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Study of a different carbonation intensity on the internal surface of cracks and inside concretes with dense aggregate and concrete recyclate

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Abstract. The paper focuses on the experimental physical - chemical analysis of different carbonation intensity by TG/DTG and FTIR method on the selected surfaces of concretes with aggregate and with concrete recyclate, with and without polymer and steel fibres. By means of thermal analysis and infrared spectrometry, carbonation intensity from the specific amount of calcium carbonate and portlandite was tested on specimens scratched from the internal surface of cracks and on specimens from the inside of concrete. It seems that bending macrocracks do not significantly accelerate the process of carbonation in the depth of concrete. But they bring various corrosion agents to concrete. As regards the evaluation of carbonation intensity of different concrete samples, fibre concretes with concrete recyclate are more suitable for structural purposes than fibre concretes with dense aggregate.

1. Introduction

The best way of preventing occurrence of cracks in a system that is bound by hardened cement, is to use fibres. Cracks in concrete can occur due to tensile stress caused by plastic shrinkage, drying shrinkage or autogenous shrinkage. At the same time, fibres can also improve other properties, such as bending or impact strength, or abrasion resistance. Fibre concretes are modern building materials with sufficiently proven mechanical and deformation properties. Long polymer fibres purposefully improve the mechanical properties of the concrete composite. However, the presence of fibres in the concrete composite can also have a negative impact [1]. The dispersed fibres are able to improve the low tensile strength of concrete, however, at the cost of exceeding the critical shear stress of the fibre - cement contact. Poor adhesion of fibres to the cement matrix, the exceeding of the shear stress or corrosion can lead, according to some authors, to an increase in permeability and subsequently to a decrease in the durability of the cement composite. On the other hand, however, some experts believe, that the addition of both long and short fibres to concrete increases its durability on the grounds of preventing the occurrence of microcracks at all stages of concrete curing.

The aim of the paper is to assess the influence of macrocracks on the intensity of carbonation in the loaded structures of fibre concretes. Under macrocracks we understand cracks with a width of 0.1 mm and more, which are visible to the naked eye. The paper seeks to answer the question whether macrocracks created when loading fibre concrete with steel or polymer fibres, accelerate the process of carbonation in the depth of concrete or not. The paper compares fibre concrete with dense natural aggregate with fibre concrete with its coarse aggregate fraction substituted with concrete recyclate.



2. Experimental setup

The paper focuses on examining the carbonation of fibre concretes with dense natural aggregate and fibre concretes with the coarse aggregate fraction substituted with concrete recycle. It also studies combinations of steel and polymer fibres added to concretes.

Beams containing flexural cracks from the previous flexural strength testing were kept in a carbonation chamber with a CO₂ concentration of 98% for a period of 9 months.

For the purposes of the experiment, three specimens were chosen and labelled B2/2 (1% of polymer fibres), C2/1 (1% of polymer fibres + concrete recycle), and DA3/2 (0.15% of steel fibers). The three specimens were tested for carbonation intensity by means of thermal analysis and infrared spectrometry with Fourier transformation. Material for the analysis of the studied specimens was taken by scraping the surface layer A) from the surfaces of cracks in the middle depth of sectional area of beams dimensions 100x50x400 mm, B) from the surfaces split off from the inside of concrete (distance from the beam surface is min. 25 mm).

2.1. Process of carbonation

Carbonation of concrete is a chemical process caused by a reaction of carbon dioxide with the components of hardened cement paste in concrete. Portlandite present in the hardened cement paste reacts with carbon dioxide to form calcium carbonate (note: in the form of calcite or aragonite).

The equation (1) of the process of concrete carbonation:



With a decreasing amount of portlandite, also the pH of the covering layer of concrete is decreased. In case the pH decreases below 9, the so-called depassivation of the reinforcement occurs, and its corrosion begins. Carbonation damage to concrete can be detected by means of a 1% solution of phenolphthalein. In case the pH decreases below 9.4, the typical violet colouration does not occur. That part of concrete which does not change colour after the solution application, shows signs of carbonation [2].

