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LABORATORY ANALYSIS OF SELF-HEALING CEMENT COMPOSITION BASED ON CALCIUM LACTATE AND BACTERIA

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A B S T R A C T

The research aims to evaluate the feasibility of using bacteria-based self-healing concrete using Portland cement and assess the effect of temperature on its performance. The results showed that the inclusion of calcium lactate and bacteria in the mixture accelerates the gain in compressive strength, but after 28 days of curing, the healing agent has no impact on the overall compressive strength value of the mixture. The crack width distribution analysis revealed an inverse relationship between crack width and self-healed area, with wider cracks having lower self-healing rates. Most of the healing occurs within 15 days, with only a small fraction healing between day 15 and 60 days. The study also showed that low temperatures do not produce self-healing in tested samples, and 25°C increases the self-healed area for all crack widths. Finally, chromatography tests of submerged water reveal that reaction to seal the cracks takes calcium from some external source.

Keywords: self-healing cement; carbon emission; bacteria; calcium lactate; Portland cement.

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1. Introduction

The concrete industry is a major player in the global economy, with production levels exceeding 6 million cubic meters per year [1]. Unfortunately, the industry is also one of the most polluting, contributing to around 7% of global CO_2 emissions, which is the main cause of climate change. Cement plants, a significant component of the concrete production process, emit large amounts of CO_2 due to the high temperature requirements necessary for clinker production. This process demands a substantial amount of energy, which further exacerbates the industry's impact on the environment [2-7].

Concrete is known for its compressive strength, but its low tensile strength makes it prone to cracking under load [8-11]. To prevent this from leading to structural failure, steel reinforcements are installed to carry the tensile forces after cracking. However, cracks can still affect the durability and longevity of concrete structures by increasing permeability and allowing undesirable fluids to pass through, leading to corrosion of steel reinforcements, concrete degradation, and potential structural failure [12-16]. Leakage through cracks can also affect the functionality of concrete structures such as water reservoirs, roofs, and pipes.

Man-made repairs are often costly and inefficient, especially when access to the damaged concrete is limited, such as in underground concrete structures [17-18]. Therefore, self-healing concrete has become an attractive solution for mitigating cracking, improving durability, and reducing the negative environmental impacts associated with concrete production and repair [19].

There are various techniques for enhancing the self-healing capacity of concrete, such as mineral admixtures, adhesive agents, and bacteria [20-21]. The use of bacteria for self-healing concrete has gained popularity in recent years, where the incorporation of mineral-precipitating bacterial spores with nutrients in the concrete mixture has proven effective. When the dormant bacteria comes in contact with water, it becomes active and starts metabolizing and precipitating calcium carbonate, which can fill cracks to a certain extent and reseal the concrete matrix [22-26].

While some researchers have proven the effectiveness of certain bacteria in the self-healing of concrete, there are still limitations to the practical application of this technique due to high costs and a lack of performance data. Factors such as cement type and temperature can also affect bacterial activity and biomineralization. Therefore, there is a need to understand the interaction of environmental variables with self-healing concrete to determine the feasibility of using this method in different locations and climatic conditions [27-29].

In summary, the research aims to evaluate the feasibility of using bacteria-based self-healing concrete using Portland cement and assess the effect of temperature on its performance. The ultimate goal is to develop more sustainable and environmentally friendly solutions for the concrete industry, reduce the need for man-made repairs, and contribute to the mitigation of climate change.

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2. Materials and test methods

2.1. Cultivation of bacteria

In this study, the Bacillus pseudofirmus bacterium was chosen as the subject for investigation due to its ability to form spores and produce calcite, making it an ideal candidate for concrete crack healing. The strain, obtained from LGC Group, is a spore-forming bacterium that is capable of growing in a basic medium can withstand external pH fluctuations. To ensure the bacterium's viability, lyophilized bacteria stocks were stored in glycerol at - 80 °C before cultivation in the microbiology laboratory.

