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SELECTED PROPERTIES AND MICROSTRUCTURE OF CONCRETE WITH TIRE RUBBER GRANULATE AS RECYCLED MATERIAL IN CONSTRUCTION INDUSTRY

Zuzana MARCALIKOVA[1,*], Radoslav GANDEL[1], Jan JERABEK[1], Jaromir VARAK[1]

[1] Department of Building Materials and Diagnostics of Structures, Faculty of Civil Engineering, VSB - Technical University of Ostrava, Ludvíka Podéště 1875/17, 708 00 Ostrava-Poruba, Czech Republic
[*] corresponding author: zuzana.marcalikova@vsb.cz

Abstract

The paper explores the use of recycled materials in the construction industry to promote sustainable development. There is a growing demand for recycling and innovative materials in engineering. The study specifically investigates the potential of tire rubber recyclate as a recycled raw material, comparing two different mixtures in an experimental program. These mixtures highlight the importance of utilizing local resources, aligning with the principles of the circular economy. The experimental program focuses on evaluation of mechanical properties in addition to specialized tests. Findings indicate that higher proportions of rubber granulate not only impact mechanical properties but also significantly affect durability when exposed to environmental factors.

Keywords:

Rubber recyclate;
Mechanical properties;
Durability;
Alternative materials;
Sustainability.

1 Introduction

It's challenging to envision present and future building structures without concrete, yet the very nature of concrete will undergo significant innovation to meet current demands and possibilities. Key components of concrete, including binder, filler, and water, are constrained by available raw materials and energy resources [1, 2]. Concrete is known for its quasi-brittle characteristics, exhibiting distinct properties under compression and tension. Extensive research worldwide, as well as in our country, focuses on understanding concrete properties and conducting experiments. Promising solutions include substituting cement with alternative binders [3-5], such as alkali-activated materials, hybrid concrete, or generally speaking, composites [6-8]. However, technological and rheological challenges hinder the widespread adoption of alkali-activated materials. Specialized application solutions, like hybrid beams, are worth exploring for these materials, emphasizing rigorous testing for both mechanical properties and durability. Another approach involves high-performance concrete (HPC), which still uses cement but offers superior technical and practical advantages over conventional concrete, particularly in terms of durability and environmental impact [9]. Fascinating possibilities emerge in applications such as bridge construction or integrating fibers into concrete, leading to the development of fiber-reinforced concrete [10-12]. Recycling is also being considered in concrete design, with the potential use of recycled rubber as a partial aggregate replacement [13, 14], as discussed in the referenced study presenting an experimental program comparing two mixtures with varying amounts of rubber recyclates.

In addition to the often discussed mechanical properties of concrete composites, it is also important to focus on their composition and microstructure for a better understanding of the subject. There are many methods and devices for observing the microstructure of building materials, among the most commonly used are optical and electron microscopy. Optical microscopy uses visible light and an array of lenses to illuminate specimens and provides lower resolution compared to electron microscopy, making it suitable for observing larger features and general morphology. Its advantages include simpler and cheaper equipment and simple and rapid sample preparation. Scanning electron microscopy uses a beam of electrons for illumination, which has the advantage of much higher resolution than optical microscopy and thus allows the observation of fine details at the nanometre scale. In addition to its high resolution, its advantages also include detailed information on surface and internal structure and the possibility of combining it with elemental analysis techniques such as EDS. However, it involves more complex and expensive equipment, requires extensive sample preparation and can potentially damage samples due to electron beam exposure [15].

2 Methodology and the Experimental Program

The presented work is an extended paper of the YOUNG SCIENTIST 2024 conference [16]. The study outlines an extensive experimental program, initially concentrating on the basic strength-mechanical properties of concrete. Subsequently, specialized tests assessing durability are conducted. In the first case, 70 kg/m^3 of rubber granulate is incorporated, while the second variant utilizes 140 kg/m^3 of granulate, replacing a portion of the aggregate. The manufacturer declare the granularity of the rubber granulate as 1-3 mm, with a bulk density of 448 kg/m^3 ± 5% [17]. The relevant photo documentation is provided in Figure 1.

Fig. 1: Used rubber granulate [16].

In both cases, the mixture composition per cubic meter of fresh mix included: CEM I 42.5 R cement from Hranice, Czech Republic (I - 410 kg, II - 380 kg), water (I - 185 kg, II - 172 kg), Polyether carboxylate plasticizer (I - 2.7 kg, II - 2.5 kg), Tovačov aggregate 0-4 mm (I - 930 kg, II - 700 kg), Litice aggregate 4-8 mm (I - 500 kg, II - 470 kg), and recycled rubber (I - 70 kg, II - 140 kg). In the case of mixture II, the quantity of recycled rubber was doubled. Both mixtures had a water ratio of 0.45, with the average bulk density of hardened samples for the first mixture reaching 2194.6 kg/m^3, and for the second one, 2091 kg/m^3.

Table 1 presents the average compressive strengths (in accordance with standard ČSN EN 12390-3, Testing hardened concrete – Part 3: Compressive strength of test specimens [18] and tensile splitting strengths (due to standard ČSN EN 12390-6, Testing hardened concrete - Part 6: Tensile splitting strength of test specimens [19]) measured after 28 days. These tests were conducted on cubes with a side length of 150 mm, with each test series containing 5 samples.

Table 1: Characteristics of strength and their corresponding indicators of variability [20].

Mixture	Compressive strength [MPa]	Standard deviation [MPa]	Coefficient of variation [%]	Tensile splitting strength [MPa]	Standard deviation [MPa]	Coefficient of variation [%]
Mix. I	37.97	2.09	5.49	2.40	0.33	13.86
Mix. II	22.09	0.96	4.37	1.55	0.05	3.00

The data presented in Table 1 illustrates a decrease in both compressive strength and tensile splitting strength as the proportion of rubber granules in the mixture increases. When the amount of rubber granules was doubled, there was a nearly 42% decrease in compressive strength compared to mixture I, and a 35% decrease in tensile splitting strength, also in comparison to mixture I. Additionally, mixture I exhibited a greater dispersion in the statistical dataset for tensile splitting strength testing. The coefficient of variation reached approximately 13.9%. The dispersion of compressive strength values was similar and relatively low for both mixtures, with the coefficient of variation ranging from 4.4 to 5.5%.

The following charts in Figures 2 and 3 showcase the evolution of flexural strength and compressive strength over time. These assessments were performed on beams measuring 40x40x160 mm. For one-day, seven-day, and yearly strengths, six samples were used, while for seventeen-day and twenty-eight-day strengths, twelve samples were used. These evaluations were conducted in accordance with the specifications outlined in standard ČSN EN 196-1, Methods of testing cement - Part 1: Determination of strength [21].

