

Zbigniew SUCHORAB^{1*}, Danuta BARNAT-HUNEK², Piotr SMARZEWSKI³
Zbyšek PAVLÍK⁴ and Robert ČERNÝ⁵

FREE OF VOLATILE ORGANIC COMPOUNDS PROTECTION AGAINST MOISTURE IN BUILDING MATERIALS

ZABEZPIECZENIA PRZEGRÓD BUDOWLANYCH PRZED WILGOCIĄ WOLNE OD LOTNYCH ZWIĄZKÓW ORGANICZNYCH

Abstract: The article presents information about moisture protection of building materials. The discussed parameters determining the efficiency of the water protection are material porosity, water absorptivity and surface condition of building materials. Moreover the ecological aspect of hydrophobic VOC-free preparations available on the market has been underlined. The first part of the article is a description of moisture problem in the building envelopes and the possibilities of its prevention. The special attention is put on the electric methods of moisture estimation with a special emphasis on the Time Domain Reflectometry (TDR) method. The second part of the article is devoted an experiment of model red-brick walls exhibited on capillary uptake process. For the experiment three model red-brick walls were built and prepared for water uptake process. The experiment was monitored by the capacitive and surface TDR probes thanks to which the necessity of sampling and material destruction could be avoided. Conducted experiments show the progress of water uptake phenomenon in the model walls which differ in type of protection against moisture and prove the potential of the non-invasive measurements using the surface TDR probes. Basic physical parameters of the applied bricks were determined together with the reflectometric measurements. Furthermore, Scanning Electron Microscopy (SEM) was used to analyze the hydrophobic layer continuity.

Keywords: capillary rise, red brick, surface TDR probes, hydrophobization, VOC-free preparations, volatile organic compounds, scanning electron microscopy

¹ Faculty of Environmental Engineering, Lublin University of Technology, ul. Nadbystrzycka 40a, 20-618 Lublin, email: z.suchorab@wis.pol.lublin.pl

² Department of Building Construction, Faculty of Civil Engineering and Architecture, Lublin University of Technology, ul. Nadbystrzycka 40, 20-618 Lublin, email: d.barnat-hunek@pollub.pl

³ Department of Civil Engineering, Faculty of Civil Engineering and Architecture, Lublin University of Technology, ul. Nadbystrzycka 40, 20-618 Lublin, email: p.smarzewski@pollub.pl

⁴ Department of Materials Engineering and Chemistry, Faculty of Civil Engineering, Czech Technical University in Prague, Thákurova 7, 166 29 Prague 6, Czech Republic, email: pavlikz@fsv.cvut.cz

⁵ Department of Materials Engineering and Chemistry, Faculty of Civil Engineering, Czech Technical University in Prague, Thákurova 7, 166 29 Prague 6, Czech Republic, email: cernyr@fsv.cvut.cz

* Corresponding author: z.suchorab@wis.pol.lublin.pl

Introduction

Water is considered to be one of the most important chemical compounds on Earth. It influences all living organisms and most of the processes. On the other hand, unexpected water presence may lead to many problems, often connected with buildings performance. Water presence in the building materials is a well-known and common phenomenon. It occurs both in old buildings and new objects, often being the consequence of improper building constructions.

In the building envelopes water occurs in three phases - solid, liquid and gaseous. Water presence in newly built objects is most often connected with the technology of materials production and building works regime. Together with the natural processes of moisture sorption and desorption occurring during the building exploitation, the properties of applied materials also influence the moisture performance of the envelopes [1].

It has been noticed that chemically bound water does not influence moisture properties of the building barriers [2]. Also, water vapor presence is less influencing, compared to the other types of water present in the barriers. Sorption and capillary water are the most meaningful. The problem of water migration in the building barriers is quite difficult to describe, mostly due to inhomogeneous structure of materials and barriers. What is more, the fact that the particular masonry elements like bricks or blocks are combined with mortar which has totally different parameters from the main applied material, makes it problematic. All the above mentioned factors cause serious problems for the theoretical and experimental description of water infiltration.