2.2. Concrete formulas

The tested specimens were made according to the following three formulas: 1st formula marked “B” – dense aggregate with 1% of PP fibres, 2nd formula marked “C” – concrete recycle of 0-16 mm with 1% of PP fibres, 3rd formula marked “DA” – dense aggregate with 0.15% of steel fibres. See table 1.

Table 1. Concrete formulas.

Labelling of concrete	Amount of cement	Aggregate			Water-cement ratio	Plasticizer	PP + steel fibers
	[kg/m ³]	0-4 mm	4-8 mm	8-16 mm	-	[%] of m _c	[kg/m ³]
B	490	890	100	745	0.36	1	9.1
C (recyclate)	490	890	100	633 (recyclate)	0.43	1	9.1
DA	490	890	100	745	0.36	1	11.7 (0.15%) steel

The type of cement used was CEM II/B-S 32.5R. The concretes were prepared from the natural extracted dense aggregate with a fraction of 0-4 mm from the Bratčice sand pit, from the natural

extracted dense aggregate with a fraction of 4-8 mm from Tovačov, and from the natural crushed dense aggregate with a fraction of 8-16 mm from Olbramovice. The concrete made from concrete recyclate was prepared by substituting the coarse fraction of the natural aggregate of 8-16 mm from Olbramovice with the raw concrete recyclate with a fraction of 0-16 mm by Dufonev s.r.o.

All the three formulas were plastified by means of the CHRYSOPLAST 760 plasticizer, the type of polymer fibres used was FORTA FERRO with a length of 50 mm, the type of steel fibres used was Dramix with a length of 60 mm.

2.3. Thermal analysis (TG/DTG)

Thermal analysis involves experimental methods by means of which we analyze changes in the composition and properties of the analyzed substances or mixtures under thermal load.

The basic method of thermal analysis is thermogravimetry (TG), which studies changes in mass over time depending on the temperature. From the resulting thermogravimetric curve, it is possible to read temperatures of mass changes.

A method derived from this is the derivative thermogravimetry (DTG), in which the resulting thermogravimetric curve shows the dependence of the mass change rate on temperature. This method is suitable for a differentiation of consecutive effects [3,4].

2.4. Infrared spectrometry with Fourier transformation (FTIR)

Infrared spectrometry is an analytical technique used for identification and structural characterization of organic compounds and determination of inorganic matters. The principle of infrared spectrometry is the absorption of infrared radiation in passing through the specimen. The infrared radiation includes the area of wavenumbers of $12,500 - 10 \text{ cm}^{-1}$.

Passage of radiation through a substance causes rotational-vibrational transitions of molecules. In case a change in the given rotational or vibrational states is connected with a change in the dipole moment, the radiation characteristic of a specific bond in the molecule is absorbed. Each organic compound has its own region, i.e., no two different organic compounds have the same spectral expression.

The most frequently used band of the infrared spectrometry is the so-called central region given by the wavelength range of $4000 - 400 \text{ cm}^{-1}$. In the range of $4000 - 2500 \text{ cm}^{-1}$, there are valence vibrations of hydrogen bonds. The loose O-H bond is absorbed at wavelengths of about 3600 cm^{-1} [5,6,7].

3. Results and discussion

3.1. Results of TG/DTG

Table 2. Amount of calcium carbonate and portlandite measured by thermal analysis of specimens (A – internal surface of the crack, B – the inside of concrete).

Sample	Mass % c / hm. %	
	Ca(OH) ₂ (350-550°C)	Ca(CO) ₃ (550-850°C)
B2/2 A	6.3	19.4
B2/2 B	5.3	15.8
C2/1 A	-	21.5
C2/1 B	-	18.5
DA3/2 A	-	22.4
DA3/2 B	-	18.7

Specimens were measured by a STA504 instrument (TA Instruments) in the temperature range of $50 - 1050^\circ\text{C}$. The analysis was performed with 15 – 20 mg of a specimen spread into ceramic crucibles,

the heating was performed in the N_2 atmosphere with a heating rate of 20°C per 1 minute. Figure 1 shows the sample plot of the thermal analysis with graphs of TG, DTG and DSC of the internal surface of the crack of specimen B2/2 (A). Table 2 shows the representation of portlandite and calcite in the individual specimens.