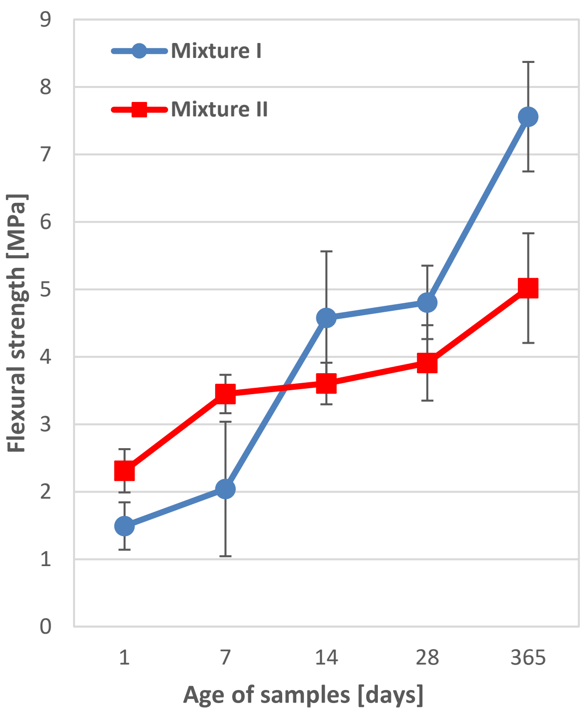

Fig. 2: Development of flexural strength [20].

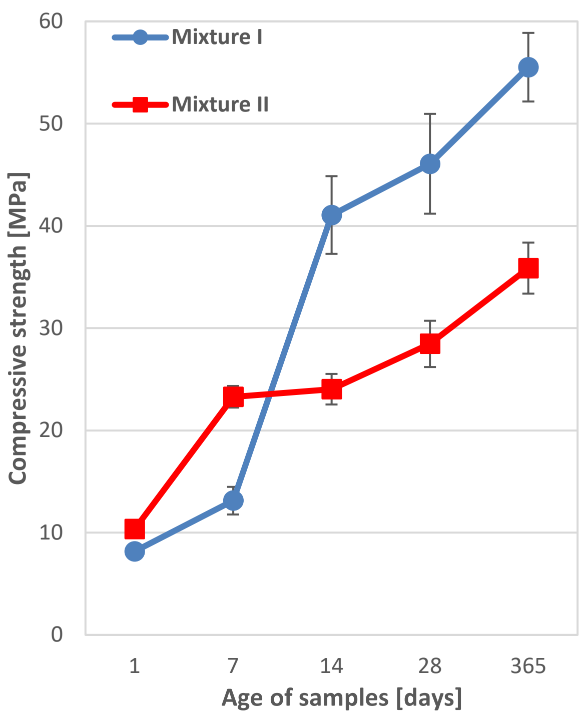

Fig. 3: Development of compressive strength [20].

From the graphs in Figures 2 and 3 above, it's noticeable that mixture 2 achieved higher values for both flexural and compressive strength within the initial 7 days, with only a slight increase from the 14th day. On the other hand, mixture 1 exhibited a notable surge in both strengths between days 7 and 14, even though with a wider range of values compared to mixture 2. By the 28th day, mixture 1 samples demonstrated a flexural strength of 4.8 MPa and a compressive strength of 46.1 MPa, while mixture 2 reached a flexural strength of 3.9 MPa and a compressive strength of 28.5 MPa. Furthermore, in mixture 1, there was observed an increase in yearly flexural strength by approximately 57% (7.6 MPa) and compressive strength by approximately 20% (55.5 MPa) compared to the standard 28-day values.

Mixture 2, on the other hand, experienced higher increase in flexural strength - the yearly value achieved approximately 28% more (5 MPa) and similar increase the compressive strength (35.9 MPa – 26 %). The progress of these tests is shown in Figures 4 and 5.

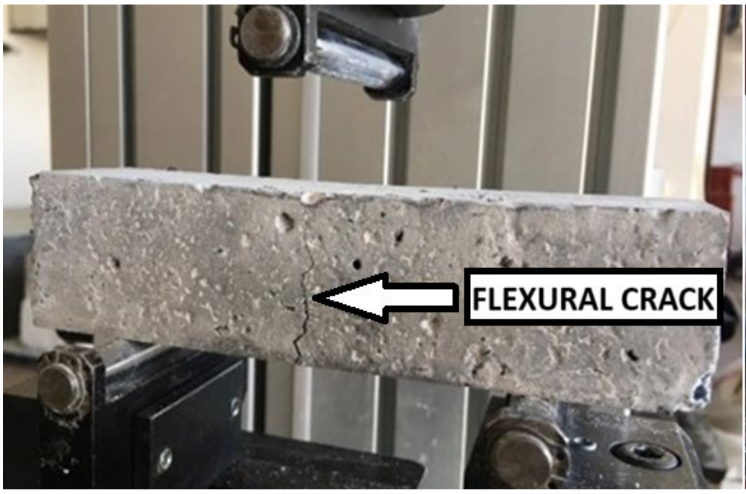

Fig. 4: Crack after test of flexural strength [16].

Fig. 5: Specimen failure after compressive strength test with load area of 40x40 mm [16].

In addition to other evaluations, the mentioned mixtures underwent durability assessments, specifically frost resistance tests (in accordance with standard ČSN 73 1371 Determination of frost resistance of concrete [22]), examined using sets of 6 beams for every mixture and specified cycle duration, each beam measuring 40x40x160 mm. Additionally, for each mixture, 3 cubes with edge measuring 100 mm was tested to resistance to water and defrosting chemicals due to standard ČSN 73 1326 Resistance of cement concrete surface to water and defrosting chemicals [23]). Bar chart in Figure 6 presents the coefficient of frost resistance following individual cycles.

