ASSESSMENT OF WATER REPELLENT TREATMENTS BY THE APPLICATION OF FT-IR-SPECTROSCOPY

A. Gerdes,
Laboratory for Building Materials
ETH Zürich, Switzerland

ABSTRACT

Water repellent treatments on concrete structures are often carried out with silicon-organic compounds. The uptake and the penetration depth of the active substance are decisive for the performance of an impregnation. So far the penetration depth is determined in most cases visually. A new method has been developed to determine the distribution of the water repellent agents in the covercrete quantitatively by using FT-IR-spectroscopy. The fundamentals of this new method and the application on different types of concrete which were preconditioned in different ways and impregnated with different types of water repellent agents are presented. It will be shown that this method is a useful tool for the assessment of an impregnation in practice and for the performance of newly developed water repellent agents.

1 INTRODUCTION

Durability of concrete is determined among other factors by its complex interactions with aggressive chemical substances. The extent of damage is related to the transport processes of the chemical substances into concrete /1/. The uptake of water or aggressive salt solutions by concrete can be drastically reduced by the application of a water repellent agent. Normally silicon-organic compounds dissolved in water or organic solvents are used. The solution is applied on the surfaoe of concrete. After the transport of the solution into the concrete by capillary suction a chemical reaction takes place, which leads to the formation of the active substance, a water-repellent silicon resin. The silicon resin forms a thin layer on the inner surface of the capillary pores. As a result the capillary water uptake is reduced if not practically prevented.

The uptake of the active substance and the penetration depth are decisive for the performance and long-term behaviour of the impregnation. The penetration depth depends mainly on factors such as suction capacity of the building material, type of the water repellent agent, type of the organic solvent and application conditions /2/. So far the penetration depth is often determined by visual methods described in detail in /3,4/. With these methods the action of water repellents can be estimated qualitatively. But in this way the determination of the content and distribution of the active substance in concrete is not possible. The influence of the type of water repellent agent or of the properties of the concrete on the performance of an impregnation cannot be characterized by this methods. Therefore, we developed in the Institute for Building Materials, ETH Zürich, a new method to determine the content of the active substance, the silicon resin, in the covercrete qualitatively and quantitatively by using FT-IR-spectroscopy. The fundamentals of this new method and the application on different types of concrete which were preconditioned in different ways and impregnated with different types of water repellent agents are presented. It will be shown that this method is a useful tool for the assessment of an impregnation in practice and for the performance of newly developed water repellent agents.

2 FT-IR SPECTROSCOPY

The FT-IR-spectroscopy is a well-known and powerful tool in chemistry for chemical analysis and determination of the structure of molecules and so-called functional groups which build up the molecules. The FT-IR-spectroscopy based on the fact that molecular vibrations occur in the infrared region of the electromagnetic spectrum and that functional groups have characterisic absorption frequencies. To take a IR-spectrum IR-light in the range from 2.5 μm to 16 μm passes through a sample. By resonance the functional groups absorb the IR-light and the intensity of the IR-light decreased. The ratio of the intensity measured before (I_0) and after (I) passing the sample means the absorption A. The IR-spectrum is usually obtained as a chart showing absorption peaks, plotted against wavelength or wavenumber which is the reciprocal of the wavelength. A typical FT-IR-spectrum for silicon resin is shown in Fig. 1. The peaks in the area from 3100 cm^{-1} to 2800 cm^{1} can be attached to functional groups and can be used to identify silicon resins as trace compounds in complex matrices. In normal concrete the content of compounds containing CH_2/CH_3-groups is limited. Therefore, the typical peaks of the CH_2/CH_3-groups can be used to determine silicon resin in concrete qualitatively. But FT-IR-spectroscopy is also a useful tool to determine compounds in complex matrices like concrete quantitatively. According to Lambert-Beer' s law (1) the value of the 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 μ must be determined experimentally.

$$A = \mu \cdot c \cdot d \tag{1}$$

Practically, the area of typical peaks in a FT-IR-spectrum must be calculated. A often used method for this calculation is the baseline-method which is especially suitable for complex matrices like concrete. For quantitative analysis of silicon resin in concrete the typical peaks of the CH_2/CH_3-groups at 2960 cm^{-1} and 2930 cm^{1} are suitable. The plot of the calculated area of the

CH_2/CH_3-peaks versus the depth represent the distribution of the active substance silicon resin in covercrete quantitatively /5-6/.

