# Experimental method of measuring the efficiency of hydrophobic surface layer of concrete structures

R. Pernicová<sup>1,a</sup>, P. Tichá<sup>2</sup>

<sup>1</sup> Czech Technical University in Prague, Klokner Institute, Šolínova 7, 166 08 Prague 6, Czech Republic <sup>2</sup>Czech Technical University in Prague, Faculty of civil engineering, Thákurova 7, 166 29 Prague 6, Czech Republic <sup>a</sup> radka.pernicova@cvut.cz

**Abstract:** The review of hydrophobic materials application on surfaces of concrete structures to achieve its protection again the environmental conditions is described in this paper. The article is focused on the characteristics of selected hydrophobic materials, chemical base of origin and their characteristics. The second part describes the possibility of experimental methods of measuring of new hydrophobic surface layer, particularly the quality and efficiency of their hydrophobic properties.

Keywords: hydrophobic impregnation; concrete; contact angel; surface tension; penetration.

## **1** Introduction

Porous building materials could absorb a lot of water due to its porous structure which may cause some negative effects on material. Temperature is one of the most significant side effects, especially below zero level. This could create cracks that can leads to deformation of integrity of the material. Another equally important negative side effect is water function as the carrier medium. Transport of ions in direction away from construction could lead to leaching of the compounds and their subsequent efflorescence on the surface [1]. In the opposite direction harmful substances such as chloride ions from de-icing salt may be carried into the material and therefore slightly damage its structure [2].

The main aim of building engineers all round the world is to provide quality isolation of the material, keep it away from humidity and avoiding the possible damage such as decrease of reliability and durability of the building constructions or its parts. Hydrophobicity of surface could be increased by using extra protective layers. This work is focused on description of characteristic of hydrophobic impregnations.

Hydrophobic impregnations have various forms of phases. The most commonly is used an emulsion, solutions, pastes and creams. An indispensable influence on quality and durability of hydrophobic product has properties of the concrete surface to which is hydrophobization applied and compliance with the technological process.

To protect outer building construction surface against external conditions, hydrophobic impregnation, impregnation or coatings could be used. Coat creates continuous film on the top of concrete that fills pores and capillaries by impregnation substance and therefore the top layer is sealed. The impregnation decrease porous characteristic of concrete and therefore strengthen its surface. As the result, capillaries and pores are filled, partly or fully. Hydrophobization of concrete generate thin layer on the surface of capillaries and pores that avoid fluid to pass through it, unlike gas and steam [3]. Pores and capillaries are internally coated but not filled.

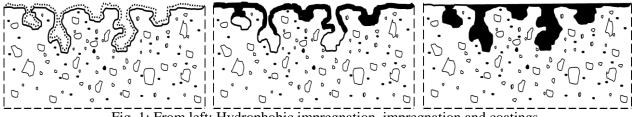


Fig. 1: From left: Hydrophobic impregnation, impregnation and coatings

# 2 Hydrophobic impregnation

The essential element of hydrophobic impregnations is silicon dioxide and other organic compounds that determine the final properties of the impregnation. Silica ensures adhesion to the surface because it is also inside the concrete substrate. Organic compounds remain on the surface layer are firmly embedded in the substrate and significantly alters the wettability of the surface of capillaries. Most of the hydrophobic impregnation is made by chemical base of following substances: silane, siloxane and siliconate [4] [5] [6] [7].

### 2.1 Silanes

Silanes have large molecules from the chemical point of view, non-hydrolyzed silanes (i.e. esters of silicic acid). Their chemical formula is SiH<sub>4</sub> and basic molecular bond is a C-Si. Silanes have a quality penetration properties and penetration achieves great depth. Therefore, materials treated with this substance are able to prevent the ingress of water, even threw micro-cracks in the surface of structure up to width four tenth of a millimeter. Silanes react with moisture on the silica gel which besides the hydrophobic has also the strengthening effect. Silanes have an ability to create chemical bond on the surface of quartz grains, which means that it is impossible to remove them from the surface. Anhydrous alcohols as well as aliphatic materials are used as solvents.

#### 2.2 Siloxanes

Siloxanes are the most frequent and suitable used hydrophobic products for different kinds of building materials, out of all mentioned hydrophobic materials. It is a functional group in organosilicon chemistry with the Si–O–Si linkage. The parent siloxanes include the oligomeric and polymeric hydrides with the formulae  $H(OSiH_2)_nOH$ ,  $(OSiH_2)_n$  and have several functional groups such as silicones, alkylalkoxysiloxanes or Alkoxy polysiloxane. Siloxane products are one-component, and since the resins are soluble in white spirit, so this impregnation cannot be used for wet or damp surfaces. Siloxanes have large molecules, achieving only a little depth of penetration. Its impregnation can be renewed, because it is possible to impregnate previously hydrophobic surface. Siloxane coating is soluble in white spirit, which helps to remove old paint. However these hydrophobic impregnations represent threat to the environment and are flammable too.