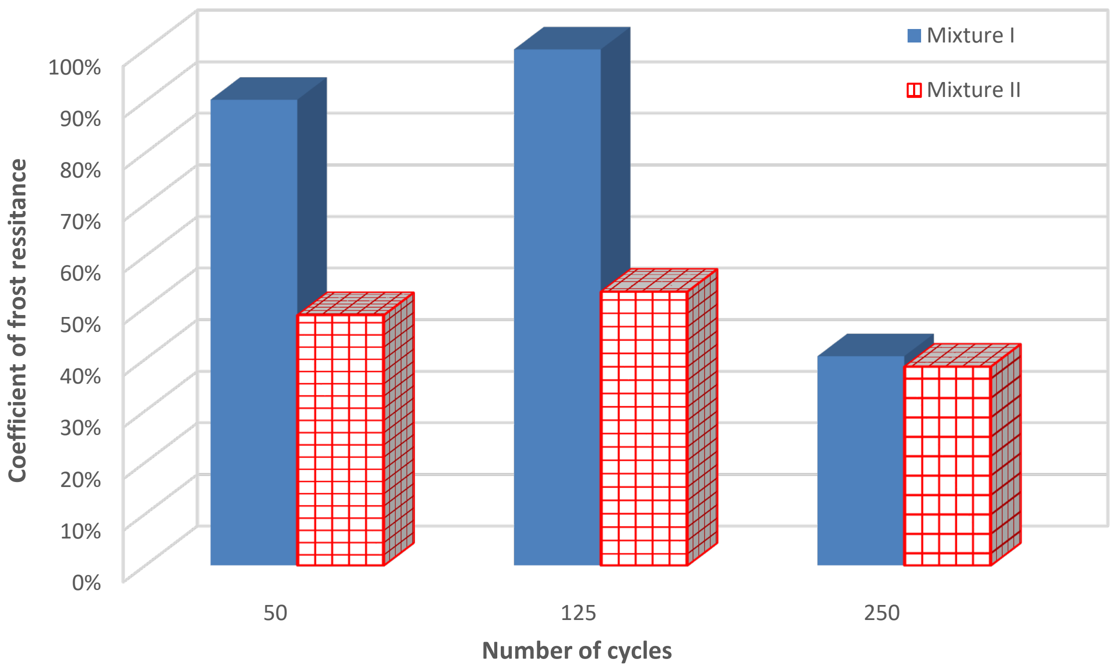

Fig. 6: Coefficient of frost resistance of tested mixtures after individual cycles [20].

Based on the findings presented in Figure 6 and in accordance with the standard ČSN 73 1371 [24], it can be concluded that mixture **I** exhibits frost resistance for 125 cycles, whereas mixture **II** does not demonstrate frost resistance even for 50 cycles.

Table 2 gives the values of the flexural (f_t) and compressive (f_c) strength ratios with respect to the individual cycles of the frost resistance test.

Table 2: The ratios of (f_t/f_c) of tested mixtures before and after frost resistance tests [20].

Number of cycles	0	50	125	250
Mix. I	10.4%	10.2%	10.8%	6.2%
Mix. II	13.7%	10.2%	8.6%	8.4%

Based on the frost resistance coefficients provided in Figure 6 and the data from Table 2, it is evident that Mixture **I** was resistant to freezing for up to 125 cycles. After 250 cycles, the samples degraded rapidly, which was also reflected in its strength characteristics (f_t/f_c ratio decreased by approximately 4.6%). Mixture **II** showed a deterioration in frost resistance at 50 cycles, when the f_t/f_c ratio decreased by 3.5% compared to the samples not subjected to the frost resistance test.

Another aspect monitored during the frost resistance test was the variation in the dynamic modulus of elasticity, assessed using the ultrasonic pulse testing method outlined in standard ČSN 73 1371 Non-destructive testing of concrete – Method of ultrasonic pulse testing of concrete [24]. This testing was also performed on beams measuring 40x40x160 mm, and the results are presented in Table 3.

Table 3: Change in dynamic modulus of elasticity after frost resistance test [20].

Number of cycles	Mix. I - Dynamic modulus of elasticity before frost resistance test [GPa]	Mix. I - The percentage decrease of the dynamic modulus of elasticity after individual cycles of frost resistance test	Mix. II - Dynamic modulus of elasticity before frost resistance test [GPa]	Mix. II - the percentage decrease of the dynamic modulus of elasticity after individual cycles of frost resistance test
0	38.83	-	33.13	-
50	41.30	3.67%	34.33	32.39%
125	42.21	1.28%	33.40	17.24%
250	41.46	31.60%	33.71	33.24%

The values in Table 3 show that there is a slight decrease in the dynamic modulus of elasticity up to 125 cycles for Mixture **I** (overall decrease up to 3,67%). After 250 cycles there is a decrease of more than 30%. Mixture **II** showed a decrease in dynamic modulus of elasticity of more than 36% after 50 cycles (except for a minor decrease of 17,24 % after 125 cycles). Throughout the entire testing period, the dispersion of values for the dynamic modulus of elasticity of mixture **II** varied.

The higher porosity of the resulting composite for both mixture as well as the surface porosity of the rubber recyclate itself caused a significant decrease in the strength-mechanical properties as well as the material characteristics of the rubberized concrete after 250 cycles of alternating freezing and thawing.

The final durability test involved assessing the resistance to water and defrosting chemicals using cube samples with an edge length of 100 mm, as shown in bar chart in Figure 7.

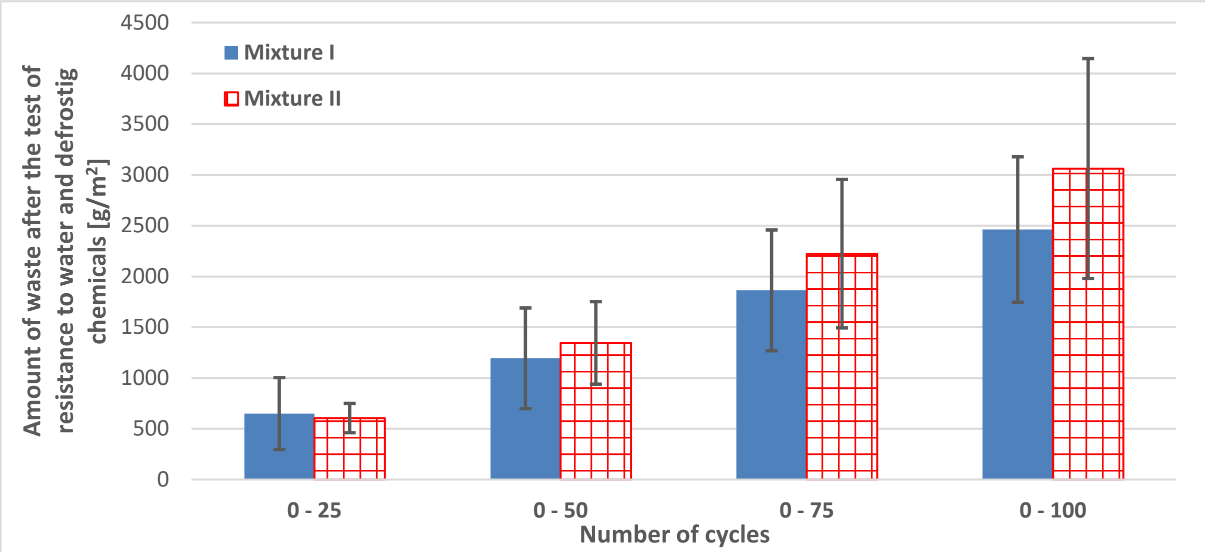

Fig. 7: The waste results of both mixtures after the test of resistance to water and defrosting chemicals [20].

