

Characteristics of the Surface near Water Repellent Layer

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SUMMARY: If the surface of concrete is impregnated with a water repellent compound such as a silane, a water repellent subsurface layer is built up and capillary absorption of water and aqueous salt solutions can be significantly reduced. The aim of this type of protective surface treatment is to increase service life of reinforced concrete structures by improving frost resistance and/or by reducing the rate of chloride penetration. Water vapor can still pass through the impregnated layer so the concrete is not sealed. This study presents experimental results that allow us to quantitatively describe characteristics of the water repellent surface near zone. The water distribution was determined by high precision neutron digital imaging. The results obtained helped explain earlier findings, which indicated that an efficient chloride barrier can only be established by deep impregnation. In a water repellent layer with a thickness below 6 mm enough water may remain on the pore walls that while reducing migration of chloride into the untreated pore space of the material may eventually be sufficient to induce corrosion to the concrete reinforcement. The study supports and justifies previous recommendations on the importance of an in-depth impregnation.

KEY-WORDS: Water repellent surface impregnation; water repellent layer; neutron digital imaging.

INTRODUCTION

Carbonation of concrete and chloride penetration into reinforced concrete structures are often the main factors, alone or in combination, for necessary early repair measures and a shortened service life of buildings and structures. Chloride is in most cases transported into the pore space of concrete when salt solutions, such as seawater or water contaminated with deicing salts, are absorbed by capillary action. Once the chloride is within the pore space of concrete it will slowly diffuse deeper into it finally reaching the steel reinforcement. When a critical concentration of chloride is reached in the region around the surface of the steel reinforcement, corrosion will start. However, if the surface of concrete is impregnated efficiently with a water repellent substance such as linseed oil or silane, capillary absorption of water or solutions may become negligible. However, in practice it has often been observed that chloride still penetrated into surface impregnated concrete, albeit with a reduced rate.

If only a very thin surface layer is impregnated, the water repellent effect may be obvious by the water pearling off the surface during visual inspection. But in practice, an aqueous salt solution is in contact with the concrete surface for longer periods, days or even weeks, if not permanently, as in the case of seawater. Experimental results have shown that a minimum thickness of the water repellent surface layer must be achieved to make sure that chloride cannot enter the pore space.

This study evaluated the thickness of different water repellent layers by neutron radiography. This allowed visualizing whether liquid water could still be present in thin water repellent layers, since if some liquid water is available, chloride ions may migrate through the layer via the still existing water bridges. In this case chloride penetration may be slowed down but it will not be prevented. Based on this premise, the minimum thickness of the water repellent layer required to act as a real chloride barrier could be established allowing the formulation of an effective chloride barrier. Furthermore, this would help in defining reliable recommendations for practical applications. The results described in this contribution may serve as a reliable basis for effective quality control.

EXPERIMENTAL

Preparation of mortar specimens

In order to study the characteristic properties of the water repellent zone beneath the treated surface in cement-based materials, mortar cubes with a 100-mm edge length were cast in steel forms. A 0.6 water-cement ratio was chosen for the mortar used in these test series and details of its composition are given in Table 1. Ordinary Portland cement type CEM I 42.5 and river sand with a maximum grain size of 2.5 mm were used. The fresh mortar was covered with a plastic sheet after compaction and stored in the workshop at a temperature of approximately 20°C for 24 hours. Then the steel forms were removed and the concrete cubes were stored in a humid room with $RH \geq 95\%$ and $T = 20 \pm 2^\circ\text{C}$ until they reached a total age of 28 days. The hardened mortar cubes were then cut into slices with a thickness of approximately 20 mm with a diamond saw. The slices with dimensions of 20 x 100 x 100 mm were further stored in the laboratory atmosphere ($T \approx 20^\circ\text{C}$ and $RH \approx 60\%$), for one week. Then the mortar slices were ready for impregnation with a silane gel, i. e., liquid silane stiffened by addition of fine clay particles.

Table 1. Composition of the mortar, the mass of cement, sand and water is given as kg/m³. Cement OPC type CEM I 42.5; river sand with a maximum grain size 2.5 mm.

