

# DETECTION OF THE SYNERGETIC INFLUENCE OF CHEMICAL AND MICROBIOLOGICAL FACTORS ON THE PROPERTIES OF CONCRETE CONSTRUCTIONS AT CHEMICAL PLANTS DURING THE LONG-TERM SERVICE

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## Abstract

Long-term operation of reinforced concrete structures in the conditions of chemical enterprises has a powerful negative impact on the physical and chemical properties of concrete, which leads to its destruction. The aim of this research is to determine the effect of biological and chemical corrosion on concrete structures in the workshop for the production of titanium dioxide by the sulphate method and the storage of finished products. In particular, chemical production for the synthesis of titanium dioxide by the sulfate method causes the rapid course of chemical (acid and sulfate) and microbiological (thionic bacteria and microscopic fungi) corrosion processes. These corrosion processes reinforce each other according to a synergistic principle. As a result, temperature-programmed desorption mass spectrometry (TPD MS) and scanning electron microscopy have experimentally proven the presence and spatial localization of colonies of thionic bacteria and microscopic fungi in concrete structures. Correlations between the intensity of biochemical corrosion and the depth of damage to the microstructures of concrete structures have been established. Moreover, a change in the chemical composition of concrete in the workshop for the production of titanium dioxide (increased SO<sub>2</sub> content and reduced CO<sub>2</sub>) and the formation of gypsum crystals (CaSO<sub>4</sub>·2H<sub>2</sub>O) as a result of the dissimilation of microorganisms was established. Also, in the storage room for finished products, calcium citrate crystals and a violation of the formation of calcium carbonate are formed in the surface layers of concrete. In addition, the results of the study can be used to develop antimicrobial and anticorrosive protective agents to stop the biochemical corrosion of concrete in a chemical plant.

**Keywords:** durability of concrete, sulfate acid, thermal properties, biochemical corrosion, microscopic structure of concrete.

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## 1. Introduction

A large number of industrial and civil structures are made of concrete. But the problem of durability and strength of concrete is still an unsolved problem for scientists and is constantly in the process of improvement. In [1], it was determined that the addition of bagasse ash in the form of fine aggregate to the concrete mortar reduced the degree of permeability. The addition of cork to the mortar increased the thermal resistance of the concrete. The use of nanomaterials [2] as an additive to concrete increases the resistance of concrete to acids and moisture. However, researchers [3] noted that more research is needed on all fine aggregates to be sure of their use in concrete.

An important task for researchers is to predict the influence of the environment on concrete structures in order to determine their safe service life. It is known that the deterioration of the viscosity of concrete under the influence of acid rain. The researchers found that the depth of destruction increases with an increase in the duration of corrosion [4, 5]. Scanning electron microscopy revealed a loose concrete microstructure with a large number of cross microcracks. In addition, scientists have found that under the action of acid corrosion, the physical and chemical properties of concrete deteriorate. Unexplained were the issues of the spread of concrete corrosion over time and the change in the chemical composition of concrete. Also, the temperature differences of freezing and thawing of the concrete mixture were not taken into account.

Modern technologies allow the use of reinforced concrete structures in the extreme conditions of livestock buildings [6]. The main reasons that influenced the development of biological and chemical corrosion in the premises were excessive moisture, animal stool and the use of acid or alkaline disinfectants [7]. Microscopic studies revealed microorganisms (micromycetes: *Penicillium* and *Fusarium*, bacteria: *Escherichia coli* and *Pseudomonas aeruginosa*), which were the cause of biological corrosion of concrete in livestock buildings [8].

The researchers combined in their experiments the effect on concrete of chemical corrosion of sulfate solution and extreme temperature difference in freeze and thaw cycles [9]. This made it possible to determine the correlation dependence of the elasticity modulus of concrete on the degree of corrosion. In addition, an increase in the products of chemical corrosion of concrete was found depending on the duration of the experiment [10]. However, the researchers point out that it is necessary to be careful when conducting research on concretes that have a short production time. During freeze and thaw cycles, young concretes can complete hydration, which can lead to erroneous interpretation of the data obtained [11].