In accordance with standard ČSN 73 1326 [23], mixture I exhibited a waste measurement of 2462.3 g/m^2 and achieved a disturbance level of 3 (disturbed) after 100 cycles. Meanwhile, mixture II, with a waste measurement of 3062 g/m^2, reached a disturbance level of 4 (strongly disturbed) after the same number of cycles.

As an additional experiment, the microstructure at the interface between rubber granules and cement paste was observed and scanning of the surface of the samples after testing of resistance to water and defrosting chemicals as a possibility of evaluating the level of disturbance. Within the experimental program an Optical Microscope KEYENCE with elementary analyser based on laser-induced breakdown spectroscopy (LIBS) was used for this purpose. LIBS systems produce high-temperature plasma by using a short laser pulse to ablate a small portion of the sample. As this material forms a hot plasma, it emits light during the cooling process. This light, characterized by discrete spectral lines, is analysed spectroscopically to determine the elements in the sample [25]. Figure 8 shows the used optical microscope with LIBS system and for comparison Scanning Electron Microscope JEOL.

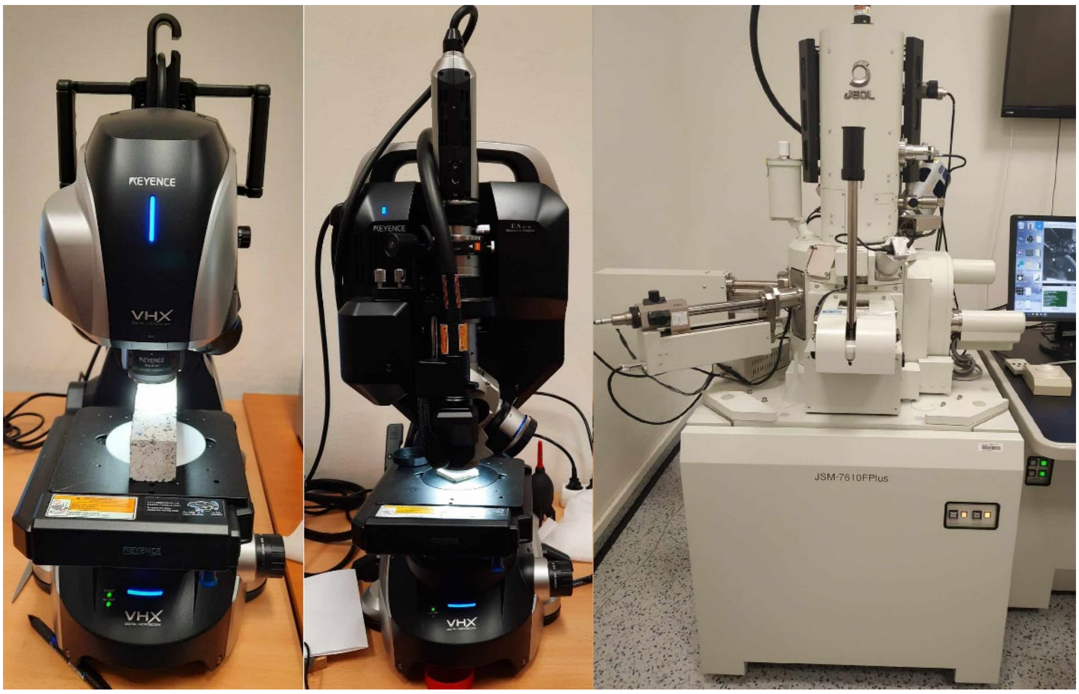

Fig. 8: From left to right: Optical Microscope KEYENCE, LIBS system and Scanning Electron Microscope JEOL

The following Figures 9, 10, 11 and 12 show the surfaces of the samples before and after 50 cycles of the test of resistance to water and defrosting chemicals. The photo documentation of the surfaces on the left side represents the samples before the test, the photo documentation on the right side represents the samples after the water resistance test.

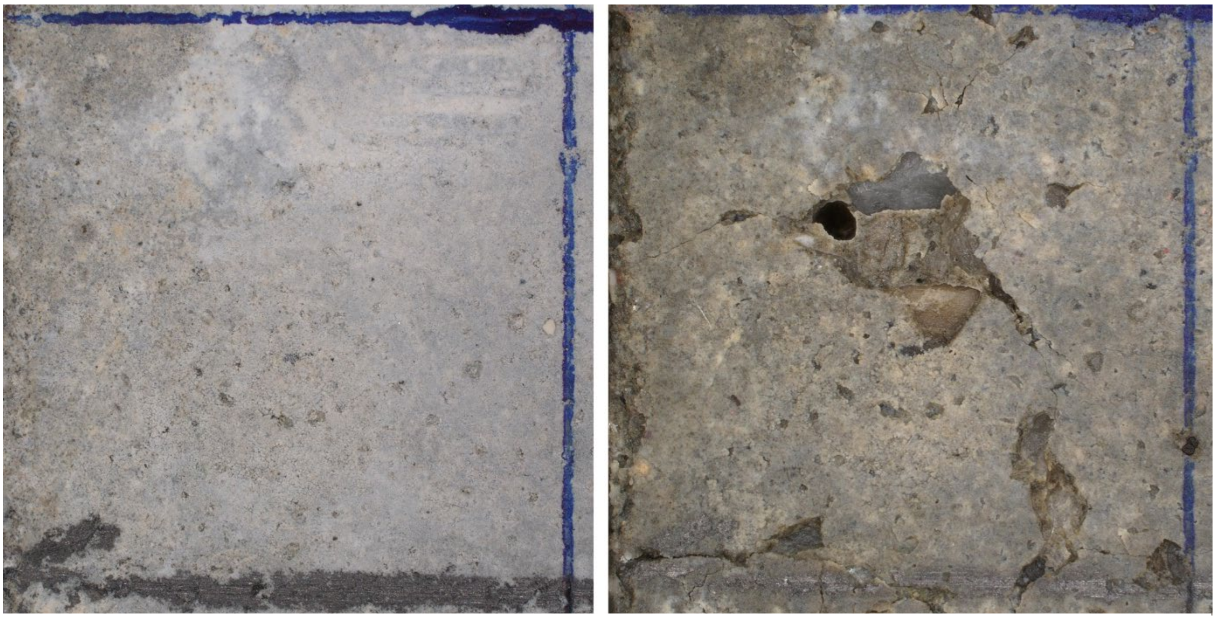

Fig. 9: Left: intact area of mixture I sample (underside of formwork); right: same area of the sample after the test of resistance to water and defrosting chemicals [20].