One of the methods of brick masonry protection against rainwater is hydrophobizing [3-5]. An important advantage of this procedure is the fact that preparations used for material impregnation form a thin, colorless layer with good adherence parameters, which is not prone to aging. Hydrophobic coating ought to be watertight and impermeable for water solutions of salt, however enable water to evaporate from the material [6].

Currently, mainly organosilicone compounds are applied for hydrophobization [7-13]. The most effective and safe preparations for hydrophobization belong to the silicone group. Among them there are two kinds of preparations: based on water and the organic solvents. Volatile substances present in the hydrocarbon preparations can be toxic and carcinogenic [14-20]. The most important regulation about Volatile Organic Compounds (VOC) emission in European Union is European Commission "Decopaint" Directive 2004/42/EC [21] concerning VOC emission limitation due to the application of organic solvents in particular paints and lacquers. It limits the acceptable VOC content in products used for decorative and renovation painting. For building renovation and conservation all the European Union members are allowed to provide their individual licenses for selling and application in strictly determined amounts of the products, which do not fulfill the VOC limits assumed in the "Decopaint" Directive.

Currently, the producers of building chemistry are facing the necessity to protect the natural environment according to the regulations of the Volatile Organic Compounds emission limitations. The most important ways to reduce VOC emission from the impregnates are the following: the application of water preparations instead of the solvent-based, the reduction of solvent content, as well as the reduction of VOC content in water preparations. Law regulations have forced the chemical concerns to develop and produce

water impregnating emulsions [22, 23]. Water emulsions of silanes form suspensions composed of two insoluble liquids. Silane is mixed with water and an emulsifier [24].

Literature analysis shows that the micromolecular oligomers have the deepest penetration of the porous materials and the poorest penetration is typical for water-soluble preparations [11, 25]. However, in many cases new emulsions with low concentration of VOC are similarly efficient in hydrophobization as the products based on organic solvents.

The aim of the research presented in this article was to evaluate the possibility to apply hydrophobic preparations based on organosilicones for impregnation of the ceramic building materials. The process of the surface hydrophobization was analyzed. Water substances were used for impregnation. The efficiency of two preparations was verified. They differed in grade of hydrolytic polycondensation, viscosity and concentration because those are the main factors influencing the final effect of hydrophobization procedure. Moisture change analysis and water transport in a standard and impregnated ceramic brick model walls were conducted using the method of continuous monitoring - Time Domain Reflectometry (TDR). The obtained results allowed to estimate the efficiency of hydrophobization using emulsion with low VOC content.

Red ceramic brick is one of the most popular building materials on the Polish building market. It is especially used in the masonries and substructures of many buildings. As a common building material it was used to build three model building barriers which were investigated for capillary uptake susceptibility.

The capacitive and modified TDR (Time Domain Reflectometry) methods were applied for the monitoring of moisture changes in the model barriers. They enabled non-invasive moisture measurement in the porous materials. The capacitive measurement method relies on the determination of the capacitance of a sensor filled with the material under test. This method enables quick moisture determination with simple, user friendly mobile device - Figure 1, which is non-invasive and does not require samples preparations, especially effective for *in-situ* measurements. Apart from water content, this method is sensitive to the electrical conductivity of the material under test, which can produce significant errors in moisture measurements [26].



Fig. 1. Capacitive moisture meter (LAB-EL, Poland)

The Time Domain Reflectometry, according to its name, is a reflectometric technique which estimates dielectric permittivity of the measured material by the determination of the velocity of the signal propagation. The TDR technique was previously successfully used for moisture determination of soils [27-33] and some building materials [34-37]. Until now the application of the TDR method for building materials was not easy due to the necessity of probes internal installation, and was only possible to be applied in laboratory conditions, mainly for soft building materials. Since several years, the surface TDR sensors have been

developed and now they seem to be prospective alternative for the non-invasive moisture determination in building materials and the walls. Figure 2 presents the example of the surface TDR probe manufactured at the Lublin University of Technology. Construction of the described sensor is presented in the article [38].