Under the influence of the environment, building structures made of concrete are subject to destruction over time. It is not often that concrete is affected by a single failure factor, but durability prediction is based on modeling one type of failure mechanism. Due to the possible synergistic effect of two or more corrosion mechanisms that increase over time, the predicted rate of failure may differ. It was confirmed in [12] that the same type of experiments carried out under laboratory conditions radically differ from field studies in real conditions under the influence of different types of corrosion causes. It also proves that research should be as close as possible to the actual operating conditions of concrete structures and a holistic approach to concrete durability indicators.

A compatible cause of concrete failure is often chemical and biological corrosion. Building structures in marine areas are affected by high concentrations of chlorides and sulfates. Further, the concrete is colonized by specific microorganisms (vibrios, arthrobacteria, pseudoalteromones, pseudomonads), accelerating the destruction process [13]. Biological corrosion of concrete is a problem in irrigation and hydroelectric canals. The main reason is the growth and reproduction of algae and cyanobacteria, which are involved in the process of destruction of concrete structures and reduce their service life [14]. In wastewater treatment plants, concrete structures are exposed to natural sulphate acid. Natural or artificial sulphate acid ( $H_2SO_4$ ), or other sulphate compounds contained in wastewater, groundwater, chemical waste adversely affect the components of concrete and reduce the life of concrete structures. Currently, no optimal standard for testing the acid resistance of cement-based materials has been defined [15].

Many researchers do not take into account the influence of interrelated chemical and microbiological corrosion of concrete, which can reinforce each other. Under laboratory conditions, the experiment proved the effect of microbial and chemical corrosion on the mechanical and physical

properties of cement stones. Concrete samples were immersed in a medium with microorganisms and  $H_2S$ . The studies were carried out using cultures of bacteria *A. Thiooxidans* and *H. Neapolitanus*. The destruction of the structure of concrete samples was more observed in the environment with *A. thiooxidans* (27 %). The results obtained in [16] prove the enhanced synergistic effect of biological and chemical corrosion on concrete in specific natural or created conditions.

The progress in reducing the strength of concrete during sulfate corrosion is an important indicator for predicting the remaining service life of engineering structures to prevent disasters. Modern studies report that sulfate ions enter into a chemical reaction with concrete components [17]. Penetrating by diffusion, capillary adsorption or convection into the cement stone, crystals of ettringite and gypsum with expansive properties are formed. The formed crystals fill the interior of the concrete until their number exceeds the possible content of the concrete. In the future, the stress increases and the concrete cracks. This process is accompanied by the loss of tobermorite, the original product of concrete hydration. Finally, concrete enters the stage of degradation and strength reduction [18].

Usually, scientists use the method of studying the stability of concrete samples immersed in a solution of sulfate acid. Then, the change in the physical parameters of concrete (strength and weight loss) is determined. There is also a method for testing concrete for the depth of penetration of sulfate ions, which can be used as an indicator of the degree of chemical corrosion. However, actual concrete damage progresses over time and all previous tests have calculated talc for a short time [19].

Temperature-programmed desorption-mass spectrometry (TPD-MS) was used to determine the chemical composition of released gases from concrete samples due to both classical desorption from the surface and thermal degradation of concrete samples during heating in vacuum at a programmed rate. TPD-MS provides information on: quantitative and qualitative characteristics of gaseous substances that are adsorbed on the surface of samples of heterophase samples of complex microstructure; thermochemical properties of the samples (destruction rate upon heating, the composition of gaseous substances formed during the indicated destruction, and features of the macro- and microstructure of solid samples [20].

The TPD MS method was used by researchers [21] to determine pyrogenic oxides from natural calcium carbonates (corals, chalk, anadara shells) and artificial composite materials containing carbonates – concrete. Theoretical models have been proposed to generalize the processing of the obtained data, and formulas and constants for reactions have been developed.

The aim of this research is to determine the effect of biological and chemical corrosion on concrete structures in the workshop for the production of titanium dioxide by the sulphate method and the storage of finished products.

Currently, there are many methods for studying the destruction of concrete in the laboratory. However, none of them is able to predict and assess the depth of damage to concrete in operated premises. In addition, the chemical process that occurs during the interconnected chemical and biological corrosion of concrete remains unexplored. The TPD MS method will allow to study the change in the chemical composition of test samples of concrete subjected to corrosion processes at different depths from the surface compared to control samples. Electron microscopy makes it possible to study the structure of concrete at the microscopic level, to determine the formation of crystals of various origins in it and the localization of microorganisms.