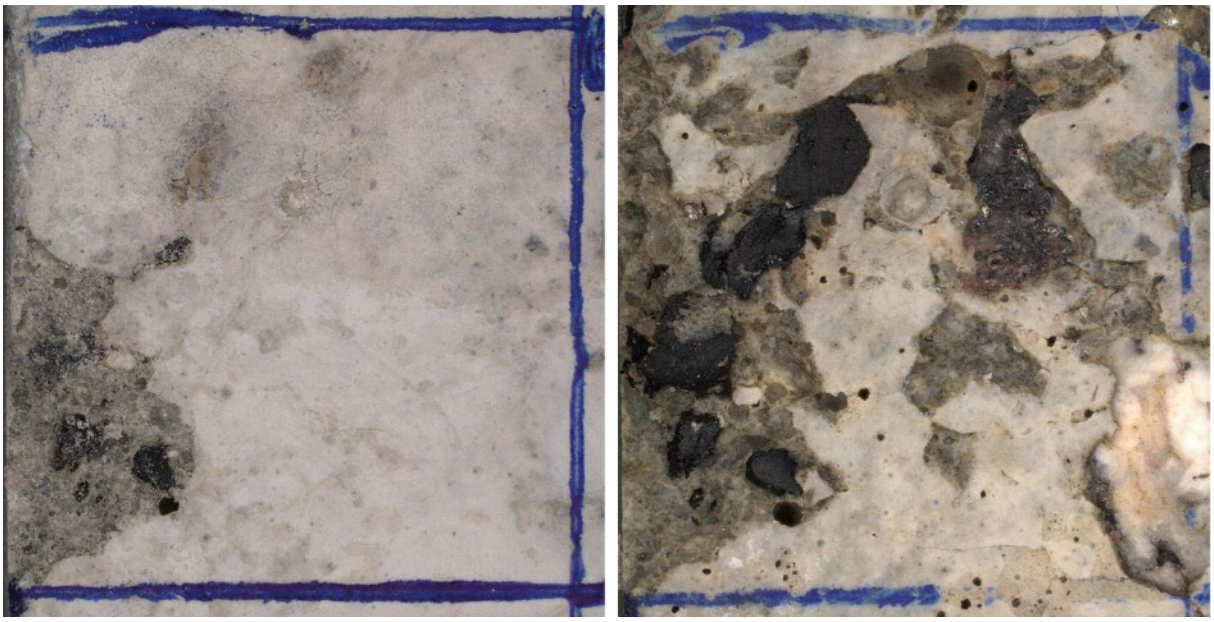

Fig. 10: Left: intact area of mixture I sample (upper exposed side of the formwork); right: same area of the sample after the test of resistance to water and defrosting chemicals [20].

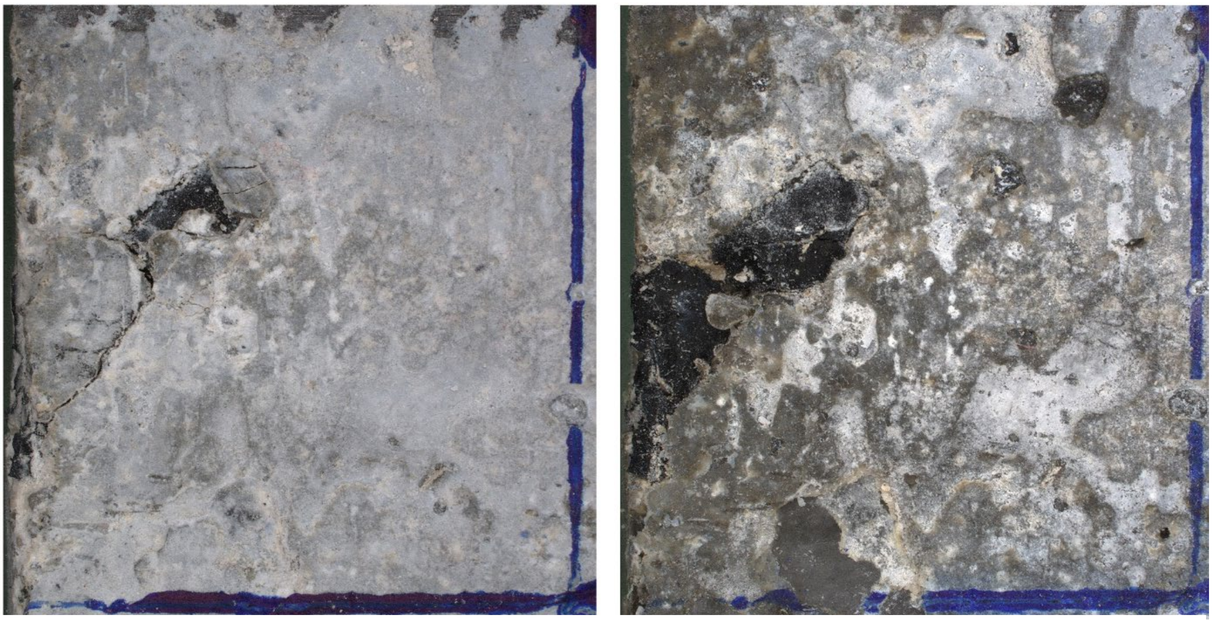

Fig. 11: Left: intact area of mixture II sample (underside of the formwork); right: same area of the sample after the test of resistance to water and defrosting chemicals [20].