3 EXPERIMENTS

3.1 PREPARATION AND CONDITIONING OF TEST SPECIMENS

For the experiments one concrete mix was used throughout. The concrete specimens are prepared with a water/cement ratio of 0.4. The content of OPC is 400 kg/m^3 and the maximum aggregate size is 8 mm. The dimensions of specimens have been chosen to be 360 x 120 x 120 mm^3. After preparation the specimens are stored at 20 °C and 70 % R.H. for 28 days. Afterwards the specimens are conditioned as shown in Table 1. The different water repellent agents are applied airless on the non-moulded surfaces of the conditioned specimens. The uptake of the water repellent agent are determined by weighing. From the impregnated specimens prisms with the dimensions of 100 x 100 x 120 mm^3 are cut, the area of the impregnated surface is 100 x 100 mm^2. The used water repellent agents and the values of uptake of the water repellent agents are also shown in Table 1.

3.2 ANALYSIS OF THE IMPREGNATED SPECIMENS BY FT-IR-SPECTROSCOPY

The specimens impregnated under laboratory conditions are prepared in the following way. Starting from the impregnated surface thin layers of 0.3 mm thickness have been removed. in the range up to 5 mm by using a specially designed milling tool. The collected very fine concrete powder is dried at 105 °C. For FT-IR-spectroscopy the samples are prepared by using the KBr-technique. Therefore, 1000 mg potassium bromide is mixed with 40 mg of the ground and dried concrete powder in a mortar. For making a transparent sample 250 mg of this mixture are compressed under vaccuum with 250 bar in a specially designed mould. With these samples FT-IR-spectra with 20-100 scans in the range of 2800 cm^{-1} to 3100 cm^{-1} are taken. The FT-IR-spectra are evaluated by the baseline-method, which is implemented in the FT-IR-spectrometer software.

3.3 PROFILES OF CAPILLARY SUCTION

In order to determine profiles of capillary suction the step-cutting method is applied. First, vertical surfaces of the specimens are coated with a epoxy resin. After the specimen are placed in contact with water, the capillary water uptake through the impregnated layer is measured for 48 hours. Then the first layer with a thickness of 0.3 mm which had been in contact with water, is cut off. For re-conditioning the specimens are dried at 50 °C for 24 hours and subsequently, stored at 20 °C and 60 % R.H. for 5 days.

TABLE 1 Preparation and conditioning of the specimens

Prism	1	2	3	4	5	6
Condi-tioning	14 d 55 %R.H. 20 ¡C	14 d 55 %R.H. 20 ¡C	14 d 55 %R.H. 20 ¡C	14 d 75 %R.H. 20 ¡C	3 d in oven at 60 ¡C	14 d 55 %R.H. 20 ¡C
Water repellent agent (WRA)	Master-Batch-Silane	Micro-emulsion	Siloxane in organic solvent	Siloxane in organic solvent	Siloxane in organic solvent	Siloxane in organic solvent
Second treatment	none	none	none	none	none	after 28 d
Consump-tion of WRA (g/m²)	153.8	266.7	168.3	113.8	451.9	160.3 132.2*
Active substance (g/m²)	15.4	21.3	10.5	7.11	28.2	10.01 8.51*

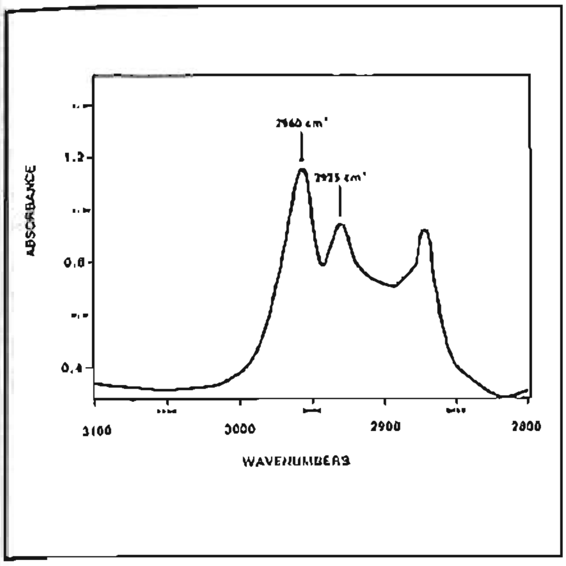
Afterwards the samples are immersed into water again. This procedure is repeated several times until the water uptake corresponds to the values of an untreated concrete.