W/C	Water	Cement	Sand
0.6	300	500	1650

Surface impregnation

The surface of the mortar slices, which was in contact with the bottom of the steel forms during casting, was selected for impregnation as this represents a formed surface in contrast to a finished surface. This formed surface was impregnated with silane gel (StoCryl HG200 from StoCretec [1]). The active ingredient (approximately 85%) is an alkyl alkoxy silane in ethanol according to the safety data sheet [2]. The main component of the active ingredient is octyl triethoxy silane and the product is formulated with a mineral thickener [3].

Three different amounts of silane gel were carefully spread uniformly over the surface: 100 g/m², 400 g/m² and 600 g/m². The differently treated samples were labeled with G100, G400 and G600, respectively. All three types of impregnated mortar specimens were then stored for four weeks in the laboratory atmosphere to allow the penetrated silane to react fully, by forming a silicon resin film on the inner surface of the mortar pores, and to reach hygric equilibrium with the laboratory atmosphere this being checked gravimetrically.

Neutron digital imaging

Cross sections of the water repellent mortar specimens and untreated reference mortar specimens were then tested in the neutron radiographic facilities NEUTRA at Paul-Scherrer-Institute (PSI) in Villigen, Switzerland. The diameter of the neutron beam was 400 mm and the collimation ratio was 550. The neutron flux during the tests was $5.1 \times 10^6 \text{ cm}^{-2} \text{ s}^{-1} \text{ mA}^{-1}$. More details on neutron radiography are described elsewhere [4-6].

First, neutron images were taken from samples at hygric equilibrium with the room atmosphere ($T \approx 20^\circ\text{C}$ and $\text{RH} \approx 60\%$). Then both silane impregnated and untreated samples were placed in water for three days. This period was sufficient for complete saturation of the samples by capillary absorption. Then neutron images were taken from these water saturated samples. From the neutron images the moisture distribution could be determined quantitatively.

RESULTS AND DISCUSSION

Neutron digital imaging

Neutron images of the cross sections of the three types of impregnated and water saturated mortar specimens are shown in Fig. 1. Since in this context, only the upper impregnated surface is of interest, just this area of the neutron images is shown in the figure. The darker the image the higher the water (or hydrogen bearing material) content. The contrast difference of the neutron transmission is significantly higher in the outer impregnated layer. The thickness of the impregnated layer can be estimated already from the results shown in Fig. 1. The following average values of the thickness of the impregnated layer were determined by visual inspection 2.0, 4.1, and 6.3 mm for samples G100, G400, and G600 respectively.

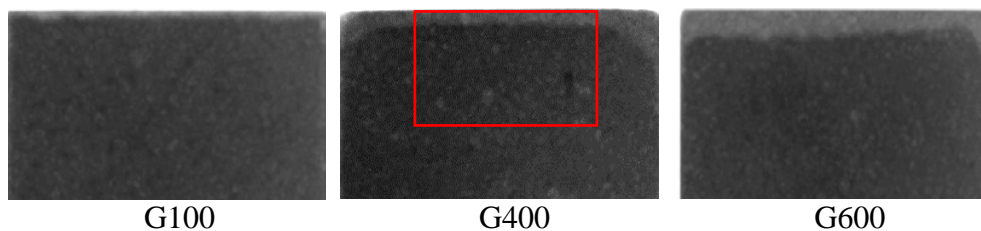


Figure 1. Neutron images as obtained from the cross section of the water saturated mortar specimens impregnated with the three different amounts of water repellent agent. Only the upper half of the neutron images taken on square slabs is shown. The thickness of the impregnation layer (lighter in color) is clearly proportional to the amount of applied water repellent agent.

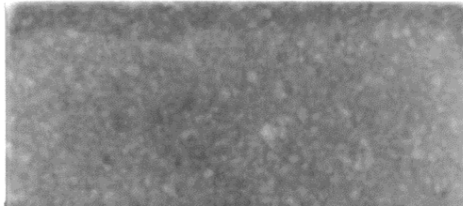


Figure 2. Neutron image of an air-dry specimen after impregnation with 600 g/m² of silane gel (G600). In this case the silicon resin in the impregnated zone appears darker because it holds more hydrogen than the rest of the mortar.