The cultivation process began with the inoculation of the lyophilized bacteria into a liquid medium to create an initial bacterial culture, which was then used to inoculate a second culture to enhance spore formation, following the method described by Tziviloglou et al. [30].

To create the healing agent, the bacteria agent, calcium lactate (200 g/L), and yeast extract (5 g/L) were mixed with a spatula in a sterile glass. The resulting healing agent was then impregnated into the lightweight aggregate (LWA), which is a porous and lightweight material that is suitable for the impregnation of the healing agent. The healing agent used in the study was 15% of the LWA weight.

2.2. Mortar mixture design

To investigate the self-healing ability of different cement types, researchers prepared three mortar mixtures for each type: Base slurry (P), Lactate only (L), and Bacteria mixtures (B). All mixtures were made with the same amount of cement, free water, and lightweight aggregate (LWA). In the base slurry (P), LWA was pre-soaked in tap water. In the lactate mixture (L), LWA was pre-soaked in calcium lactate (without the healing agent). In the bacteria mixtures (B), LWA was pre-soaked with the healing agent containing bacteria, lactate, and yeast, as described earlier.

The water to cement ratio was kept constant at 0.50 for all slurries to increase cement hydration and reduce the potential for autogenous self-healing of concrete, which could interfere with the self-healing effect of bacteria.

2.3. Specimen preparation, curing and experimental matrix

The preparation of the flexural beams (40 mm × 40 mm × 160 mm) and cubical specimens (2'' × 2'') was carried out according to the standard procedures for such tests. The flexural beams were used for visual self-healing quantification and were cracked by applying a monotonic load using a three-point bending frame. The cracked specimens were placed in water at different temperatures (25 and 50 °C) to assess the effect of temperature on self-healing.

The self-healing degree of the mortars was quantified by periodically monitoring the cracks. On the other hand, cubic specimens were fabricated to measure compressive strength.

2.4. Cracking and self-healing process of flexural specimens

After a period of 28 days of curing, the flexural specimens underwent loading to induce flexural cracking, primarily located at the bottom of the specimen. To carry out this process, a universal testing machine configured for three-point bending tests was employed. Throughout each test, the machine control system recorded the peak load and deformation values. Subsequently, the cracked specimens were placed in plastic containers filled with tap water. These containers, each containing groups of cracked flexural specimens, were subjected to a constant temperature of either 25 or 50 °C for a duration of 60 days. At specific intervals following the cracking, individual specimens were retrieved from the water to undergo crack-healing quantification through image analysis.

2.5. Image analysis of the self-healing

In this study, a camera and lens were used to capture images of the cracked specimens, and the images were aligned to quantify changes in crack width over time. The healing of crack width was calculated using the equation below:

$$\text{healing } \% = \frac{w_i - w_t}{w_i} \times 100\%$$

where w_i is the initial crack width, w_t is the crack width at time t.

The healing of crack area was also calculated by setting a threshold gray level for the crack area and using the below equation:

$$\text{healing area } \% = \frac{a_i - a_t}{a_i} \times 100\%$$

where, a_i is the initial crack area, a_t is the crack area at time t.

2.6. Chromatography

The chromatography analysis was carried out using the IC 930 Compact Flex Metrohm chromatograph.

3. Results and Discussion

3.1. Compressive strength

The test aimed to investigate the impact of lactate and bacteria on the compressive strength of cubic samples. The results indicated that all three mixtures exhibited similar trends, with a rapid increase in strength during the first 7 days of curing and a reduced rate of strength gain thereafter. After 28 days of curing, the compressive strength of all samples was quite similar and the differences between mixtures were insignificant.