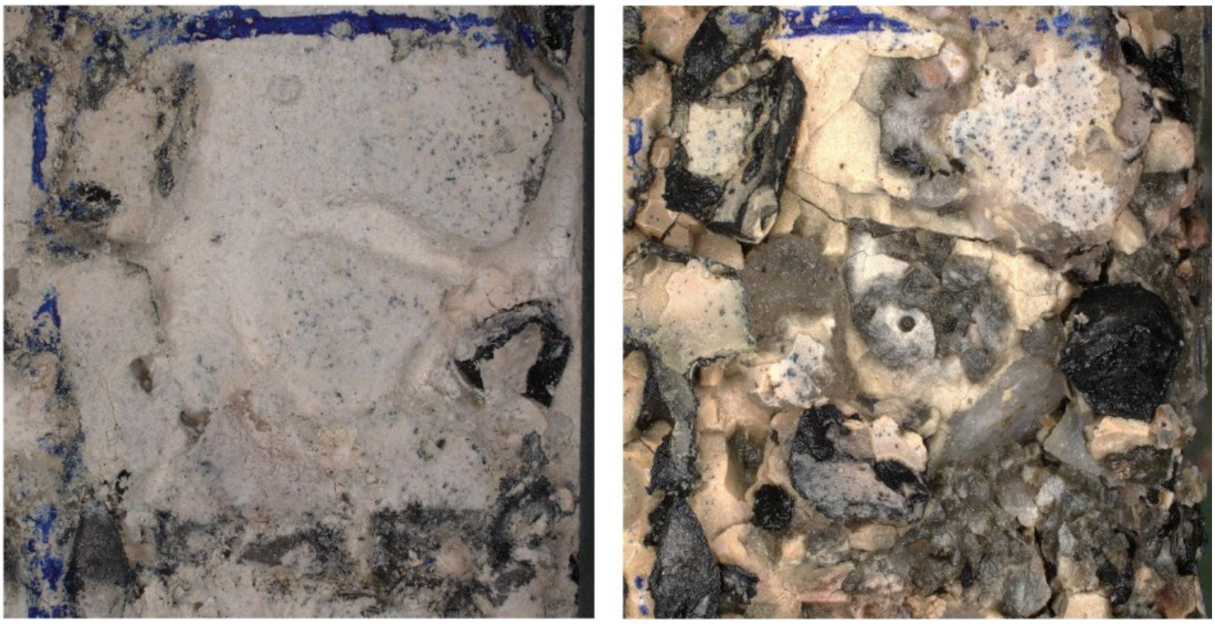

Fig. 12: Left: intact area of mixture II sample (upper exposed side of the formwork); right: same area of the sample after the test of resistance to water and defrosting chemicals [20].

It is clear at a glance from the Figures 9-12 that more degradation has occurred on the samples subjected to the test of resistance to water and defrosting chemicals with the top (filling) side of the sample (see Figures 10 a 12) of both mixtures. For a more comprehensive illustration of the surface change after the durability test, 3D reliefs of the observed areas of the samples are shown in the following Figures 13 and 14.

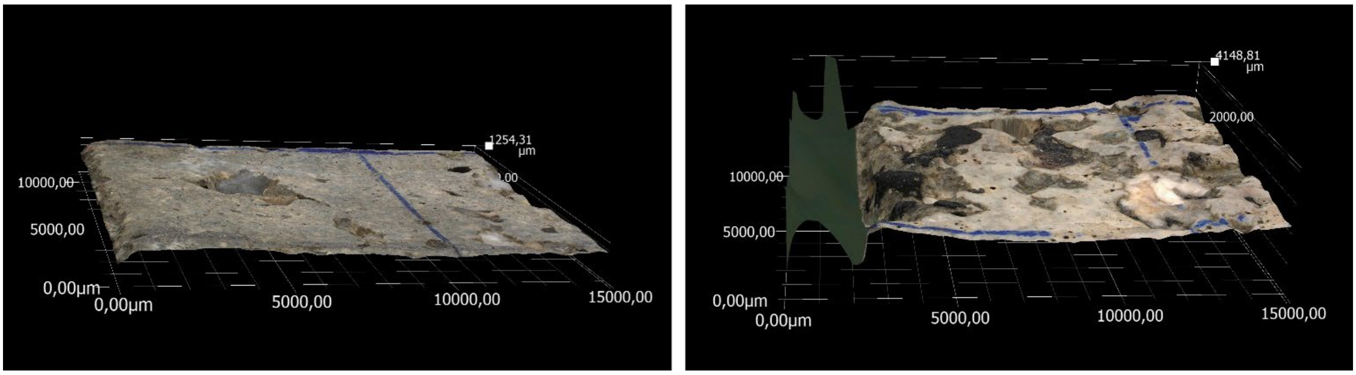

Fig. 13: Left: relief of sample of Mix. I, exposed to the durability test with the underside; right: sample of the same mixture exposed to the mentioned durability test with the upper side [20].

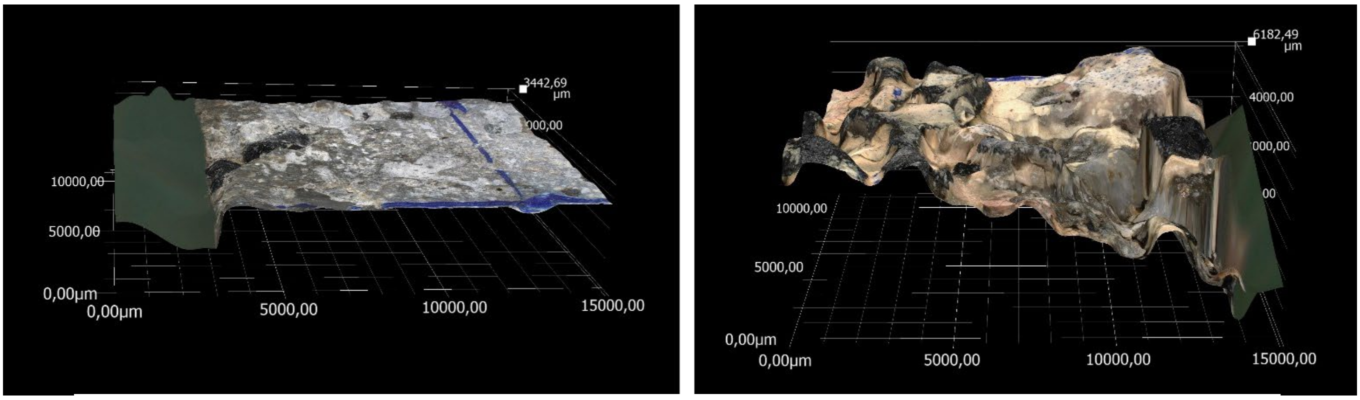

Fig. 14: Left: relief of sample of Mix. II, exposed to the durability test with the underside; right: sample of the same mixture exposed to the mentioned durability test with the upper side [20].

