Water Repellent Treatment of Building Materials
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Quality Control by FT-IR-Spectroscopy

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Abstract

The penetration of water-soluble aggressive salts into concrete can be avoided by the surface treatment with water repellent agents. For the performance of a water repellent treatment the penetration depth is crucial. One powerful tool for quality control is FT-IR-spectroscopy. In this contribution the results of a quantitative determination of water repellent agents in concrete are presented. Calibration curves have been set-up and a linear regression analysis for this curves have been carried out.

Keywords: FT-IR-spectroscopy, quality control, calibration curve

1 Introduction

Deep penetration of aqueous aggressive salt solutions into concrete and frost damage can be avoided by surface treatment with water repellent agents. The performance of a water repellent treatment is strongly determined by the penetration depth and the content of active substances in the covercrete. In practice penetration profiles must be checked by a rigorous quality control. One powerful tool for quality control is the FT-IR-spectroscopy. Details of this technique are described in [1, 2]. Friese and Protz combined FT-IR-spectroscopy with microscopy [3]. With this technique, called FT-IR-microscopy, they studied the penetration of water repellent agents into bricks. Franke, Pinsler and Oly studied penetration profiles of of water repellent agents which were used for the treatment of different types of natural stones, bricks and mortar with FT-IR-microscopy as well [4].They found that FT-IR-microscopy can be used for the semi-quantitative determination of water-repellent agents in different building materials. Effects, like evaporation of solvents or also small values for the penetration depth can be observed in this way.

The applicability and precision of an analytical technique in chemistry, however, can only be justified by statistical analyses of the numerical results only. In order to check the performance of FT-IR-spectroscopy for the determination of water repellent agents in the covercrete different test series have been carried out. The main aim of this contribution is to investigate variability of test results and reliability of the method.

2 Quantitative FT-IR-spectroscopy

2.1 Fundamentals of FT-IR-spectroscopy

FT-IR-spectroscopy is a useful tool to determine organic compounds in complex matrices such as concrete qualitatively and quantitatively. In normal concrete the content of organic compounds containing CH2/CH3-groups is limited. In FT-IR-spectra the peaks in the area from 3100 cm-1 to 2800 cm-1 can be attributed to CH2/CH3- groups which are parts of water repellent agents based on silicon-organic compounds. Therefore, the typical peaks of the CH2/CH3 groups can be used to determine the content of the active substance, i.e silicon resin, in concrete quantitatively.

2.2 Qualititative analyses using the KBr-technique

For FT-IR-spectroscopy the samples are prepared by using the KBr-technique. Therefore, a fixed amount of potassium bromide is mixed with a small amount of ground and dried concrete powder in a mortar. For making a transparent sample a part of this mixture is compressed under vaccum with 250 bar in a specially designed mould. With this platelets the FT-IR spectra are measured.

Usually, the KBr-technique is not the best method for the quantitative determination because of non reproducable scattering of the IR radiation on the surface of the transparent sample. Furthermore, an inhomogenous distribution of the powder in the sample and a variation of the thickness of the transparent sample can be not be totally avoided. These problems in preparation technique leads to a relative standard deviation of approximately 10% [5]. Nevertheless, for the investigation of concrete powder the KBr-technique is the only suitable method for sample preparation and in this case the precision is satisfactory.

2.3 Quantitative evaluation of the FT-IR-spectra

According to Lambert-Beer´s law the value of absorption A of a typical peak of the investigated substance is proportional to the amount c of the compound. The thickness of the sample d and the Lambert-Beer coefficient a must be determined experimentally.

$$A = a \times c \times d \qquad (1)$$

Practically, the intensity or the area of typical peaks in FT-IR-spectra must be determined. With this values the absorption A can be calculated. A widely used method to calculate the peak area is the baseline-method which is especially suitable for complex matrices like concrete [6]. A schematic sketch of the baseline method is shown in fig.1. First, for the application of the baseline method the boundary wavenumbers must be defined (wavenumber x and wavenumber y). For this purpose normally the two minima left und right of the peak which should be integrated are used. Second, the integration of the peak area is carried out. It is necessary for this method that the same boundary wavenumbers of the investigated product are used for all analyses. For each product the boundary wavenumbers must be defined again.