However, samples prepared with calcium lactate (*L*) and with calcium lactate plus bacteria (*B*) exhibited the highest strength after three days of curing, with 14% and 12% more strength, respectively, than the average strength of samples prepared from base slurry. After seven days of curing, the strength of *L* and *B* samples was 9% and 7% higher than

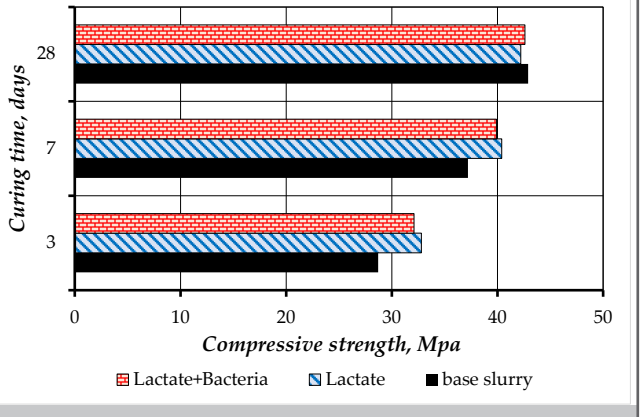

Fig. 1. Compressive strength of tested samples in regard with time

that of base sample, respectively. Hence, the findings suggest that the inclusion of calcium lactate accelerates the gain in compressive strength. However, after 28 days of curing, the healing agent has no impact on the final compressive strength value of the mixture.

3.2. Crack width frequency and relationship between crack width and self-healed area

The three-point bending test was conducted to create cracks in hardened mortar samples for a self-healing study. Although effective, the test produced uncontrolled crack dimensions that varied between samples. The authors analyzed the cracks statistically and found that a Beta probability distribution described the crack width well. Most cracks measured between 0.125 mm and 0.225 mm, with a maximum of 0.475mm. The self-healing study revealed an inverse relationship between crack width and self-healed area. All cracks below 0.075mm self-healed, while wider cracks had lower self-healing rates (fig. 2).

3.3. Analysis of images on visible cracks

Figure 3 displays the self-healing of a hardened mortar specimen made with bacteria, cured at 25 °C. The figure shows the crack immediately after flexural loading (0 days), after 15 and 60 days of curing. The image was adjusted to improve contrast and sharpness. After 15 days, 2/3 of the crack is recovered, and a narrowing effect is visible in part of the wider section.

3.4. Effect of lactate and bacteria on the self-healing of samples

The study analyzed the self-healing capabilities of different mixture types in cement samples with varying initial crack widths. The results showed that all mixtures achieved 100% self-healing for cracks smaller than 0.1 mm width. However, for larger crack widths, the mixtures with lactate only and lactate + bacteria showed a higher self-healed area compared to base slurry (fig. 4). The authors also assessed the self-healing by percentage of cracks with a self-healed area larger than 75%. Results showed that lactate and lactate + bacteria mixtures had a higher capability of self-healing cracks (74 and 55 %) than base slurry (26%). The results suggest that adding lactate can increase the self-healing capabilities of cement mixtures.

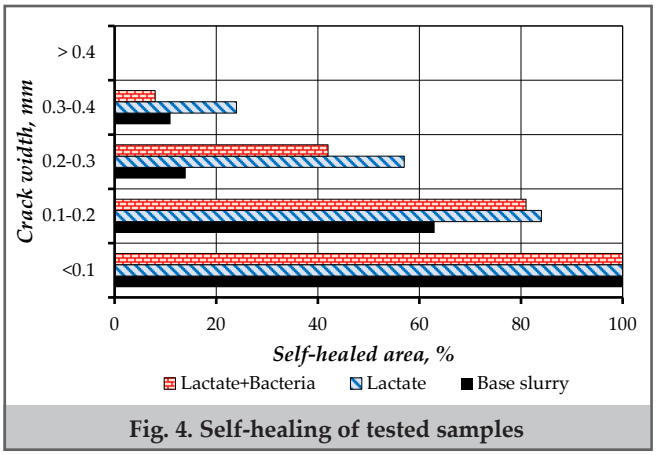

Fig. 4. Self-healing of tested samples

3.5. Effect of temperature on the self-healing

The self-healing capabilities of samples conditioned for 60 days at different temperatures are shown in Figure 5. It worth noting that that low temperatures (below 5 °C) did not produce self-healing in samples, therefore was not shown in graph.