The darker area in Fig. 2 represents the impregnated subsurface zone below the surface where the polymerized silane was applied. Enough hydrogen is fixed in it, in both OH⁻ and CH_x-groups to originate the scattering of neutrons from the neutron beam. This makes it appear darker on the image. The thickness of this zone corresponds to the thickness of the zone with reduced water content shown in Fig. 1. The technology to visualize the polymerized silane films opens up new possibilities and will be further developed in the future.

The neutron transmission value of untreated and silane impregnated mortar specimens was also quantitatively evaluated. Figure 3 shows the transmission profiles as obtained from untreated specimens stored in the laboratory atmosphere as well as water saturated. As expected, the neutron absorption of the water saturated specimens is much higher. The difference between the two lines shown in Fig. 3 is approximately constant, that means samples are homogeneously filled with water. In this case the water content of the air dry samples was 6.5% by volume, and 18.4% by volume for the water saturated ones. Consequently, the distance between the two lines in Fig. 3 represents a homogeneous water content of 11.9 % by volume. From these data we can calculate that in the hydration products of the dry mortar there is approximately 4.1 % by volume of water chemically bound.

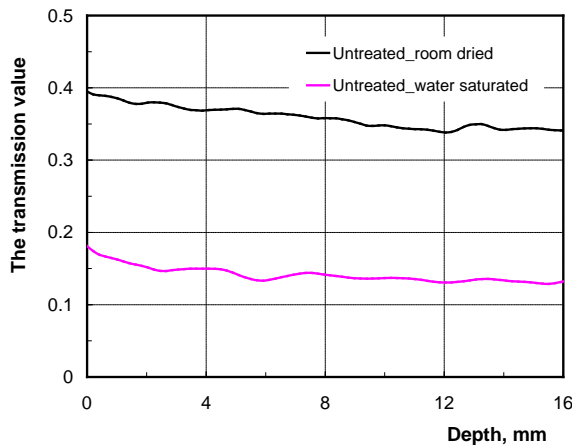


Figure 3. Neutron transmission value as a function of the distance from the surface, later to be impregnated, measured on both untreated water saturated and room dried samples.

The neutron transmission value determined on the three cross sections of the surface impregnated specimens G100, G400 and G600 are shown in Fig. 4. The transmission of samples G100 in the bulk of the specimens is nearly constant but close to the impregnated surface transmission increases. The influence of the silane impregnation in samples G100 can be observed to a depth of approximately 2 mm. In the air dried specimen there is less water in the surface near zone. That means that a certain part of the pores which are filled by capillary condensation in untreated specimens are empty after water repellent treatment, because the walls of these pores are covered by the polymerized silane film. Very fine pores, in the order of some nanometers, could still contain capillary condensed water under the environmental relative humidity. It is quite obvious that the influence of the reduced water content on neutron transmission is much stronger than the influence of the polymerized silane. Nevertheless for a quantitative analysis the contribution of the polymerized silane has to be taken into consideration.

The situation in the G400 samples is similar but the influence of the water repellent layer reaches deeper into the material indicating a thicker layer, while for the G600 specimens the layer attains a thickness of approximately 6 mm and the water content is strongly reduced in this layer. There is only a small difference between the transmission of the dry and the water saturated specimens in the subsurface impregnated zone and the water content in the water saturated state approaches that of the room dried samples. Nearly no water has been taken up by capillary absorption. This situation can be considered to be the necessary requirement for the establishment of a reliable chloride barrier.

Water distribution

The water distribution was calculated in the subsurface zone —indicated with the rectangular frame marked on the G400 sample— shown in Fig. 1 for the water saturated specimens. Results are shown in Fig. 5. For the case of untreated specimens the moisture content is essentially homogeneously distributed all over the cross section. The observed slight decrease of water content close to the surface may be attributed to a small water loss during handling before taking the first neutron image.

For the impregnated specimens, however, the influence of the water repellent subsurface zone can clearly be observed where the water content is significantly reduced. The width of the water repellent zone can also be observed quite clearly. In samples G100 a water repellent layer with a thickness of approximately 2 mm was measured which increases to approximately 4 and 6 mm for the G400 and G600 samples, respectively. What is most important, however, is the fact that in samples G100 and G400, although the water content in the water repellent zone is certainly substantially reduced there is still a certain amount of water present. In contrast, the G600 samples show only a minimum amount of water, barely different between the wet and dry samples. From these results it can be concluded that deep impregnation, as shown for the case of the G600 samples, is necessary for an efficient chloride barrier, since, as mentioned previously, the small amount of water remaining in the impregnated specimens G100 and G400 may still transport a certain amount of dissolved chloride ions into the concrete.