## 2. Materials and methods

### 2. 1. Methodology for the study of concrete samples for determining thermal resistance using TPD MS

To study the heat resistance, concrete samples were obtained in the titanium dioxide production workshop and placed for storage of finished products in different areas: on the surface and at a depth of 0.5 cm; 1.0 cm, 1–2 cm. Control samples of concrete were obtained in the office center of this chemical enterprise, which also coincides with the pilot production facilities in terms of operation.

Programmed temperature desorption mass spectrometry (TPD MS) was used to determine the chemical composition of concrete samples and the rate of failure upon heating. The TPD MS

setup consists of a high-temperature furnace and an MX-7304 gas mass spectrometer (JSC SELMI, Sumy, Ukraine). Concrete samples weighing 5–10 mg were subjected to vacuum to 10–3 Pa, heated from 40 to 900 °C at a rate of 15 °C/min. Simultaneously, every minute, the mass spectrum of the released gas mixture was recorded on a mass spectrometer. Peaks of ions with the corresponding molecular masses ( $m/z$ ) were identified in the mass spectra: 18, water; 44 – carbon dioxide CO<sub>2</sub>, 64 – sulfur dioxide SO<sub>2</sub> [22].

## 2. 2. Method for conducting microbiological studies

For microbiological studies, washings of building structures were carried out in experimental rooms from the surface and at a depth of 0.5–2 cm.

For microbiological studies, washings of building structures were carried out in experimental rooms from the surface and at a depth of 0.5–2 cm.

The cultivation of *Acidithiobacillus thiooxidans* was carried out on a liquid nutrient medium WAKSMAN with the addition of the following components to the meat-peptone broth: S – 10.0; (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> – 0.2; KH<sub>2</sub>PO<sub>4</sub> – 3.0; MgSO<sub>4</sub>·7H<sub>2</sub>O – 0.5; CaCl<sub>2</sub>·6H<sub>2</sub>O – 0.025; FeSO<sub>4</sub>·7H<sub>2</sub>O – traces; pH 4.0 [23].

To grow microscopic fungi, Czapek-Dox medium was used with exposure in a thermostat for 5 days at a temperature of 30 °C [24].

## 2. 3. Scanning electron microscopy methodology

The microstructure of the concrete samples was studied with a SEM 106 and scanning electron microscope (JSC SELMI, Sumy, Ukraine). Concrete samples were placed on metal substrates and coated with silver. Preliminary fixation of films of microorganisms on the concrete surface was carried out with glutaraldehyde at a concentration of 2.5 % in a phosphate buffer solution with pH 7.2 [25]. The SEM photo was taken in the secondary electron mode at magnifications from 200 to 5000 times.

## 3. Results and discussion

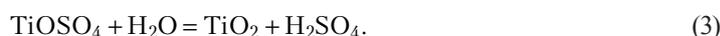
### 3. 1. The research results of the causes of the formation of biochemical corrosion of concrete in the workshop for the production of titanium dioxide by the sulfate method

The studies were carried out at a chemical plant located in the city of Sumy, Ukraine. The workshop for the production of titanium dioxide by the sulphate method was put into operation in 1975. Walls, columns and ceilings are made of class C 20 precast concrete. The roof system consists of metal trusses.

The technology for the production of titanium dioxide by the sulfate method consists in processing the raw material (ilmenite concentrate) at high temperature with sulfate acid in a concentration of 91.5–95 %, depending on the stage of production and the purity of the raw material. Titanium and other metals contained in the ore pass into the form of water-soluble salts (according to reactions (1), (2)):



After going through the following stages, including chemical reduction, purification, precipitation, hydrolysis (reaction (3)) and calcination, TiO<sub>2</sub> crystalline fractions in the form of rutile are obtained:

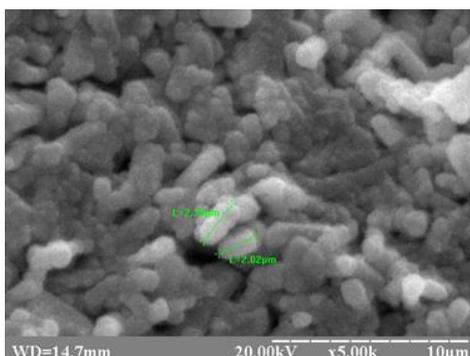


The production of titanium dioxide is accompanied by the release of vapors of sulphate acid, sulfur dioxide SO<sub>2</sub>, hydrogen sulfide H<sub>2</sub>S and elemental sulfur, which settles on the surface of building concrete structures [26]. As a result, conditions are created for concrete corrosion (**Fig. 1**).