The results obtained with lactate + bacteria provide conclusive evidence that a temperature of 25 °C promotes an increase in the self-healed area for cracks of all widths. This finding aligns perfectly with the characteristic behavior of Bacillus pseudofirmus, the mesophilic spore utilized in

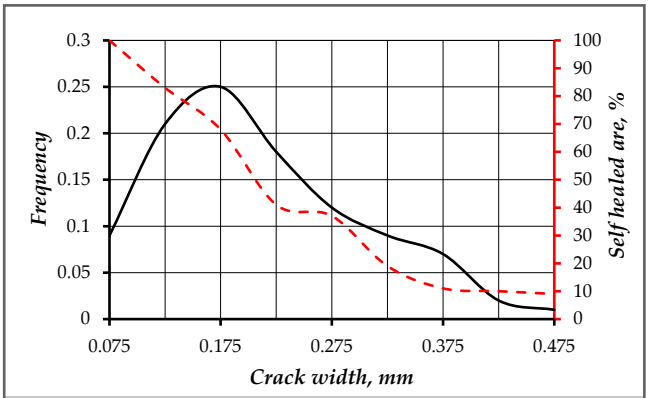

Fig. 2. Self-healed area and crack width frequency versus crack width after 60 days of curing

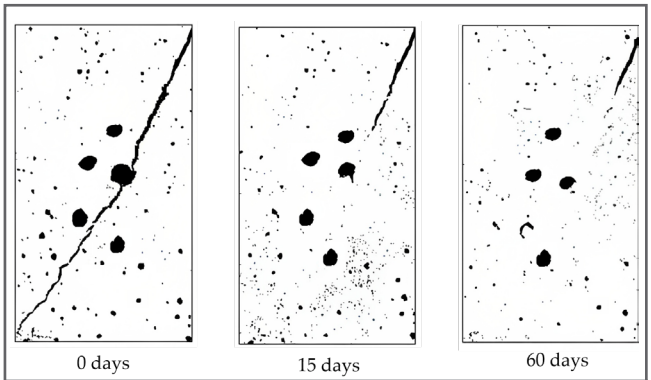

Fig. 3. Self-healing of samples versus time

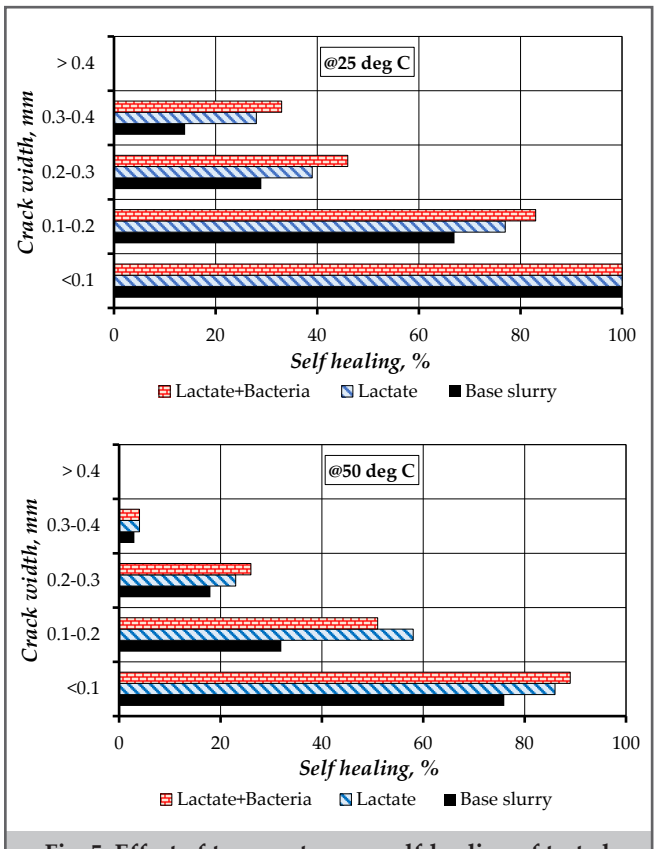

Fig. 5. Effect of temperature on self-healing of tested samples

this study, as it exhibits optimal growth within a moderate temperature range typically between 20 and 40 °C.