The plot of the calculated area of the CH2/CH3-peaks versus the depth represents the distribution of silicon resin in covercrete semi-quantitatively. For the quantitative determination of the content a calibration curve must be

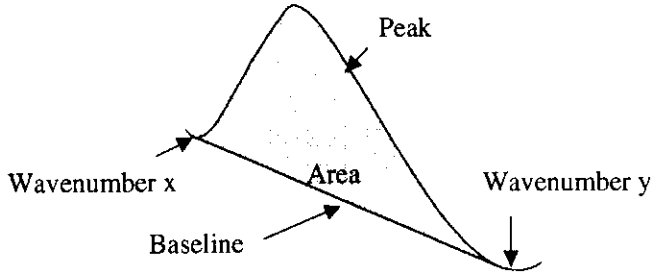

Figure 1: Sketch of the baseline-method3. Experiments

set up using the peak area of standard samples with a well known concentra-tion of the investigated silicon resin. With these data a plot of the content of active substance versus peak area can be establishe. This calibration curve is the base for the calculation of the unknown content of active substance [5].

For the interpretation of the results statistical methods must be used to characterize the precision of the measured contents.

3 Experiments

3.1 Preparation of the concrete specimens

For the experimental investigations test specimens have been prepared according to SIA 162 [7]. The composition of the investigated concrete is as follows. The maximum aggregate size is 16 mm. The content of Portland Cement CEM I 42.5 is for all mixes 350 kg/m3. The water/cement ratio is 0.35, 0.40, 0.45 and 0.50, respectively. After demoulding the concrete specimens are stored at 20 °C and 70% R.H. for 28 days. From these concrete elements cores with a diameter of 75 mm have been drilled. Afterward from these cores discs with a thickness of 2 mm are cut using a specially designed saw. The discs are dried at 105 °C until constant weight.

3.2 Treatment of the discs with different water repellent agents

For the impregnation of the specimens two types of water repellent agents, i.e an undiluted silane (100%) and a aqueous silane emulsion have been chosen. After drying the mass and capillary porosity of the discs were determined gravimetrically. In the next step the discs have been immersed in the water repellent agent for 24 hours. Then the saturated discs are weighted

again to determine the mass of the adsorbed water repellent agent. For polymerization the discs are stored in a closed box for 14 days in which the atmosphere is saturated with the undiluted silane or water. Under these conditions the loss of water repellent agents can be nearly neglected. Untreated reference specimens have been stored under the same conditions but not in silane saturated air.

3.3 Preparation of the specimens for FT-IR-spectroscopy

First, the discs are ground to very fine concrete powder using a specially designed mill. The duration of grinding is 30 sec, the mill works with 1400 rpm. For the FT-IR-spectroscopy samples are prepared according to the KBr-technique. Therefore, 1000 mg potassium bromide is mixed with 40 mg of the ground and dried sample in a mortar for seven minutes. For making platelets 250 mg of this mixture are compressed under vacuum with 250 bar in a mould. With these samples FT-IR-spectra in the range of 2800 cm-1 to 3100 cm-1 are taken with 20 scans. The FR-IR-spectra are evaluated by the baseline-method, which is implemented in the FT-IR-spectrometer software.

4 Results and Discussion

4.1 Calibration curve

After immersion of the discs into the water repellent agents the uptake has been determined gravimetrically. These values have been used to calculate the content of the water repellent agent in the platelets which is tested by FR-IR-spectroscopy. In fig. 2 and fig. 3 the peak area as a function of the content of adsorbed water repellent agents is shown. With these data a linear regression analyses has been carried out. The calculated functions are shown in fig. 2 and fig. 3 by straight lines.

The values for the linear regression coefficient are not excellent for a chemist but suitable for the application in practice. This calibration curve is only admissible for the investigated commercial product.