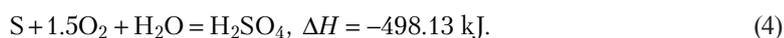
**Fig. 1.** Consequences of corrosion processes in the workshop for the production of titanium dioxide ( $\text{TiO}_2$ ) by the sulphate method

In the workshop for the production of reinforced concrete structures, colonies of thionic bacteria were found (**Fig. 2**), which use sulfur in the course of their metabolism.



**Fig. 2.** SEM images of *Acidithiobacillus thiooxidans* isolated from the surface of concrete structures in a titanium dioxide production facility

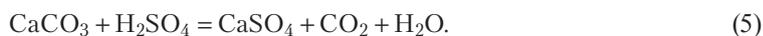
These microorganisms belong to the sulphur oxidizing bacteria (SOB) group. *A. thiooxidans* forms a biofilm on the surface of concrete structures and is responsible for the microbiological corrosion of concrete. *A. thiooxidans* are purely acidophilic microorganisms. Immediately after hardening, the concrete has a slightly alkaline pH of 9.0. Such an environment is suitable for the growth of neutrophilic bacteria, which in the process of metabolism reduce the pH to from 6.0 to 4.0. Further, acidophilic microorganisms *A. thiooxidans* are included in the process, receiving energy by oxidizing elemental sulfur to sulfate acid according to reaction (4):



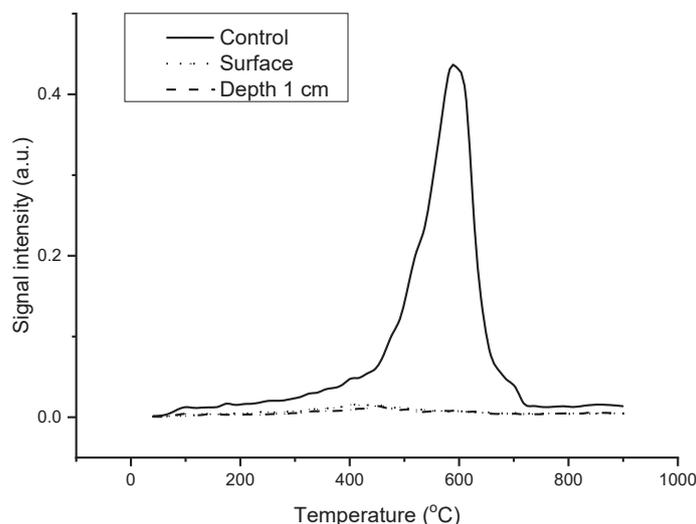
The study of the destructive effect of the sulfate environment and the metabolism of *A. thiooxidans* bacteria on concrete was determined by the TPD MS method (**Fig. 3**).

The results obtained indicate a high intensity of  $\text{CO}_2$  release from the control sample of concrete: 0.43 when heated to a temperature of 600 °C. The samples obtained from the surface and from a depth of 1 cm contain almost no carbonates. The data obtained indicate a change in the chemical composition of concrete under the action of biochemical corrosion. As a result, calcium