Fig. 2. Surface Time Domain Reflectometry probe (Lublin University of Technology)

Materials and methods

Before rising the model walls, material used was examined for basic physical parameters. The determination of real density, apparent density, volume of the open pores, open and total porosity was conducted with the use of the regulation: EN 1936:2010 [39].

The setup for capillary rise phenomenon consisted of three model walls made of the red ceramic bricks of 210×120×65 mm dimensions (Fig. 3). Bricks of the masonry were combined with cement mortar 10 mm wide. The first wall, also described in the following paper [40], was not impregnated with any hydrophobic product and in the further part of the article it is referred as “the Standard”.

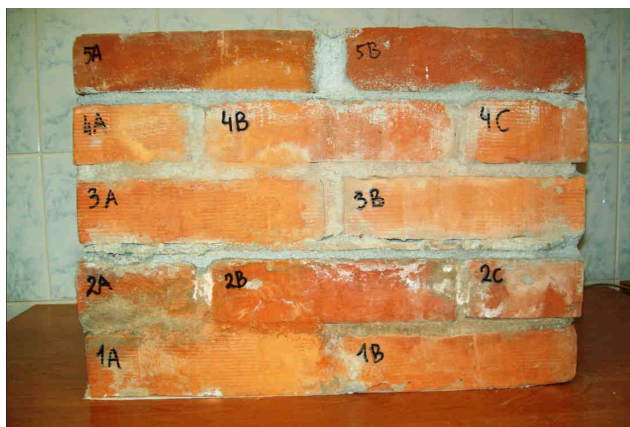


Fig. 3. Photograph of the exemplary model wall constructed for determination of capillary uptake phenomenon

The next two walls were similar with respect to the used materials and geometry. However they were impregnated with suitable hydrophobic preparations P1 and P2:

P1 - water solution of methylsilicone resin in potassium hydroxide (KOH),

P2 - water-soluble silicone.

Before the experiment, all the walls were seasoned in laboratory conditions for the period of 30 days in a room temperature $20\pm 2^{\circ}\text{C}$ and relative humidity $60\pm 5\%$. The aim of the seasoning procedure was to obtain the acceptable low relative moisture between 2 and 3% suggested by most of the hydrophobic preparation producers.

Then, two walls were hydrophobized according to the suggestions of the preparation producers by double covering with a brush. According to the above mentioned suggestions, the preparations were diluted in the following proportions 1:6 and 1:8, respectively. Then, all the samples were seasoned for the period of 7 days in laboratory conditions to start polycondensation and formation of the polysiloxane gel on the bricks surface.

The capacitance sensor (Fig. 1) and the modified TDR probe (Fig. 2) were applied for the capillary rise test in the Standard walls. This enabled non-invasive measurements of moisture changes. For the impregnated walls (P1 and P2) only Time Domain Reflectometry sensors were used. Measuring setup, presented in Figure 4 consisted of:

- three model walls placed in the suitable water containers,
- LB-796 capacitive moisture meter (LAB-EL, Poland) (Fig. 1),
- TDR multimeter (LOM/EasyTest, Lublin, Poland),
- surface TDR probe (Lublin University of Technology, Poland) (Fig. 2),
- PC computer as the control station.