This observation is in good agreement with results from tests to assess the effectiveness of surface impregnation with respect to chloride penetration. If the water repellent zone is not thick enough the rate of chloride penetration is reduced but not eliminated. To establish a real chloride barrier, absolutely necessary for a significant increase in service life, a water

repellent zone as established in specimens type G600 is necessary [7]. The remaining water in samples type G100 and G400 is sufficient to transport a reduced amount of chloride through the barrier that will eventually reach the concrete reinforcement. For this reason deep impregnation is required as previously recommended [8-12] if an effective and long lasting chloride barrier is to be established in concrete.

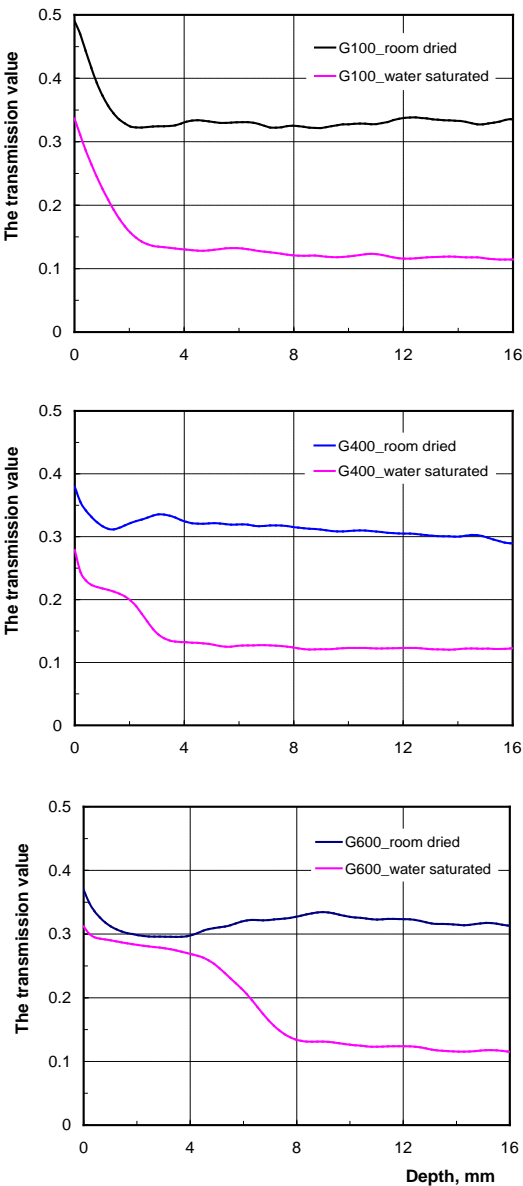


Figure 4. Neutron transmission value as measured on the images for the three types of water saturated and room dry mortar specimens G100, G400 and G600.

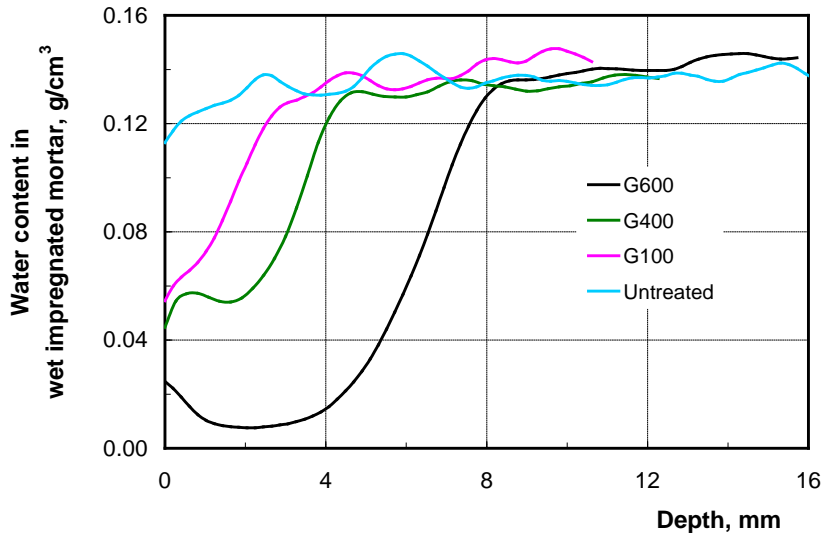


Figure 5. Water content in surface impregnated and water saturated mortar specimens G100, G400 and G600. For comparison the water distribution in untreated mortar is also shown.