4 RESULTS AND DISCUSSION

4.1 EVALUATION OF THE FT-IR-SPECTRA

The evaluation of a FT-IR-spectrum for silicon resin shows that the peaks at 2960 cm^{-1} and 2925 cm^{-1} can be attached to the CH_2/CH_3-groups. The FT-IR-spectra for untreated and impregnated concrete is shown in Fig. 2. In the spectrum of the impregnated concretethe peaks of the CH_2/CH_3-groups can also be observed. Therefore, this peaks can be used for the determination of the silicon resin content in covercrete because the intensity of the peaks are sufficient and no influence of organic contamination in the investigated concrete can be detected.

FIG. 1 FT-IR-SPECTRUM OF SILICON RESIN



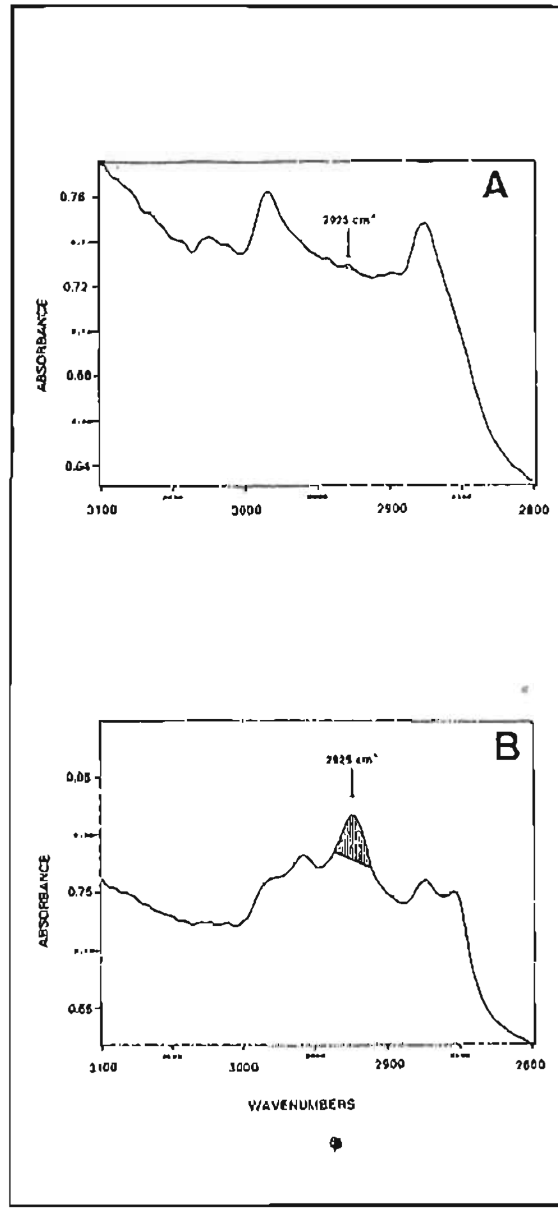


FIG.2 FT-IR-spectra of an untreated (A) and impregnated (B) concrete

4.2 CHARACTERISATION OF AN IMPREGNATION CARRIED OUT WITH DIFFERENT TYPES OF WATER REPELLENT AGENTS

In the first series the influence of the composition and structure of the water repellent agents on penetration depth was to be investigated. Therefore, the penetration profiles and the water suction profiles are determined for three types of water repellent agents. The penetration profiles as determined by FT-IR-spectroscopy are shown in Fig. 3. For the master-batch-silane the content of the active substance decreases significantly from the surface to a depth of 0.5 mm. In a depth of 0.75 mm no active substance can be detected anymore. In this range the peak area which is sligthly above zero can be attached to small amounts of impurities in concrete. For the micro-emulsion and the siloxane dissolved in an organic solvent the obtained curves are very similar. The active substance of these water repellent agents can be detected up to a depth of 0.75 mm. In Fig. 4 the water suction profiles for the three water repellent agents are shown. The water uptake for all water repellent agents is very similar to the depth of 0.25 mm. In the range from 0.25 to 0.50 mm the values for the water uptake increase and reach the value of an untreated concrete in a depth of 0.75 mm. It is obvious that the results of the penetration profiles correspond to the results of the water suction profiles. A decrease of the experimentally determined area corresponds to an increase of the water uptake. The results show clearly that the developed method using the FT-IR-spectroscopy can be applied to determine the penetration depth of different types of water repellent agents.