The measured values of all specimens show a higher amount of calcium carbonate on the surface of the crack (A) than inside concrete (B). The higher amount of calcium carbonate indicates a higher extent of concrete carbonation in the area of the crack.

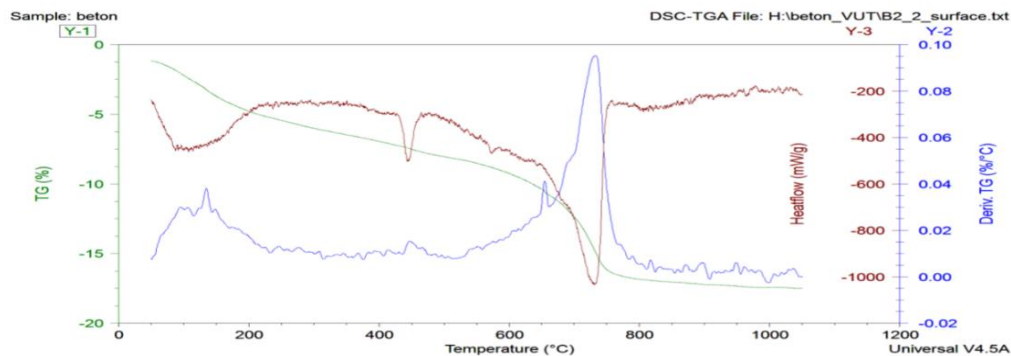


Figure 1. Specimen B2/2 – Plot of the thermal analysis of the internal surface of the crack (A).

3.2. Results of FTIR

Infrared spectrometry allows for measuring specimens of all physical states. One of the basic solid state measuring techniques uses tablets of potassium bromide, which transmits a wide region of the infrared spectrum. A small amount (1 – 10 mg) of the crushed specimen is homogenized with 300 – 400 mg of KBr. Subsequently, it is pressed into a tablet, giving rise to the so-called glassy modification of KBr, in which the loss of the intensity of the passing infrared radiation caused by reflection is small [9].

Using infrared spectrometry with Fourier transformation, we determined presence of portlandite in the specimens. Always two specimens from each formula were compared: specimen (A) from the crack surface, specimen (B) from the inside. 1 mg of the spread specimen was homogenized with 150 mg of KBr, and the tablet prepared from it was measured on the neighbouring iZ10 FTIR module of the Nicolet iN10 microscope (Thermo Scientific) by means of a transmission technique in the spectrum range of 4000 to 400 cm^{-1} , using resolution of 4 cm^{-1} . Attention was given especially to the band of 3643 cm^{-1} , which corresponds to the vibration of OH bond in portlandite. A comparison of spectra of the measured specimens is shown in the following charts in figure 2-4.

In specimen B2/2, a higher intensity of band 3643 cm^{-1} was measured in the part of specimen taken from the inside (B) than in the area of the crack surface (A). In specimen C2/1, portlandite occurs only inside (B) and in a very low concentration. Specimen DA3/2 was fully carbonated in both the monitored part (A and B), and calcium carbonate does not occur only in the form of calcite, but also in the form of aragonite, especially on the surface of the crack.

Values obtained from the thermal analysis show a higher amount of calcium carbonate present in the fibre concrete in the area of the surface of cracks than in the inside of concrete, in all the three specimens tested.

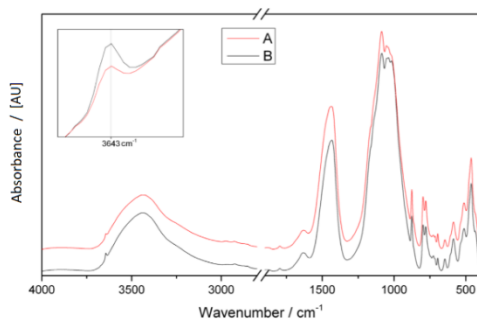


Figure 2a. Specimen B2/2: Infrared spectrum (A – internal surface of a crack, B – the inside of concrete).