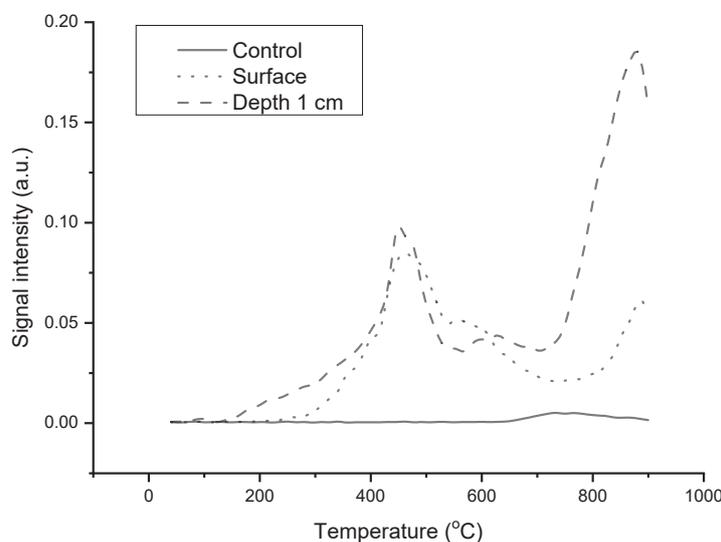
carbonate is destroyed under the influence of sulfate acid, and the amount of sulfate, on the contrary, increases according to reaction (5):



In further studies, the intensity of sulfur dioxide release in test concrete samples was determined (Fig. 4).



**Fig. 3.** Thermograms of CO<sub>2</sub> release ( $m/z = 44$ ) from the sample obtained in the titanium dioxide production shop



**Fig. 4.** Thermograms of SO<sub>2</sub> release ( $m/z = 64$ ) from the sample obtained in the titanium dioxide production shop

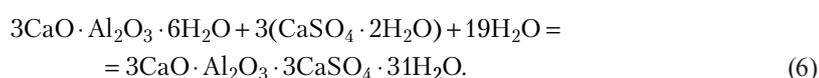
Heating of the control sample of concrete showed the absence of SO<sub>2</sub> in it. A concrete sample taken from a depth of 1 cm when heated gives two peaks of sulfur dioxide emission. A less intense peak (0.10) of SO<sub>2</sub> release at t 500 °C and a maximum (0.18) at t 900 °C. The concrete sample in the obtained surface also gives an intense peak of sulfur dioxide emission at a temperature of 500 °C with a signal intensity of 0.7. A decrease in the signal peak for SO<sub>2</sub> release is observed at t 900 °C.

As a result of the study, it can be argued that sulfate acid is the main chemical factor in corrosion processes. The formation of which occurs in two ways. The first is the own chemical

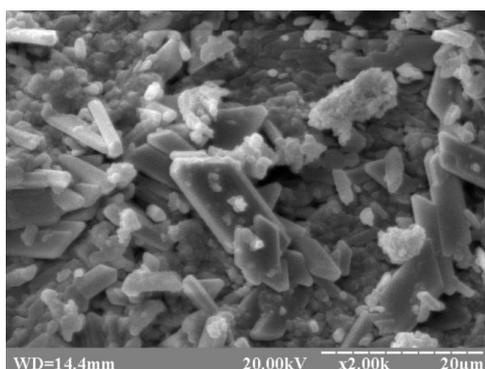
production of titanium dioxide, accompanied by the release of sulfate acid vapor and sulfur oxides, which, when interacting with water condensate on the concrete surface, also turn into sulfite and sulfate acids. The second way is microbiological, that is, the release of sulfate acid as a result of the vital activity of thionic bacteria [27].

According to the chemistry of corrosion processes, two directions can be distinguished – acid and sulfate corrosion. Actually, acid corrosion consists in the interaction of insoluble alkaline components of concrete (lime and calcium carbonate) with acids with the formation of more soluble sulfates, which are washed out from the concrete surface with water or form brittle precipitates of crystal hydrates (**Fig. 5**).