The low molecular oligomeric alkylalkoxysiloxanes are mostly one-component hydrophobic impregnation that spontaneously polymerize for the presence of atmospheric moisture. They are capable of binding chemically to the surface of quartz grains, which is the reason why they are the best option for hydrophobic impregnation of silicone materials (i.e. concrete and cement products). The impregnation bond to quartz grains increase abrasion resistance of coating and at the same time prevent by coating removability. The low molecular oligomeric alkylalkoxysiloxanes are the most effective penetrate into porous materials comparing others hydrophobic impregnations. They may have either paste or cream form, in that case, are most commonly use on fresh concrete surface to achieving deeper penetration of hydrophobic impregnation.

Alkoxy polysiloxanes are solutions of polysiloxane resins transferred into an aqueous emulsion. The polysiloxanes impregnating agents are water-soluble and its hydrophobic effect creates after evaporation of water that means, they can be applied to damp surfaces. For the production of polysiloxane impregnating the emulsifiers, leach out of the polymer film, are used. That is the reason, why the final hydrophobic effect will appear after a certain time. More precisely, called polymerized siloxanes, silicones are mixed inorganic-organic polymers with the chemical formula  $[R_2SiO]_n$ , where R is an organic group. These materials consist of an inorganic silicon-oxygen backbone chain (-Si-O-Si-O-) with organic side groups attached to the silicon atoms. Silicon as hydrophobic agent can be used in form micro emulsions, which create aqueous emulsion while they are mixed with water. Silicone micro emulsions have an advantage in ability to be miscible with water and therefore there is no need for organic solvent.

### 2.3 Siliconates

Siliconates are hydrophobic substance that reacts with carbon dioxide to form a gel of silicic acid. Chemically, they are methylsiliconate of sodium or potassium and have small polar molecules, which do not achieve significant penetration depth into the concrete structure. Incurred silicic acid gel is not soluble, therefore coating is irremovable. In the reaction siliconates with carbon dioxide sodium or potassium hydroxide is released. These new products may cause the efflorescence on surface of concrete, which appears the most often after application at lower temperature and could devaluation of concrete products as the result

## **3** Measurement methods

Currently, we have several measuring methods which determine level of hydrophobicity of concrete structures.

### 3.1 Measuring of contact angle and surface tension

Most of them measure directly or indirectly contact angle formed by tangent the surface of the liquid drop to the contact with surface of the solid phase. Hydrophobic material, which creates water-repellent layer on surface of concrete, causes an increase of water contact angle. The degree of hydrophobic efficacy is determined by the contact angle  $\Theta$  formed between the tangents to the drop of water on surface with the washer.

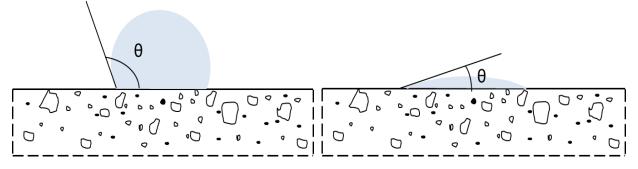


Fig. 2: Wettability of surface: hydrophobic (left) and hydrophilic (right)

There are 4 categories depending on the size of contact angle. The lower value of the contact angle means better hydrophobic properties of concrete specimens [8].

Tab. 1: Categories of hydrophobicity according the contact angle
--

hydrophilic	hydrophobic	super-hydrophobic	ultra-hydrophobic
$\Theta < 90^{\circ}$	$120^\circ > \Theta > 90^\circ$	$150^\circ > \Theta > 120^\circ$	$\Theta > 150^{\circ}$

Contact angle is one of the few directly measurable properties of the three-phase interface of a solid / liquid / gas. The balance in three-phase system describes Young's equation of equilibrium of forces on the interface.

$$\gamma_{sg} = \gamma_{sl} + \gamma_{lg} \cdot \cos \Theta$$
 or  $\cos \Theta = (\gamma_{sg} \cdot \gamma_{sl}) / \gamma_{lg}$  (1)

In this equation  $\gamma_{lg}$  represents the value of the interfacial tension at the interface of the two relevant phases. This equation assumes the perfectly smooth homogeneous surface [9].

Measuring the static contact angle of drop on non-porous, solid flat surface is done by the help of video cameras or goniometer. Drop of water adjacent to the solid is observed by a microscope equipped with a goniometric eyepiece allowing direct reading contact angle or evaluation later from drops image. Determination of the contact angle by this method is within 1° of accuracy. Indirect determination of contact angle is based on strain-gauge methods (capillary elevation on the vertical plate, balancing Wilhelmy plate). Based on that, detected contact angle can calculate surface energy of the material and use this information to better understand the hydrophobic behaviour [10].

## 3.2 Measurement of hydric properties

Basic parameters of liquid moisture transfer, namely saturated moisture content, maximum absorbency, absorption coefficient are another kind of measurement to detect the hydrophobicity level of building materials. Transport of liquid moisture was studied using the standard sorption experiment, the one-dimensional absorbency of the sample, which is based on a classic experiment of soaking from the water surface. Obtained measured data shows clearly that we can construct the dependence of the cumulative moisture content, from which we can directly determine the moisture absorption coefficient A  $[kg/m^2h^{1/2}]$  using the linear part of the increase in weight [11].