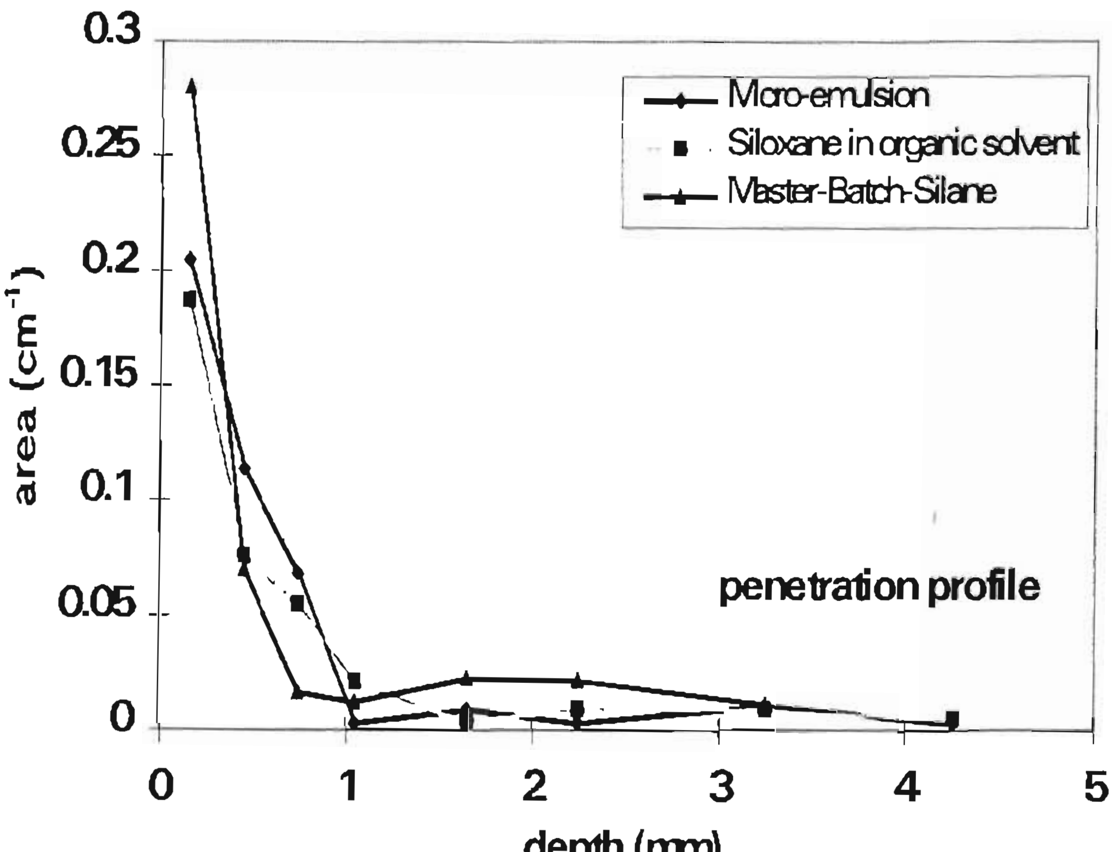


FIG 3 Penetration profiles of water repellent agents in concrete, impregnated with different types.