Specifically, for narrow cracks measuring less than 0.1 mm, the self-healed area achieved a remarkable 100% recovery for both the temperatures of 25 °C and 50 °C.

3.6. Self-healing of a single crack

The study conducted self-healing measurements in predetermined intervals starting from 0 days and final measurement was made at 60 days by considering the crack width. However, the crack-repair versus time analysis involved measuring the total self-healing area without considering the initial width to track the crack-healing evolution. Figure 6 presents the total self-healed area for one crack sample from 0 to 60 days. However, since only one crack was analyzed for the crack evolution, Figure 6 does not entirely follow the trends presented above, which considers all the cracks in the sample.

The results show that the specimens gradually increased the total self-healed area until day 40 after the initial cracking, after which the healing plateaued and remained relatively constant. Additionally, the results suggest after 15 days of curing, the crack-healing is approximately two-thirds of the final total self-healed area.

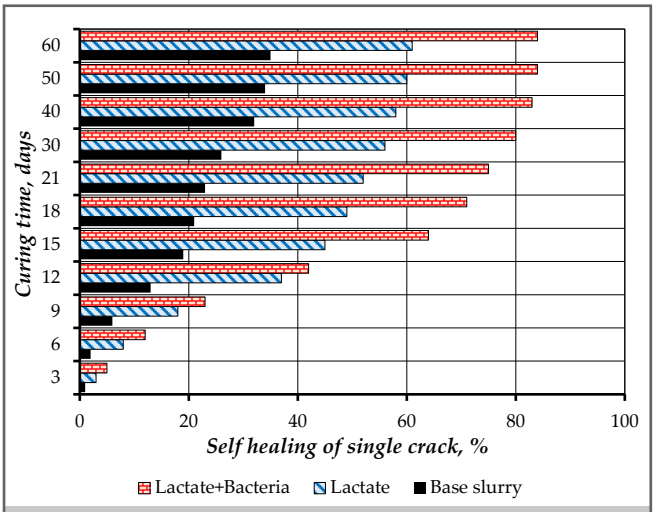

Fig. 6. Self-healing of a single crack for all tested samples

3.7. Chromatography

The authors conducted chromatography on water used for soaking the flexural specimens. The first water sample was collected at the beginning of the experiment, before the samples were soaked. Therefore, the first water sample did not have any contact with concrete specimens. After the 60 days of keeping the samples submerged, a second water sample was collected and analyzed. According to Table 1, the results show a decrease in calcium and magnesium cations and an increase in sodium and potassium. The decrease in calcium ions is consistent with the hypothesis that the reaction to seal the cracks takes calcium from some external source. Hence, part of the calcium cations found in the water would be responsible for the formation of calcium carbonate in the cracks. The increase in sodium and potassium cations could be due to the transfer of concrete components to the water.

Table 1

Results of chromatography tests

Ions	Water before submerging specimens, ppm	Water after 60 days of submersion, ppm
Na+	52.5	278.4
K^+	36.1	293.1
Ca^{2+}	39.3	2.4
Mg^{2+}	12.4	1.5

Conclusion

The study investigated the effect of lactate and bacteria on the compressive strength and self-healing capabilities of Portland cement mixtures. The results showed that the inclusion of calcium lactate in the mixture accelerates the gain in compressive strength, but after 28 days of curing, the healing agent has no impact on the compressive strength of the mixture. The crack width distribution analysis revealed an inverse relationship between crack width and self-healed area, with wider cracks having lower self-healing rates. Most of the healing occurs within 15 days, with only a small fraction healing between day 15 and 60 days. The study also found that adding lactate can increase the self-healing capabilities of cement mixtures, with lactate and lactate + bacteria mixtures having a higher capability of self-healing cracks than base samples. The study also showed that low temperatures do not produce self-healing in tested samples, and 25 °C increases the self-healed area for all crack widths. Finally, chromatography tests of submerged water reveal that reaction to seal the cracks takes calcium from some external source. Taking into account this phenomena, impact of divalent ions of submerged water in self-healing should be investigated in future researches.