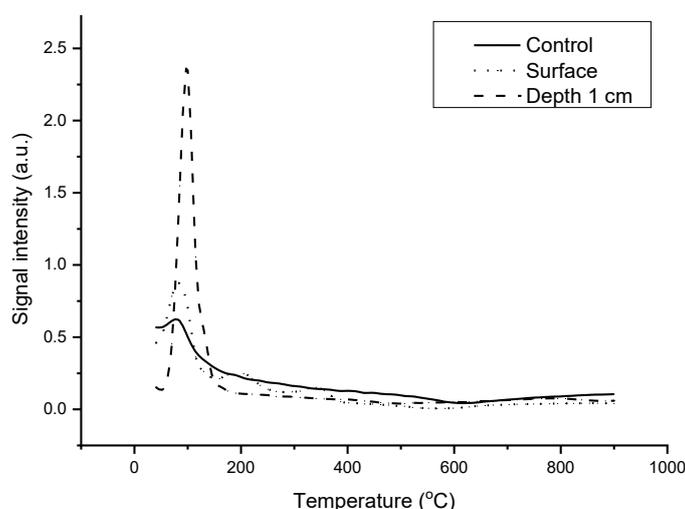
In the presence of gypsum crystalline hydrates ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ), sulfate corrosion of concrete starts. In this case, as a result of the reaction (6) of gypsum with tricalcium aluminate ( $3\text{CaO} \cdot \text{Al}_2\text{O}_3$ ), calcium hydrosulfoaluminates ( $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 31\text{H}_2\text{O}$  – ettringite) are formed in the pores and microcracks of concrete. The volume of ettringite crystals increases up to 30 times compared to the initial reagents, this causes internal stress and destruction of the cement stone [28]:



As a result of the biochemical destruction of concrete, cracks are formed through which microorganisms migrate and moisture accumulates. **Fig. 6** shows the results of water removal from concrete samples during heating.



**Fig. 5.** SEM images of gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ) crystals obtained from the surface of concrete structures in a titanium dioxide production plant



**Fig. 6.** Thermograms of  $\text{H}_2\text{O}$  release ( $m/z = 18$ ) from the sample obtained in the shop for the production of titanium dioxide

From the control sample of concrete and the sample from the surface, when heated to 100 °C, water quickly evaporated with a low intensity of 0.5–0.6. However, from a sample obtained from a depth of 1 cm, moisture was released with an intensity of 2.25. The results obtained indicate the accumulation of a significant amount of H<sub>2</sub>O in the depth of the concrete that got here due to cracks in the concrete.

### 3. 2. Research results of the mechanism of development of biochemical corrosion in the storage room for finished products

The storage room for finished products was built in 1975. The floors, foundation and plinth are made of reinforced concrete, the frame is metal (Fig. 7).

Due to constant temperature changes (freezing-thawing), a large amount of moisture and long-term operation of the building in concrete structures, the surface layer is destroyed. The degradation of the surface layer promotes the growth of microorganisms – autotrophs, which begin the assimilation of concrete. Micromycetes are able to use concrete as a nutrient medium for nutrition. Waste products of micromycetes have a bad effect on building structures and residents of houses. It has been established by researchers that among the rich variety of micromycetes, fungi of the *Penicillium* and *Aspergillus* families are most common [29].

In the storage room for finished products, the fungus *Aspergillus fumigatus* was isolated on the surface of concrete structures (Fig. 8).



Fig. 7. The results of long-term destruction of concrete structures in the storage room for finished products under the influence of the environment

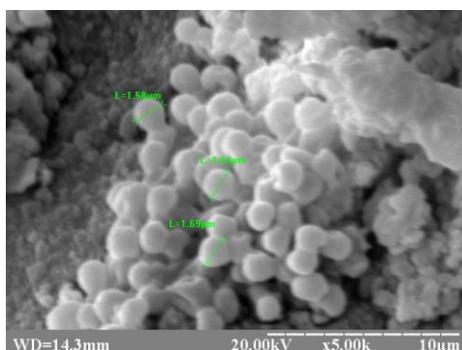


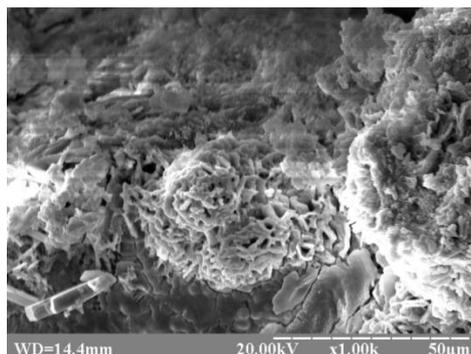
Fig. 8. SEM images of *Aspergillus fumigatus* obtained from the concrete surface in the finished product storage room

Among the metabolites of micromycetes, the most corrosive effect on concrete is exerted by organic acids that can react with lime and limestone in the composition of concrete, due to the