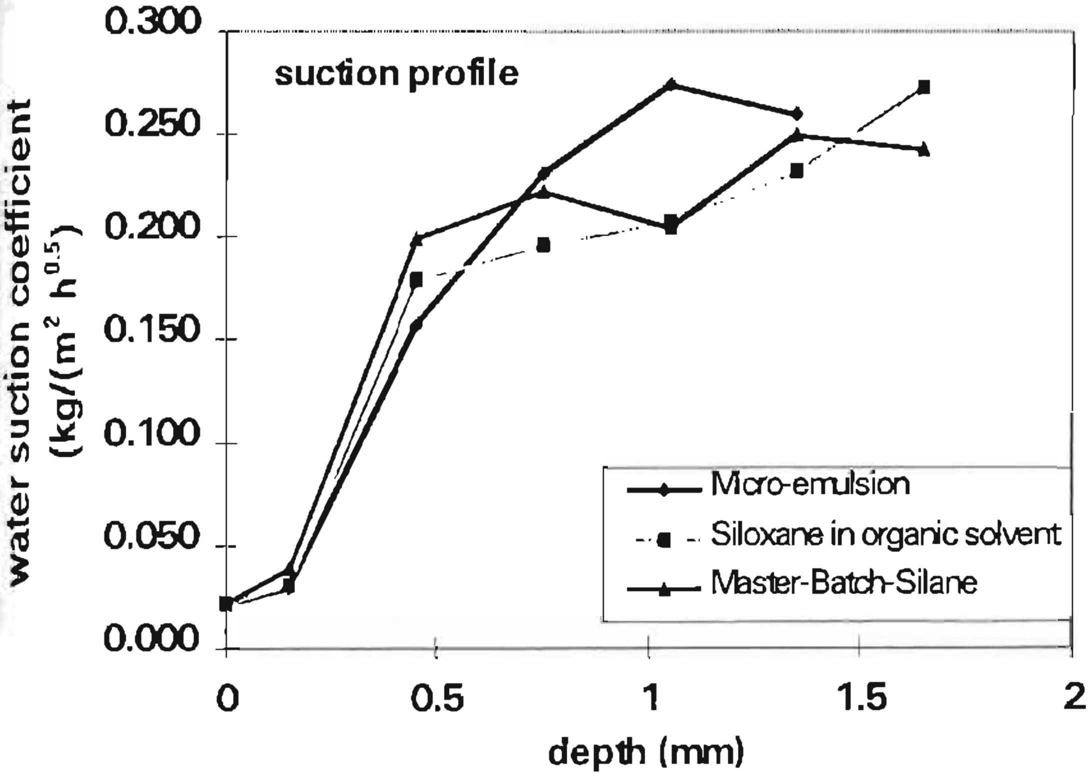


FIG 4 Water suction profiles for concrete, impregnated with different types of water repellent agents

4.2 CHARACTERISATION OF AN IMPREGNATION CARRIED OUT UNDER DIFFERENT CONDITIONS

In a second series the influence of the application conditions on penetration depth of siloxane dissolved in an anorganic solvent has been studied. The penetration profiles are shown in Fig. 5 and the suction profiles are shown in Fig. 6. The penetration profiles of the specimens impregnated under "wet" conditions (75% R.H.; 20 °C) show that the concentration of the active substance is very high near to the surface. In a depth higher than 0.5 mm no active substance can be detected. These results can be verified by the results shown in the suction profiles. From a depth of 0.5 mm the water uptake of these specimens correspond to values of untreated companion specimens. The significance of moisture content on the transport of water repellent agents into concrete can be confirmed by results obtained on specimens, pre-dried in an oven at 60 °C. The content of silicone resin in a depth of 1.5 mm is significantly higher as compared to specimens impregnated under "wet" or "lab" conditions (55% R.H. 20 °C). If after a period of 28 days the water repellent agent is applied a second time the best penetration depth can be observed. Up to a depth of 2 mm the amount of silicone resin is significantly higher as compared to the results described above. These results indicate that in practice an impregnation should be repeated if possible after a minimum delay of 14 days. The results of the penetration profiles can be verified by the results of the suction profiles. The water uptake of the specimens impregnated under different conditions correspond to the amount of silicon resin in the covercrete. A critical amount of silicon resin is necessary to achieve a full water repellent state.