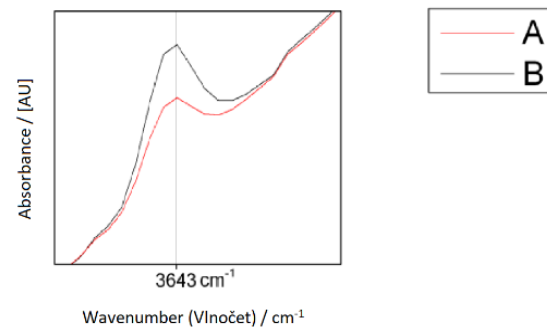


Figure 2b. Detail of a chart of a wavenumber of the vibration of the OH bond in portlandite. Specimen B2/2: Infrared spectrum (A – internal surface of a crack, B – the inside of concrete).

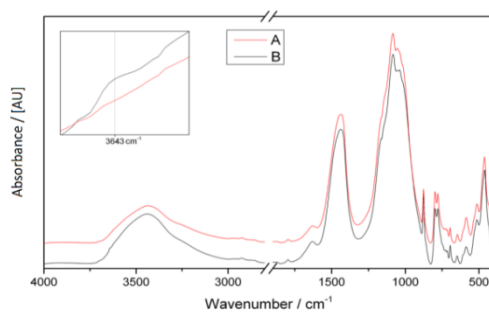


Figure 3. Specimen C2/1: Infrared spectrum (A – internal surface of a crack, B – the inside of concrete).

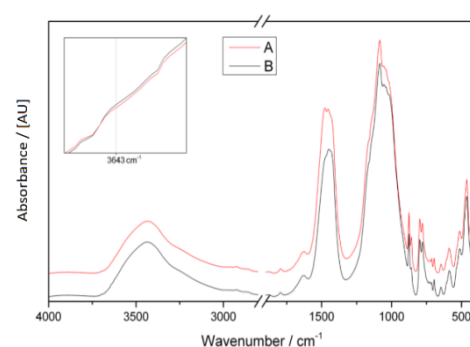


Figure 4. Specimen DA3/2: Infrared spectrum (A – internal surface of a crack, B – the inside of concrete).

The measurement with Fourier transformation infrared spectrometry (FTIR) showed that specimen B2/2 with an addition of 1 % of PP fibres had the best result in terms of the lowest extent of carbonation. Specimen C2/1 with concrete recylcate and 1% addition of PP fibres showed a better result than specimen DA 3/2 with coarse aggregate and an addition of 0.15% of steel fibres. The specimen of fibre concrete with steel fibres (DA 3/2) was fully carbonated on the crack surface as well as in the inside of concrete. This higher susceptibility to carbonation is probably caused by an occurrence of a high number of microcracks during the hydration shrinkage of concrete. Considering the different elasticity moduli of polymer and steel fibers, tensile microcracks can occur in the solid steel-fiber skeleton during shrinkage of concrete at the initial stage of setting [8]. In this case, steel fibres seem to be less advantageous for their low plasticity than the polymer ones. Fibre concrete made from concrete recylcate with an addition of polymer fibres resisted depth carbonation in the experiment better than steel fibre reinforced concrete with dense aggregate, in which carbonation occurred with a greater extent and intensity.

4. Conclusions

On the basis of the TG/DTG and FTIR tests performed on fibre concrete specimens of three formulas, it is possible to make the following conclusions:

a) Bending macrocracks (more than 0.1 mm wide) do not significantly accelerate the process of carbonation in the depth of concrete due to low ventilation of air with a natural content of CO₂. On the

internal surface of the crack, carbonation occurs with approximately the same intensity as on the free surface of the concrete of an open structure.

b) If durability of fibre concretes is evaluated narrowly, only from their susceptibility to carbonation, then fibre concretes with concrete recyclate are more suitable for structural purposes than fibre concretes with dense aggregate. The bearer of a higher durability of fibre concretes with concrete recyclate, however, is not the concrete recyclate itself, but the higher plasticity of polymer fibres in comparison with the steel ones.

c) Although macrocracks themselves do not significantly accelerate the process of carbonation inside concrete, they bring CO₂, water and other substances to concrete and in the case of crossing steel reinforcement and a crack, accelerated corrosion occurs.

Acknowledgement

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