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New sampling method to improve the reliability of FTIR analysis for Self-Compacting Concrete

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HIGHLIGHTS

- Drilling method for Fourier Infrared Spectroscopy involves the risk of sampling aggregate.
- New method of scrapping the concrete sample avoids the risk of sampling aggregate.
- Results of FTIR indicate a faster carbonation for scrapping than for drilling method.
- Carbonation process in crushed concrete sample is faster than cubic concrete sample.

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ABSTRACT

Carbon dioxide uptake during concrete carbonation has become an emerging problem, as more attention is paid to sustainable development. Therefore an advanced test methods has been adopted to measure process of carbonation. A Fourier Transformation Infrared Spectroscopy analysis was applied to define a progress of carbonation in Self-Compacting Concrete. Two types of samples were studied – a typical cubic samples and crushed concrete. Samples were kept in carbonation chamber for 56, 112 and 168 days in controlled conditions. A new sampling method is presented in the paper. Applying the proposed procedure a deeper progress of carbonation process is revealed, when compared with a standard drilling technique. Achieved findings were verified with Termogravimetric Analysis/Differential Thermal Analysis.

1. Introduction

As more attention is paid to the sustainable development, what was stressed by many authors [1–7], a new tools to its estimation has been developed. In case of the concrete technology, one of the most important issue is a carbon footprint. According to ISO/TS 14067:2013 [8] definition, a carbon footprint is a sum of the greenhouse gas emissions and consumptions in a product, expressed as CO_2 equivalents (CO_2e) based on a life cycle assessment (LCA). In comparative analysis of construction materials and techniques, an environmental impact is often expressed as a total sum of CO_2e emission in construction lifetime. Therefore a huge attention is paid to calculate such a balance with sufficient accuracy. According to ISO/TS 14067:2013 [8], to obtain a total result, a cradle – to – grave scenario should be considered. As Naik [9] emphasized, only the total environmental impact during life cycle of concrete should be considered. In such an approach, the emission of CO_2 to atmosphere and the consumption of carbon dioxide due to i.e. carbonation should be calculated. Considering end-of-life stage approach Kuroda and Kikuchi [10] calculated that the total CO_2 balance is lower about 5.5% when comparing with the traditional approach. Therefore the analysis of CO_2 amounts absorbed in carbonation process, has become an emerging problem, as stressed Pacheco Torgal et al. [11].

Self-Compacting Concrete (SCC) is one of the most innovative concrete technology and has been applied in many countries [12]. Although cost of SCC mixture is higher than regular concrete, unique properties of Self-Compacting Concrete make this technology the only solution to some technical problems. One of the best example of SCC application is the Burj Khalifa (Dubai), the world's highest building. Approximately 12500 m3 of SCC was pumped in the building foundation [13]. However, when facing requirements of sustainable development we have to stress that SCC technology is no longer perceived as attractive

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because it demands a larger amount of cement. It results with the increase of carbon footprint.

Process of concrete carbonation concerns the exposed concrete surfaces. However, in the end-of-life stage, when concrete is demolished to a concrete rubble, process accelerates, as an exposed surface increases, what was pointed out by Lagerblad [14]. Therefore a number of studies focus on the issue of concrete CO_2 uptake taking this stage of construction's life to account. As Woyciechowski [15] noticed to determine a carbonation process test methods can be divided into; laboratory methods and in-situ chemical indicators. A chemical indicators are one of the simplest methods to determine the depth of the carbonation in concrete. Among a number of such pH indicators, the phenolphthalein solution seems to be the most popular. Although this method is a very convenient method, as it is applied on the fresh concrete fracture taken in the construction site. Unfortunately it does not indicate exactly the advance of carbonation process, see [16]. The more accurate are the laboratory techniques. To indicate the advance of carbonation process in concrete the following methods are usually used: thermogravimetric analysis (TGA), X-ray diffraction analytical method (XRD), gammadensimetry and Fourier Transformation Infrared Spectroscopy (FTIR), which were discussed in [15–19]. Another method to determine the process of carbonation is SEM analysis, which was applied in number of studies [20–22]. Although this method provides the accurate image of carbonation products, it refers to a limited local region. The FTIR method has been chosen in our further study.

In the FTIR method, analysis of the individual spectra allows to identify a chemical compound in the sample. When infrared light passes through the sample, each functional group resonates in its characteristic absorption frequencies of the spectra. Obtained data represents the fingerprint of the bound between the molecules. The spectrum is usually divided into regions: near-, mid- and far-infrared. As Ylmén and Jäglid [23] stressed, the carbonate ion has four characteristic vibration modes in the mid-IR region. There are three common polymorphs of anhydrous $CaCO_3$: calcite, aragonite and vaterite. The most stable one is calcite and it is usually investigated in FTIR analysis. Such transformation is represented by the wave number between 1410 and 1510 cm^{-1}. Even for simple compounds containing one functional group, the spectrum consists of multiple absorption bands. In case of the concrete, the analysis is difficult due to complex nature of the material and spectra are usually not that evident as pointed out by Lee et al. [24].

