positions of the peaks at 2 theta angles reported in the literature [35]. No significant differences have been observed in diffractograms when comparing chemical and biological corrosion, as seen in Fig. 8.

However, lower peaks corresponding to portlandite were recorded for the sample after biological corrosion. At 36.5° 2 theta, the area of the peak after biological exposure corresponded to 11.52 compared to 19.17 for the portlandite peak of the sample after chemical corrosion. This indicated a lower content of portlandite in the composite structure. This finding revealed that in the sample after the bacterial action was a more significant degradation of the composite matrix compared to the chemical action similarly as in [36].

Surface Products

After the biological and chemical sulfate corrosion simulation experiment, various visual surface changes of concrete composites were observed compared to the pre-experiment specimen surface. The formation of the new sulfate-based product was visible on the surface in the form of a white coating or separate crystals (Fig. 9). Surface precipitates of biological and chemically affected samples showed a different morphology.

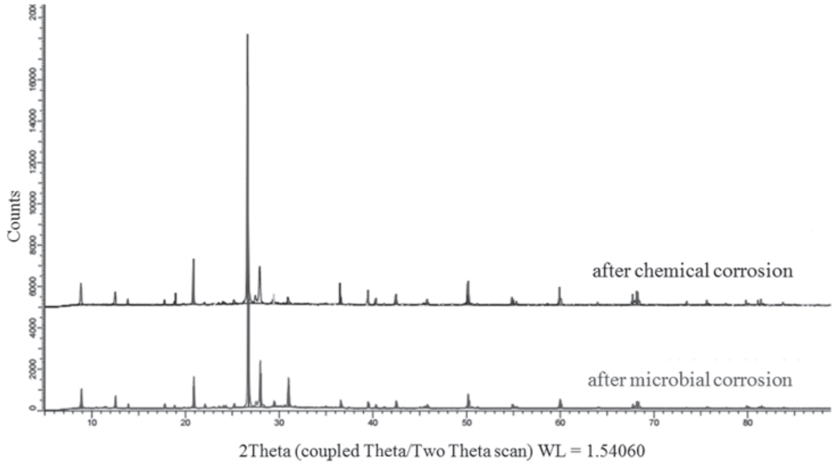

Fig. 8. XRD diffractogram of concrete after chemical and biological actions.

The surface compounds formation was found to be more significant due to biological sulfate action compared to the chemical one. This is in accordance with the fact that sulfate concentrations in biological solution were almost 5 times higher than in the chemical solution. EDX analysis of both samples' precipitates revealed the presence of Ca, Si, O, and S, indicating the presence of sulfate compounds such as gypsum, anhydrite, or bassanite, in accordance with the literature knowledge [36-37].

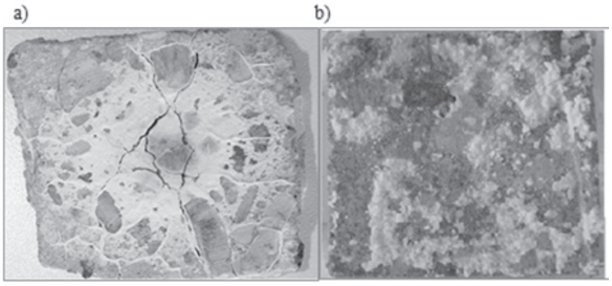

Fig. 9. New compounds on the surface of the a) biologically and b) chemically affected samples.

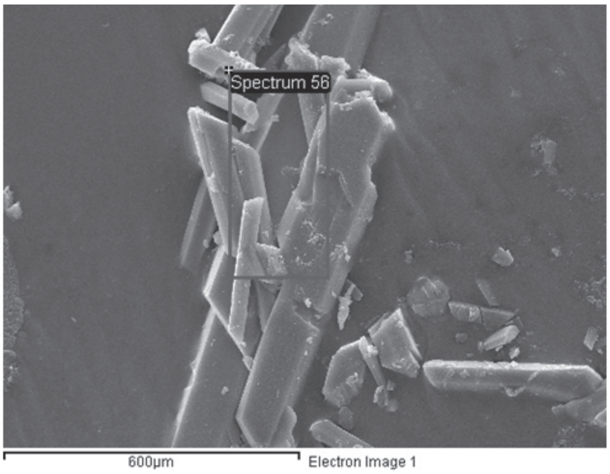

Fig. 10. SEM micrograph of the surface compound of the chemically affected sample.

The presence of phosphorus, which is a component of the nutrient medium in the form of K_2HPO_4, was also observed in the biologically affected sample. The semi-quantitative analysis of precipitates in the chemically affected concrete sample recorded 30.4 wt.% Ca, which was 2.8 wt.% less than that of the biologically affected sample.

XRD analysis of the surface products confirmed the presence of gypsum and bassanite as seen in Fig. 11.

Conclusion

This paper describes our investigation of acidic corrosion caused by artificial sulphuric acid and biogenic sulphuric acid. The chemically based and biologically based deteriorations have been evaluated through the leached quantities of such basic chemical elements as calcium, silicon, iron and aluminum, and sulfate concentrations' variations in solutions, as well as through surface and mineralogical changes.

- Correlation analysis points to the great difference in leaching behaviour of the main concrete components due to chemical and biological sulfuric acid attacks.
- New compound formation on concrete surfaces resulting from biological sulfate action was found to be more significant than from the chemical one.
- Biological and chemical surface precipitates of affected samples showed a different morphology.

Concrete with sulfate-resistant cement seems to be more resistant to artificial sulfuric acid than against biologically produced sulfuric acid. This is likely connected with a complexity of processes due to bacteria and their microenvironment effects.

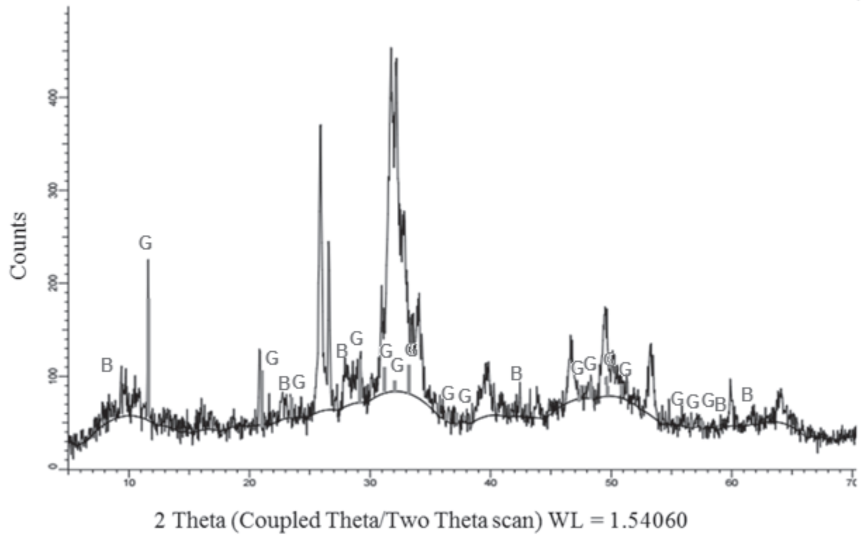

Fig. 11. XRD diffractogram of the surface product (G = gypsum, B = bassanite).

Acknowledgements

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Conflict of Interest

The authors declare no conflict of interest.

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