4.2 Untreated concrete

For the determination of the background 12 platelets of untreated concrete have been analysed by the same method as the treated specimens. With the

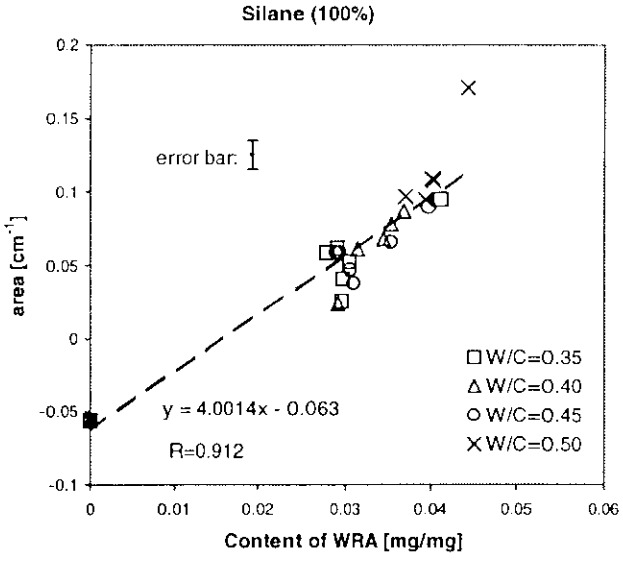

Figure 2: Silane (100%) - Peak area versus content of water repellent agent (WRA)

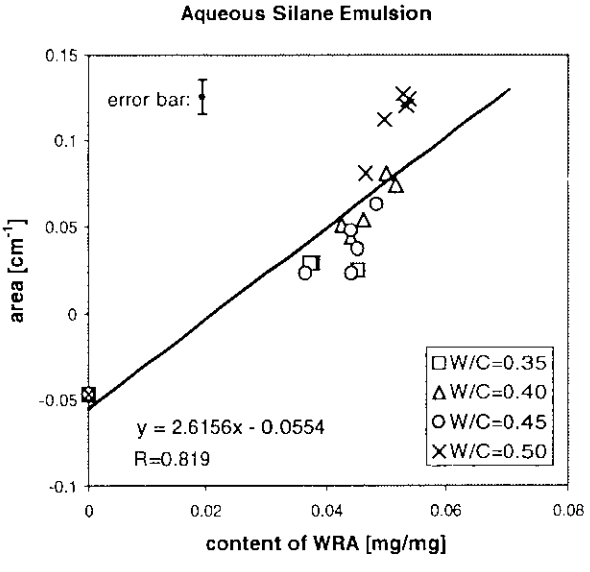

Figure 3: Aqueous silane emulsion - Peak area versus content of water repellent agent (WRA)

determined areas the average value x and the standard deviation s have been calculated. The values are given in table 1. Values given for the aqueous emulsion have a limited significance only. In Ref. [8] it is shown that this type of agent hardly penetrates into concrete. At first glance it is surprising that for the area negative values have been calculated. This can be explained in the following way. For the baseline-method it is essential to use the same range of wavenumber for each specimen, treated or untreated. In the case of untreated specimen the values for the area under the baseline can be higher than the values for the area over the baseline. Therefore, the integration of the total area leads to a negative value for the total area (see fig. 3). If the limiting wave numbers are better choesen, i. e. a narrow band width is selected, negaitve values can be avoided.

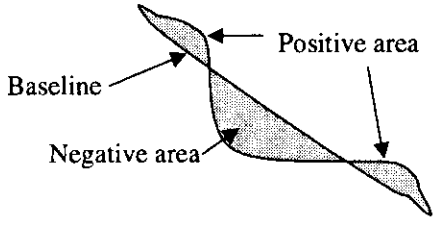

Figure 4: Explanation of a negative value for the total area

Table 1:

	Aqueous silane emulsion	Silane (100%)
Range of wavenumber [cm^{-1}]	2974 - 2948	2975 - 2946
Average value x	- 0.047	- 0.056
Standard deviation σ	0.017	0.014

5 Conclusions

From the presented results the following conclusions can be drawn:

- The penetration profile of water repellent agents can be determined by means of FT-IR-spectroscopy quantitatively.

- FT-IR-spectroscopy allows us to determine the presence of water repellent agents in porous building materials.
- It is possible to establish calibration curves for different water repellent agents
- By means of a calibration curve quantitative penetration profiles can be obtained.
- Thus method may serve as a solid basis for a rigorous quality control. It is possible to check in a practical application if the required penetration depth has been reached.

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