Conclusion

1. Nguyen, T. H., Ghorbel, E., Fares, H., Cousture, A. (2019). Bacterial self-healing of concrete and durability assessment. *Cement and Concrete Composites*, 104, 103340.

2. Van Oss, H. G., Padovani, A. C. (2002). Cement manufacture and the environment: part I: chemistry and technology. *Journal of Industrial Ecology*, 6(1), 89-105.

3. Van Oss, H. G., Padovani, A. C. (2003). Cement manufacture and the environment part II: environmental challenges and opportunities. *Journal of Industrial Ecology*, 7(1), 93-126.

4. Devi, K. S., Lakshmi, V. V., Alakanandana, A. (2017). Impacts of cement industry on environment. An overview. *Asia Pacific Journal of Research*, 1, 156-161.

5. Huntzinger, D. N., Eatmon, T. D. (2009). A life-cycle assessment of Portland cement manufacturing: comparing the traditional process with alternative technologies. *Journal of Cleaner Production*, 17(7), 668-675.

6. Mohamad, N., Muthusamy, K., Embong, R., et al. (2022). Environmental impact of cement production and Solutions: A review. *Materials Today: Proceedings*, 48, 741-746.

7. Veliyev, E. F., Aliyev, A. A. (2021). Innovative technologies as a priority factor of the oil and gas industry development. *ANAS Transactions. Earth Sciences*, 2, 81-93.

8. Suleimanov, B. A., Veliyev, E. F., Shovgenov, A. D. (2022). Well cementing: fundamentals and practices. *Moscow-Izhevsk: ICS*.

9. Suleimanov, B. A., Veliyev, E. F., Vishnyakov, V. (2022). Nanocolloids for Petroleum Engineering: Fundamentals and Practices. *John Wiley & Sons*.

10. Suleimanov, B. A. (2006). Specific features of heterogenous systems flow. Moscow-Izhevsk: ICS.

11. Suleimanov, B. A., Veliyev, E. F. (2016). The effect of particle size distribution and the nano-sized additives on the quality of annulus isolation in well cementing. *SOCAR Proceedings*, 4, 4-10.

12. Aliyev, A. A. (2021). Improvement of rheological properties of alkaline-activated geopolymers using anhydrous-based process fluids. *Prospecting and Development of Oil and Gas Fields*, (3 (80)), 60-67.

13. Veliyev, E. F., Aliyev, A. A. (2022). Comparative analysis of the geopolymer and Portland cement application as plugging material under conditions of incomplete displacement of drilling mud from the annulus. *SOCAR Proceedings*, 1, 108-115.

14. Vrålstad, T., Skorpa, R., Werner, B. (2019, March). Experimental studies on cement sheath integrity during pressure cycling. SPE-194171-MS. In: *SPE/IADC International Drilling Conference and Exhibition*. *Society of Petroleum Engineers*.

15. Kuanhai, D., Yue, Y., Yi, H., et al. (2020). Experimental study on the integrity of casing-cement sheath in shale gas wells under pressure and temperature cycle loading. *Journal of Petroleum Science and Engineering*, 195, 107548.

16. Zhang, H., Xu, Y., Gan, Y., et al. (2019). Combined experimental and numerical study of uniaxial compression failure of hardened cement paste at micrometre length scale. *Cement and Concrete Research*, 126, 105925.

17. Austin, S., Robins, P., Pan, Y. (1999). Shear bond testing of concrete repairs. *Cement and concrete research*, 29(7), 1067-1076.