CONCLUSIONS

Based on the results presented in this contribution it can be concluded that:

- Water repellent surface layers with a thickness below 6 mm still allow penetration of a certain amount of water when in direct contact with water. This water may permit chloride ions to be transported via “water bridges”. Chloride ingress into the pore space is slowed down but not prevented.
- To prevent chloride penetration into concrete, deep impregnation with silane is necessary. A minimum thickness of the water repellent surface layer of 6 mm must be achieved.
- After water repellent surface treatment a strict quality control must take place to ensure that the minimum thickness of the water repellent layer has been achieved.

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References

- [1] StoCretec. 2011. StoCryl HG200.
http://www.stocretec.de/119132_DE-Produkte-Produkte_A-Z.htm?prodId=PROD0537
- [2] Sto Scandinavia. 2014. StoCryl HG200 Säkerhetsdatablad. (Product Safety Data Sheet)
http://www.sto.se/sv/produkter_system/produkter/produkt_161210537.html
- [3] HAAG, C. 2009. *Ecological and economic benefits of an impregnation with StoCryl HG 200* Institute for Building Materials Report, ETH Zurich
http://www.stofi.fi/58295_FI-Betong-Report_ETH_StoCrylHG200.pdf
- [4] PLEINERT, H. 1998. *Determination of Moisture Distributions in Porous Building Materials – Neutron Signal Transfer Analysis*. Building Materials Reports No.10, Aedificatio Publishers Freiburg (1998)
- [5] PLEINERT, H., H. Sadouki, F. H. Wittmann. 1998. *Determination of Moisture Distributions in Porous Building Materials by Neutron Transmission Analysis*. Materials and Structures 31:218-224.
- [6] WITTMANN, F. H., P. Zhang, T. Zhao. 2011. *Damage of Concrete under Combined Actions*, Restoration of Buildings and Monuments, 17:321-330.
- [7] ZHAN, H., F. H. Wittmann, T. Zhao. 2005. *Relation between the Silicon Resin Profiles in Water Repellent Treated Concrete and the Effectiveness as a Chloride Barrier*, Restoration of Buildings and Monuments 11:35-46.
- [8] MEIER, S. J., F. H. Wittmann. 2011. *Water repellent Surface Impregnation of Concrete: Guidelines and Recommendations*. In Basic Research on Concrete and Applications, Proc. ASMES Int. Workshop, ed. Wittmann F. H., Mercier O., pp. 49-66. Aedificatio Publishers, Freiburg, Germany.
- [9] MEIER, S. J., F. H. Wittmann. 2005. *Hydrophobieren von Betonoberflächen – Empfehlungen für Planung und Applikation; Traitement hydrofuge de la surface des structures en béton – Recommendations pour la planification et l'exécution*, Swiss Federal Office for Roads, ASTRA Report No. 591.
- [10] MEIER, S. J. 2003. *Grundlagen und Möglichkeiten einer Hydrophobierung von Betonbauteilen*. Building Materials Reports No. 21, Aedificatio Publishers, Freiburg.
- [11] GERDES, A., F. H. Wittmann. 2003. *Hydrophobieren von Stahlbeton, Teil 1: Transport und chemische Reaktionen silicium-organischer Verbindungen in der Betonrandzone*. Restoration of Buildings and Monuments 9[1]:41-64.
- [12] GERDES, A., F. H. Wittmann. *Hydrophobieren von Stahlbeton, Teil 2: Ausführung einer Hydrophobierung – Voruntersuchung, Durchführung und Qualitätssicherung*. Restoration of Buildings and Monuments 9[2]:117-137.