The change in surface areas of the individual samples after the test of resistance to water and defrosting chemicals are expressed in Table 4.

Table 4: Comparison of the surface areas of the samples before and after the test of resistance to water and defrosting chemicals [20].

Sample	Surface area before the test [µm^2]	Surface area after the test [µm^2]	Percentage increase in surface area
Mix. I, underside	101845846.65	103421568.89	1.55%
Mix. I, upper side	104532202.14	113244899.87	8.33%
Mix. II, underside	104877202.51	105314275.07	0.42%
Mix. II, upper side	119656766.83	146508914.96	22.44%

From the Figures 13 and 14 and from the results from Table 4, it can be concluded that sufficient compaction and proper surface treatment have a significant effect on the resistance to water and defrosting chemicals of a given composite. In the case of mixes 1 and 2, there is negligible change in the surface of the specimens exposed through the underside (from the formwork). On the other hand, for both mixtures subjected to the above test through the top side, there is a significant change in surface area, with an increase of 8,33 % for Mixture I and up to 22,44 % for Mixture II, which, together with the disturbed area profile in Figure 14, indicates a significant penetration of water and chemicals into the sample structure.

The interface between rubber granules and cement paste was also observed using an optical microscope. The Figures 15 and 16 show this interface.

Fig. 15: The interface between rubber granules and cementitious matrix [20],

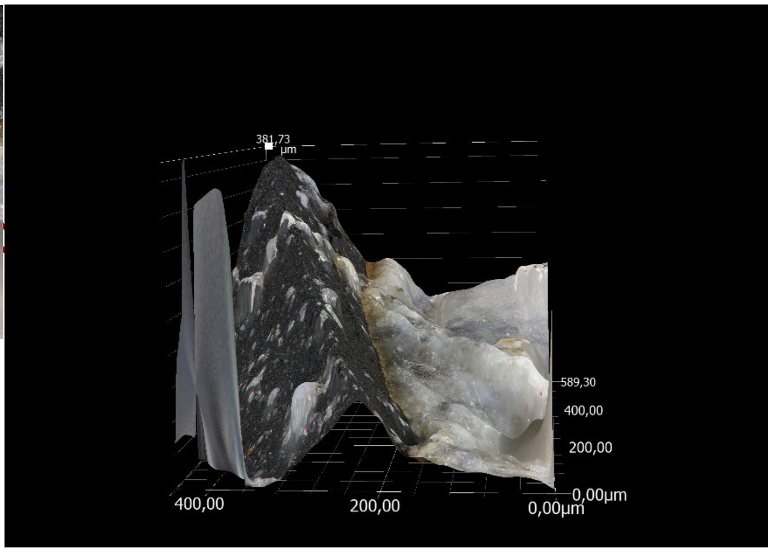

Fig. 16: 3D view of the interface between rubber granules and cementitious matrix [20].

Figures 15 and 16 show the morphology of beam sample with dimension 40x40x160 mm, disturbed by bending. During the three-point bending test, cracks occurred in areas of the cement matrix, the rubber granulate remained intact and separated only sporadically from the concrete structure, indicating favourable adhesion between the rubber recyclate and the cement matrix.

Basic elemental analysis of the main components of the composite was also determined using an optical microscope with LIBS system. Figure 17 shows the chemical analysis of the rubber granulate, Figure 18 of the cement matrix and Figure 19 of the granite aggregate. However, it is essential to note that this is only a basic elemental analysis, which generally identifies the given material. For a better understanding of the chemistry of concrete, an analysis of the mineralogical composition focusing on the identification and development of hydration products is necessary.

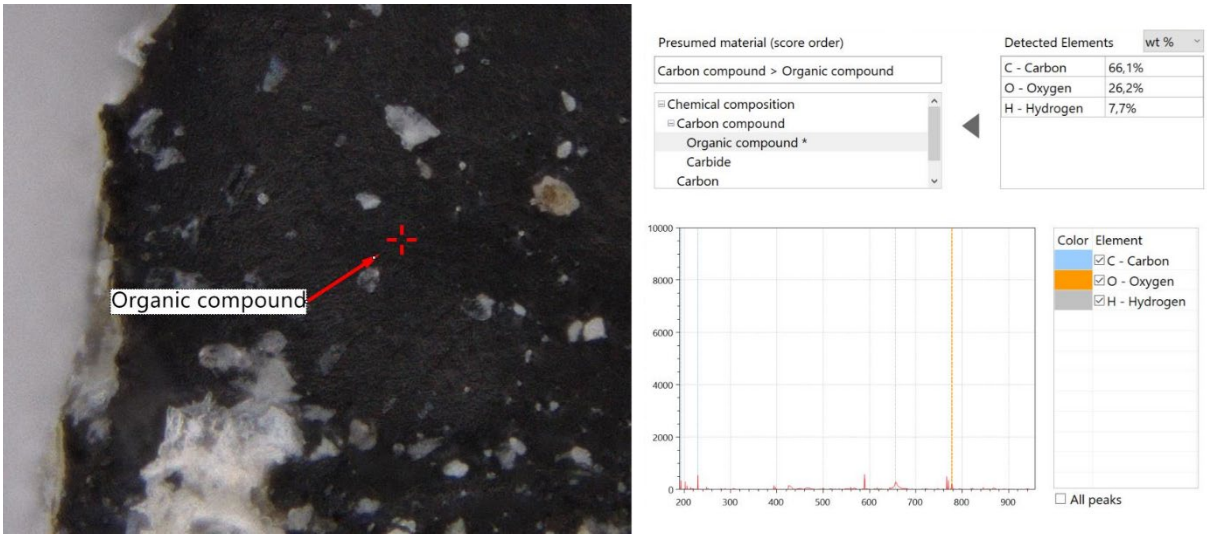

Fig. 17: Results of elemental analysis of the used rubber recyclate [20].