A FTIR spectrum is essentially a graph of infrared light absorbance or transmittance as a function of frequency or wavelength. Typical units of frequency used in FTIR spectra are wave numbers, with the symbol cm^{-1}.

There are variety ways of sample preparation. In case of concrete, the most common is drilling or sawing a sample form examined depth, as it has been reported in number of scientists [16,17]. Gathered powder is mixed with potassium bromide (KBr), which is transparent to the infrared light and pressed in a mechanical press to a transparent pastilles. The ratio of investigated material to KBr described in a number of study [16,17] was 1:10 and material was collected from layers of 1.5–5 mm depth. Such a collecting method in case of concrete is very uncertain. This is due to fact, that approximately 70% of concrete volume is an aggregate and quantity of investigated material is vestigial. Therefore there is no guarantee, that collected material represents the cement matrix where a carbonation process occurs and does not contain the aggregates. Thus a new sampling method is needed.

In the study, a FTIR analysis was applied to determine the CO_2 uptake in two types of SCC, which were kept in carbonation chamber for 56, 112 and 168 days. A novel method of material sampling was described and the obtained results were compared with the traditional drilling method. Achieved FITR data were correlated with the phenolphthalein indicator.

Next a thermal analysis TGA/DTA (Thermogravimetric Analysis/ Differential Thermal Analysis) was carried out for a chosen sample to verify the developed method. TGA technique bases on the relationship of mass change in specific temperature. The temperature at which certain reaction occurs is characteristic for the individual chemical compounds. In TGA analysis of concrete Poole and Sims [18] have described characteristic scopes of mass loss in specific temperature range. The principle of the DTA method is to measure the temperature difference between the sample and the reference material. Both materials are heated simultaneously under linear temperature program. The difference is recorded as a function of temperature. Application of both test methods allows for accurate interpretation of achieved results. The TGA/DTA analysis was applied to compare difference of $CaCO_3$ collected with different method and verify the traditional drilling with the proposed scrapping method of sampling.

2. Materials and experimental procedure

We investigated SCC of C30/37 class. Recipe was developed previously by Skanska Research and Innovation Centre. In the study 0.15 m^3 of mixture was prepared. Concrete mixture composition was calculated per one cubic meter and is presented in Table 1. Obtained properties of concrete mixture are presented in Table 2.

Chemical properties of used cement were tested according to EN 196-2:2013 [25]. Obtained results are presented in Table 3. Loss of ignition was measured in temperature 950 ± 25 °C.

Two types of sample were casted in standard forms – 150 × 150 × 150 mm cubic and cylinders with 300 mm height and 150 mm diameter. Samples were cured in water, according to EN 13295:2004 [26], for 28 days. Next, samples were kept in standardized laboratory conditions of 21 ± 2 °C and 60 ± 10% RH, for another 90 days. After curing time, samples were divided into two types – a standard 150 × 150 × 150 mm cubic and crushed concrete obtained from cylinders. Cylinders were crushed with a hammer drill to concrete rubble which was reflecting a concrete after demolition. Size of crushed concrete ranges from 8 to 31.5 mm, see Fig. 1.

Further, samples were placed in the carbonation chamber for 56, 112 and 168 days under controlled conditions of 1% concentration of CO_2, 21 °C temperature and 60% RH. Reference samples were kept in laboratory condition, next to the carbonation chamber, according EN 13295:2004 [26]. The carbonation chamber had a perforated trays, that were spinning around. It ensured uniform conditions for all the samples placed in the chamber.

Carbonation progress in the samples kept in the carbonation chamber was measured according to EN 13295:2004 [26] using 10% phenolphthalein in water solution. Samples kept for 56 days were additionally tested with a Rainbow Test indicator. As the Rainbow Test indicates only the pH range between 9 and 13, for further study (112 and 168 days periods) we use only phenolphthalein solution.

Next, each sample was tested with a Fourier Transformation Infrared Spectroscopy. In the study the Nicolet iS10 FTIR Spectrometer was used. During the measurement the temperature and relative humidity were kept in the constant level: 20 °C and 60% respectively. Tested material in FTIR analysis was collected from the following layers: 0–5 mm, 5–10 mm, 10–15 mm, 15–20 mm, 20–25 mm.

Regarding the disadvantages of the drilling sampling method, a new method has been developed and verified. This method assumed collecting tested material form the breakthrough of the sample with scraping tested material with a hard graver from the surface. Then, the powder was manually sifted to separate fine sand grains. Such approach provided a possibility of removing the coarse aggregate and collecting material from the cement matrix where carbonation process occurs. To compare the above described method with a drilling one, cubic samples exposed 56 days for CO_2 corrosion were investigated using both methods. In drilling method the table drill, with a 1 mm manual adjustment of drilling depth was used.

Table 1
Self-Compacting Concrete (SCC) mixture.

Concrete mixture	kg/m^3
Cement CEM II/B-M 32,5R	340
Fly-ash	190
Sand 0–2 mm	652
Gravel 2–16 mm	985
Admixtures superplasticizer	3.15
Water	160