18. Jingtai, N., Youwen, P., Congcong, W., Bowen, W. (2012). Cement loop damage-based fracture mechanism during repair of casing failure well. *Procedia Earth and Planetary Science*, 5, 322-325.

19. Zhang, W., Zheng, Q., Ashour, A., Han, B. (2020). Self-healing cement concrete composites for resilient infrastructures: A review. *Composites Part B: Engineering*, 189, 107892.

20. Ferrara, L., Van Mullem, T., Alonso, M. C., et al. (2018). Experimental characterization of the self-healing capacity of cement based materials and its effects on the material performance: A state of the art report by COST Action SARCOS WG2. *Construction and Building Materials*, 167, 115-142.

21. Muhammad, N. Z., Shafaghat, A., Keyvanfar, A., et al. (2016). Tests and methods of evaluating the self-healing efficiency of concrete: A review. *Construction and Building Materials*, 112, 1123-1132.

22. Jonkers, H. M., Thijssen, A., Muyzer, G., et al. (2010). Application of bacteria as self-healing agent for the development of sustainable concrete. *Ecological Engineering*, 36(2), 230-235.

23. Vijay, K., Murmu, M., Deo, S. V. (2017). Bacteria based self healing concrete – A review. *Construction and Building Materials*, 152, 1008-1014.

Лабораторный анализ состава самовосстанавливающегося цемента на основе лактата кальция и бактерий

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Реферат

Цель исследования - оценить возможность применения самовосстанавливающегося бетона на основе бактерий с использованием портландцемента и оценить влияние температуры на его характеристики. Результаты показали, что включение в смесь лактата кальция и бактерий ускоряет набор прочности на сжатие, но после 28 дней твердения реагент самовосстановлений не оказывает влияния на общее значение прочности смеси на сжатие. Анализ распределения ширины трещин выявил обратную зависимость между шириной трещин и площадью самовосстановления, при этом более широкие трещины имеют более низкую скорость самовосстановления. Большая часть трещин восстанавливается в течение 15 дня, и лишь небольшая часть - в период с 15-го по 60-й день. Исследование также показало, что низкие температуры не вызывают самовосстановления в исследуемых образцах, а температура 25 °C увеличивает площадь самовосстановления при любой ширине трещин. Наконец, хроматографические анализы погруженной воды показывают, что для реакции восстановление трещин необходим кальций из какого-то внешнего источника.

Ключевые слова: самовосстанавливающийся цемент; эмиссия углекислого газа; бактерии; лактат кальция; портландцемент.

Kalsium laktat və bakteria əsaslı öz-özünü bərpa edə bilən sement tərkibinin laboratoriya analizi

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Xülasə

Tədqiqatda bakteria və portlandsement əsaslı öz-özünü bərpa edən sement tərkibi hazırlanmış və müxtəlif temperaturlarda tədqiq olunmuşdur. Nəticələr göstərir ki, kalsium laktat və bakterianın əlavəsi sementin möhkəmliyin qısa müddətdə yaranmasına gətirib çıxarır, lakin sıxılmaya qarşı müqavimətin son qiymətinə təsir etmir. Çatların qalınlığının tədqiqi göstərmişdir ki, daha qalın çatlar daha gec bərpa olunur, nəinki, qalınlığı az olan çatlar. Bərpa əsasən ilk 15 gündə baş verir, 15-60 günlər arasında bərpanın faizlə nisbəti çox kiçik olur. Tədqiqatlar həmçinin göstərmişdir ki, çox aşağı temperaturlarda sementin bərpa olunması müşahidə olunmur və ən çox bərpa 25oC temperaturda müşahidə olunmuşdur. Son olaraq, sement nümunələrinin saxlandığı suyun xromotoqraf analizi göstərmişdir ki, bərpa reaksiyası üçün kənar mənbələrdən kalsium tələb olunur.

Açar sözlər: öz-özünü bərpa edən sement; karbon emissiyası; bakteria; kalsium laktat; Portland sement.