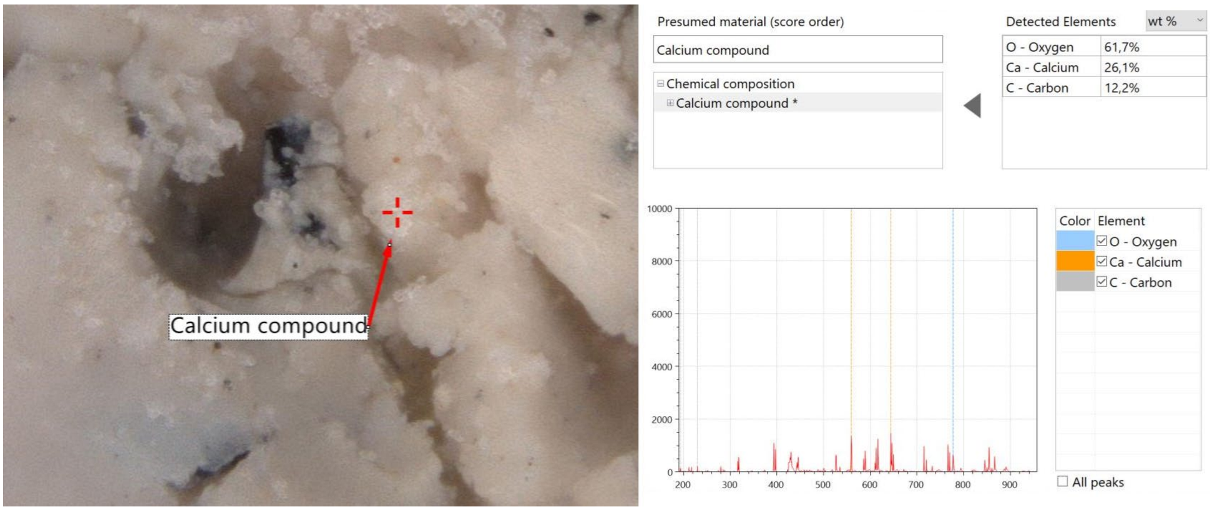

Fig. 18: Results of elemental analysis of the cement matrix [20].

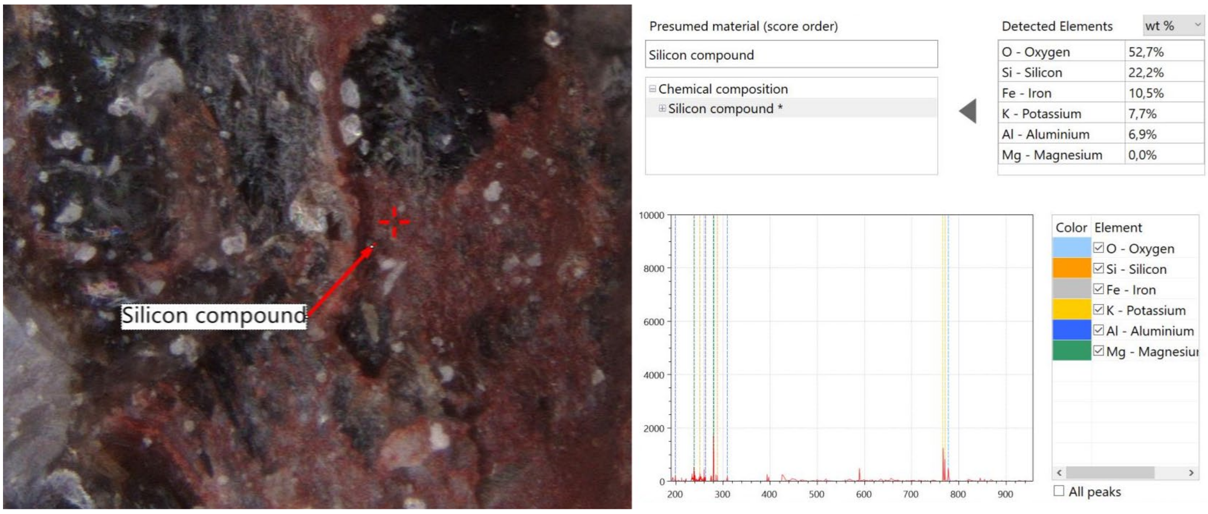

Fig. 19: Results of elemental analysis of the used granite aggregate [20].

3 Discussion and Conclusion

The study explored the potential utilization of recycled rubber from old tires as a partial replacement for aggregate in concrete, observing the strength-mechanical and durable properties of two mixtures.

Increasing the rubber granulate content from 70 kg to 140 kg per cubic meter led to a reduction in 28-day compressive strength by approximately 42% and tensile splitting strength by approximately 35%. Initially, both mixtures exhibited similar development in compressive and flexural strength over the first 7 days, with mixture II showing higher strength characteristics. However, from the 14th day onwards, mixture I demonstrated a more significant increase in strength. It is evident that the presence of the rubber granulate increases the strength characteristics in the initial stages of curing of the composite.

In durability tests, mixture I displayed better performance, achieving frost resistance for 125 cycles, while mixture II was deemed not frost-resistant. Frost resistance significantly decreased for both mixtures, with mixture I, flexural strength dropping from 5.2 MPa to 1.9 MPa between the 125th and 250th cycle, and mixture II from 3.9 MPa to 1.9 MPa after 50 cycles. The dynamic modulus of elasticity was also affected by frost resistance, decreasing notably for both mixtures.

Additionally, the resistance to water and defrosting chemicals revealed a higher level of disturbance (4) for mixture II after 100 cycles, where the waste increased by 50% compared to mixture I. As it turned out, the rubber granulate was adversely affected by low temperatures, which resulted in an overall deterioration of the composite's frost resistance. In addition, its presence significantly reduced the resistance to penetration of water and defrosting chemicals.

Alongside to the test of the resistance to water and defrosting chemicals, the possibility of evaluating this test by changing the area exposed to a given degrading agent after 50 cycles has been experimentally tested. This requires further testing to determine the correlation coefficient between the change in surface area and the amount of waste after individual cycles.

A basic elemental analysis was also conducted to identify the input raw materials, along with a detailed examination of the interface between the rubber granules and the cementitious matrix of the sample subjected to the flexural strength test. This insight, coupled with a preliminary visual inspection of the fracture surface of the sample after the flexural strength test, revealed favourable adhesion between the rubber granulate and the cementitious matrix, which could be attributed to the roughness of the rubber granule surface and excellent adhesive properties of the rubber material and cementitious matrix. However, further investigation and specification of this phenomenon is necessary.

Overall, mixture I outperformed in various aspects, except for compressive and flexural strength in the initial 7 days, making mixture II suitable for structural elements requiring rapid initial strength. This could be due to the higher stiffness and strength of the rubber recyclate than the initial strength of the cement matrix. In case of future research, there is a possibility to verify this by using a quick-setting cement or by replacing the filler with rubber granulate in HPC. Further, increasing the rubber granulate content had a less adverse effect on flexural strength compared to compressive strength, suggesting potential use in reinforced concrete. Nonetheless, the durability of the resulting composite was compromised with higher rubber granulate content. While incorporating granulate aligns with the principles of circular economy, its effectiveness depends on meeting structural element requirements.

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