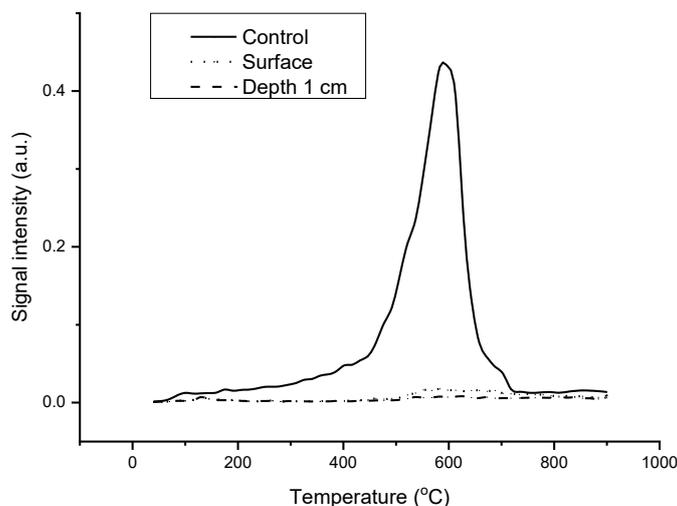
alkaline nature of the latter. As a result, carbonates are destroyed, and calcium passes into the form of salts of organic acids. Calcium salts of acetic, malonic and lactic acids are highly soluble and washed out of concrete. Citric acid forms poorly soluble calcium citrates (reaction (2)), the crystals of which are visible in the XPS photo (**Fig. 9**).



Thus, organic acids prevent the process of lime carbonization, as evidenced by the results of a mass spectrometric analysis of  $\text{CO}_2$  release from concrete samples affected by *Aspergillus fumigatus* compared to the control (**Fig. 10**).



**Fig. 9.** SEM images of calcium citrate crystals on the surface of concrete samples in the finished product storage room



**Fig. 10.** Thermograms of  $\text{CO}_2$  release ( $m/z = 44$ ) from the sample obtained in the finished product storage room

In the sample obtained from the control room, when heated to  $t = 600$  °C,  $\text{CO}_2$  is released with an intensity of 0.4 and gives a clear peak. The results indicate the content of limestone in the sample. In test specimens, samples of concrete from the surface and a depth of 1 cm did not emit  $\text{CO}_2$ . The results obtained confirm that as a result of the metabolism of the fungus *Aspergillus fumigatus* [30, 31], found on the surface of concrete structures in the storage room for finished products, the formation of limestone (calcium carbonate) is disrupted.

In addition, an experiment was carried out mass-spectrometric analysis of water evaporation during heating (**Fig. 11**).

The loss of water in the control sample occurs when heated to 100 °C with an intensity of 0.6, then evaporation proceeds gradually relative to the degree of concrete destruction. On the

thermogram of moisture evaporation in the test samples, two peaks are observed when heated to 100 °C and 200 °C. At the same time, the intensity of H<sub>2</sub>O evaporation either increases to 1.0 or decreases to 0.3 intermittently. The results emphasize the high degree of destruction of the surface layer of concrete by biochemical corrosion [32].

Biochemical processes in concrete, which occurred under the influence of environmental factors and biological corrosion, influenced the release of chemical elements from concrete when heated. The thermogram (Fig. 12) shows the results of sulfur dioxide release from concrete samples.

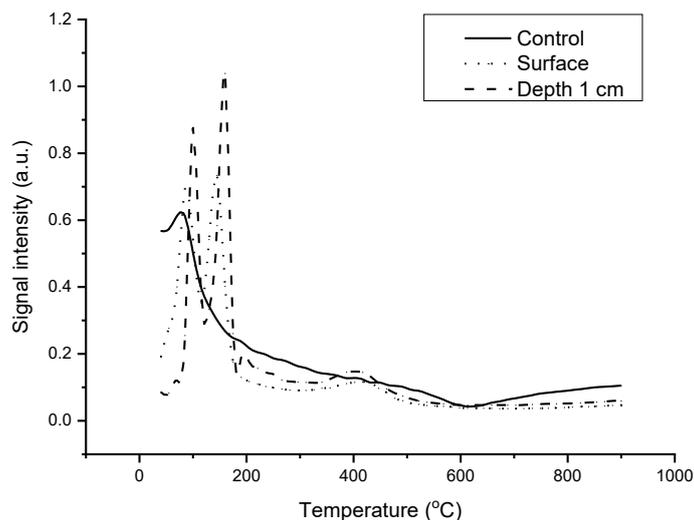


Fig. 11. Thermograms of H<sub>2</sub>O release ( $m/z = 18$ ) from the sample obtained in the finished product storage room