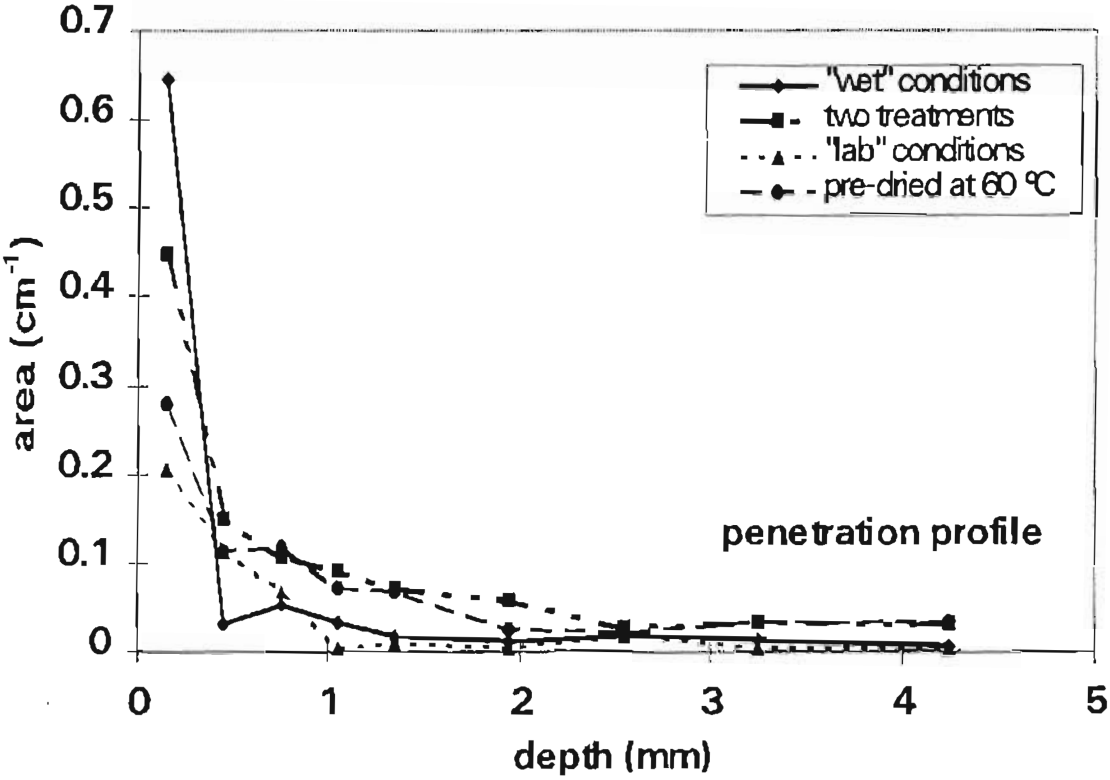


FIG 5 Penetration profiles for concrete impregnated under different application conditions

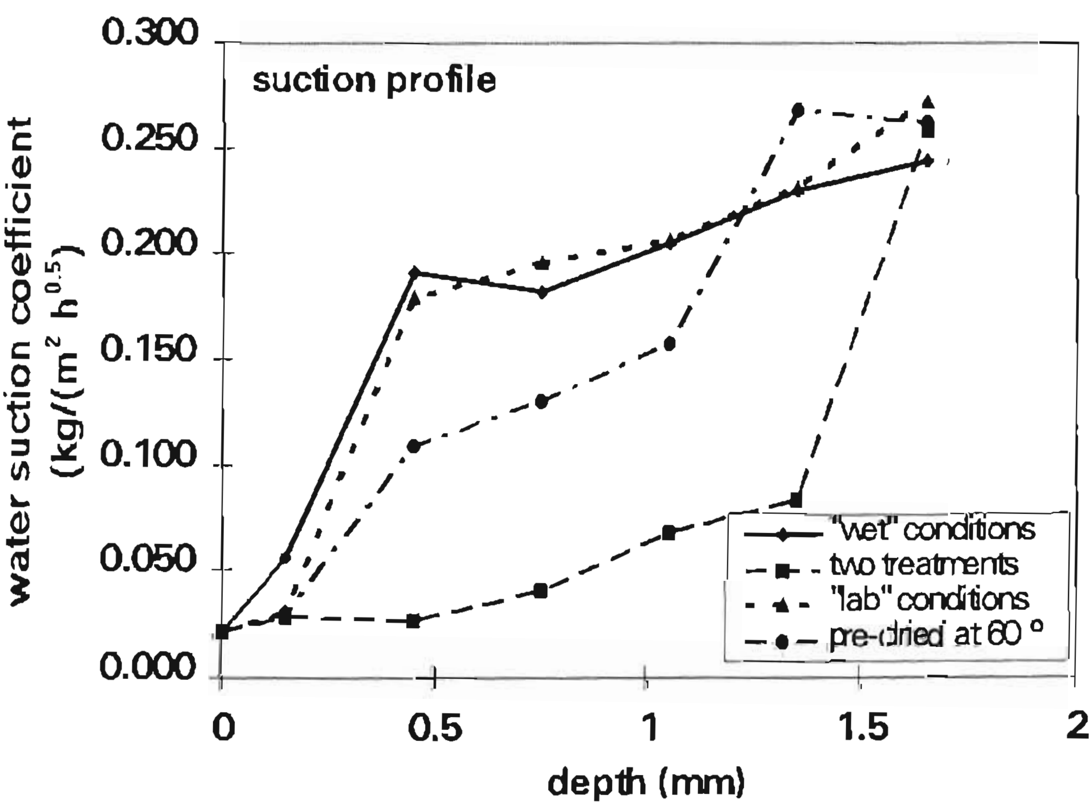


FIG 6 Suction profiles for concrete impregnated under different application conditions

5 CONCLUSIONS

- The penetration profile of water repellent agents can be determined quantitatively by means of FT-IR-spectroscopy in an efficient way.
- In practical applications the climatic conditions have an extreme influence on the quality, i.e. the depth of a surface impregnation.
- The combination of FT-IR-spectroscopy with suction profiles is a useful tool for the assessment of an impregnation in practice and for the performance of new developed water repellent agents.

6 REFERENCES

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