#### 3.3 Measurement of penetration depth

The most important criterion of the hydrophobic impregnation durability is the penetration depth. Standard ČSN EN 1503-2 divides impregnation into two groups: I Class means penetration to a depth of 10 mm; II Class means depth more than 10 mm. Depth of impregnation is performed by informative qualitative phenolphthalein alkalinity test of concrete at borehole. Based on colorimetric test, it is in the reaction of phenolphthalein with concrete. The concrete itself has high alkalinity (pH 12-13). In contact with the impregnation concrete is neutralized and its alkalinity is significantly reduced. Main principle of the test is to immerse the borehole into the phenolphthalein solution, finding out the depth of penetration. The depth is determined by coloring concrete. The colorless solution of phenolphthalein dissolved in ethanol is turned purple at a pH greater than 9.5 and at a pH lower than 9.5 the solution stays colorless.

#### **3.4 Conductivity measurement**

Quality assessment of already applied hydrophobic impregnation on the sample surface is most commonly performed using conductivity measurement. Experimental device works on principle of measuring ac voltage of +/- 700 mV and a frequency of 20 Hz, which operates through electrodes on pre-defined surface area. In place of the impregnation failure tension is transmitted into the concrete and it is subsequent measurement on the other non-impregnated side of concrete specimen. The final value is a function of the number of defective places. The bigger is the measuring values the more damaged areas are in the hydrophobic impregnation. Escalating tension curve indicates of bad adhesion of hydrophobic impregnation to the concrete surface.

# **4** Conclusion

The interaction between the solid and liquid is the key role in understanding chemical and physical processes in the civil engineering, specifically in the area of interaction of building material with the external environment. The influence of UV radiation and other environmental conditions has to be taken into account, causes decay of organic ingredients and therefore hydrophobic ability is decreasing. Siloxanes are the most suitable for the most different kinds of building materials, out of all mentioned hydrophobic materials. However, it still depends on the particular material solutions and the exactly situation of the building in deciding which hydrophobic agent is best to use. Choice of an unsuitable hydrophobic product can have fatal consequences for the concrete product. Most hydrophobic impregnations are unrecoverable from the surface and they can be removed only mechanically therefore selection of suitable Impregnation is really important.

## Acknowledgement

The research was supported by Grant Agency of the Czech Republic under the number GACR 15-10591S.

# References

- [1] R. Pernicova, Analysis of formation and testing of efflorescence on concrete elements, Advanced Materials Research Vol. 1025-1026 (2014) p. 641-644.
- [2] D. Dobias, R. Pernicova, Diffusion of Chloride Ions in Ultra High Performance Concrete, Advanced Materials Research Vol. 1106 (2015) p. 21-24.
- [3] J. Soucek, P. Tichá, Modeling Interior Environment of Historical Building and Determination of Critical Places for Application of Nanotextiles Protection, Advanced Materials Research, (2014), Vols. 1025-1026, p. 1141-1144.
- [4] J. Vetchy, Hydrofobní impregnace betonu, MCT Texty o betonových výrobcích, (2015) p.1-6
- [5] V. Bažant, Technické použití silikonů, (1959), SNTL Praha.
- [6] A. Selande, Hydrophobic impregnation of concrete structures Effect on concrete properties, Ph.D. Thesis, (2010), Royal Institute of Technology, Stockholm, Sweden. [5] V. Bažant, Technické použití silikonů, (1959), SNTL Praha.
- [7] J. Blitz, C. Little, Fundamental & Applied Aspects of Chemically Modified Surfaces, (1999), Royal Society of Chemistry, Cambridge, UK. [5] V. Bažant, Technické použití silikonů, (1959), SNTL Praha.
- [8] I. Krasny, Measurement of contact angel and determination of surface free energy of plastic materials, Thesis, (2010), Tomas Bata University in Zlin, Faculty of technology, Czech Republic [5] V. Bažant, Technické použití silikonů, (1959), SNTL Praha.
- [9] J.Holub, Smáčivost povrchu vůči kapalinám a druhá viskozita kapalin, Brno: Vysoké učení technické v Brně, Fakulta strojního inženýrství, (2010) pp. 38
- [10] O. Chmela, Different imaging techniques for investigation of treatment effects on various substrate surfaces, Proceedings of the 21st Conference STUDENT EEICT (2015). pp. 351-355.
- [11] S. Roels, J. Carmelit, H. Hens, O. Adam, H. Brocken, R. Černý, Z. Pavlík, C. Hall, K. Kumaran, L. Pel, R. Plagge, Interlaboratory Comparison of Hygric Properties of Porous Building Materials, In Journal of Thermal Envelope and Building Science, Vol. 27, (2004), pp. 307-325.