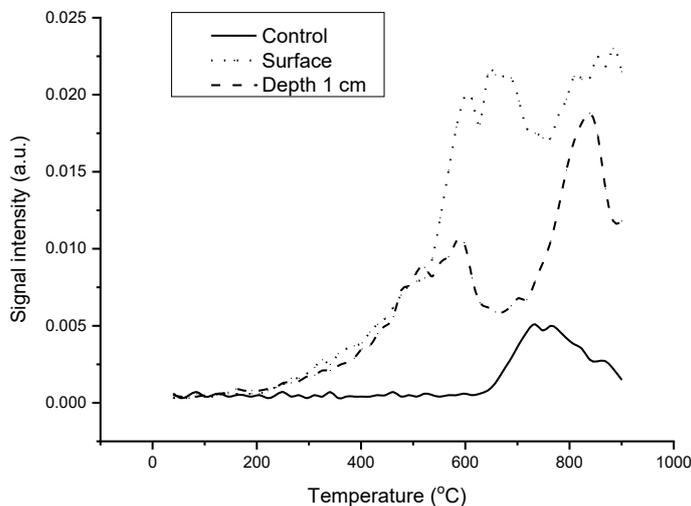


Fig. 12. Thermograms of SO<sub>2</sub> release ( $m/z = 64$ ) from the sample obtained in the finished product storage room

In concrete samples obtained from the control room, SO<sub>2</sub> is released with a low intensity of 0.004 at  $t = 800$  °C. Experimental samples of concrete, when heated to a temperature of 600–800 °C, emitted sulfur dioxide with an intensity of 0.01 to 0.022 [33]. The TPD MS results indicate the typical SO<sub>2</sub> content of control and test concrete samples.

However, during the experiment, samples were taken at different depths from different sections of concrete structures, where the extent of biochemical corrosion could reach different distributions.

Concrete samples were obtained from a building structure that had been subjected to uncontrolled biochemical corrosion for a long time. Therefore, the study has certain limitations associated with the fact that it was quite difficult to select a similar object for comparison and control. For a reliable difference in the results obtained, samples for research were taken from different buildings of a chemical enterprise, which had approximately the same building age. Therefore, the experiment cannot be considered perfect, but it allows one to predict the development of biological and chemical corrosion over time.

#### 4. Conclusions

Acidithiobacillus thiooxidans bacteria, which affect the chemical composition of concrete, were isolated by the sulfate method in the workshop for the production of titanium dioxide. During TPD-MS, the CO<sub>2</sub> level in the test concrete samples is practically absent, but the intensity of SO<sub>2</sub> emission is proportionally increased compared to the control sample. Depending on the degree of corrosion, the intensity of evaporation of H<sub>2</sub>O and O<sub>2</sub> in test concrete samples exceeds the level by 30–35 % compared to the control ones. In concrete, gypsum crystals (CaSO<sub>4</sub>·2H<sub>2</sub>O) detected by electron microscopy were obtained from the surface of concrete structures in a titanium dioxide production workshop.

In the storage room for finished products, microscopic and electron microscopic studies from the surface of concrete structures isolated the fungus *Aspergillus fumigatus* and calcium citrate crystals. In the test specimens, concrete samples from the surface and a depth of 1 cm, CO<sub>2</sub> was not released as a result of the metabolism of the fungus *Aspergillus fumigatus*, which prevents the formation of calcium carbonate. The TPD MS results indicate the typical SO<sub>2</sub> content of control and test concrete samples in the finished product storage room.

The TPD MS method is proposed for studying the biochemical corrosion of concrete, determining the chemical composition of concrete, which occurs under the action of an aggressive environment and the dissimilation of microorganisms. Based on the results obtained, it is possible to develop antimicrobial and anticorrosive protective coatings to stop the biochemical corrosion of concrete in a chemical plant.

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