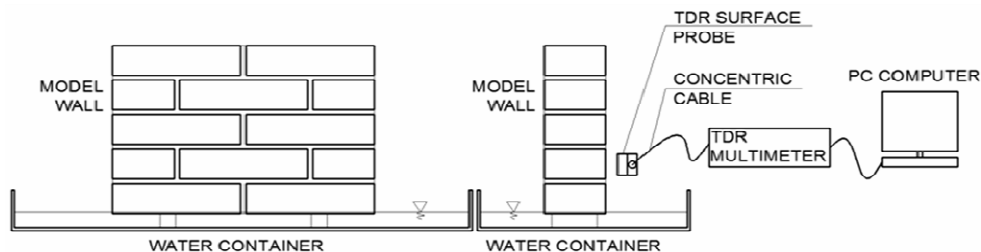


Fig. 4. Measuring setup for non-invasive measurements of water uptake inside the walls

The particular bricks in all prepared model walls were marked with the following symbols 1A, 1B, 2A, 2B, 2C, etc., as presented in Figure 3. The digit meant the particular layer of the bricks. The prepared wall was dried in 105°C and put into the water container with constant water level 10 mm above the bottom edge of the measured wall. The moisture changes of the wall were monitored by the capacitance (only the Standard wall) and the surface TDR sensors for the period of 30 days with 24 hour intervals. The TDR instrumentation applied for the experiment enabled readouts of the electromagnetic pulse propagation along the measuring elements of the surface probe, which were used for the calculation of the effective dielectric permittivity (depending on surface sensor construction) and thus moisture. Calibration experiments were presented in the following articles [38, 41]. Capacitive probe was pre-calibrated by the device producer (LAB-EL, Poland) for most of the typical building materials and enabled quick moisture measurement without the necessity to calibrate the sensor.

Conducted research was supplemented with the Scanning Electron Microscopy (SEM) microstructural examination of the standard and impregnated bricks to analyze the

hydrophobic coating in the structure of the ceramic bricks. The applied equipment was the scanning electron microscope FEI Quanta 250 FEG.

Results

The following physical parameters of brick used for model walls were determined according to EN 1936:2010 [39] regulation: volume of the open pores $V = 19.66 \text{ cm}^3$, apparent density $\rho_b = 1.69 \text{ g/cm}^3$, real density $\rho_r = 2.44 \text{ g/cm}^3$, open porosity $\rho_o = 26.67\%$, total porosity $P = 33.57\%$.

Standard, non-impregnated with hydrophobic products model wall was examined using two technologies - by the capacitive and surface TDR sensors. Both techniques enabled the determination of the dynamics of moisture increase due to capillary forces in the standard model wall made of red ceramic brick. The process is presented in the figures presented below. Figure 5 shows the average data obtained by capacitive measurements whereas Figure 6 depicts average data obtained by the TDR equipment with the application of the non-invasive surface TDR sensor.

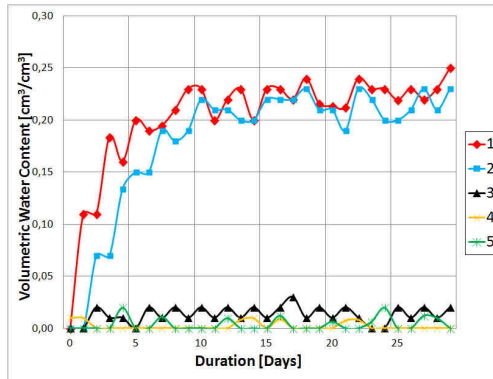


Fig. 5. Model wall made of the standard brick - capillary rise phenomenon determined with capacitive probe

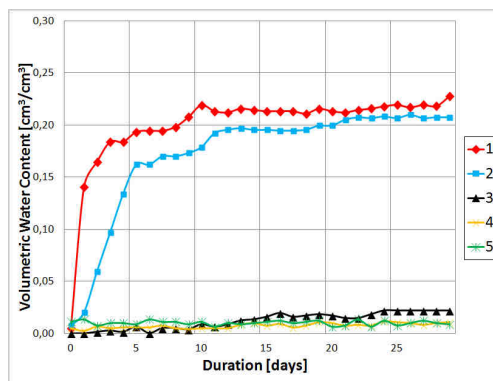


Fig. 6. Model wall made of the standard brick - capillary rise phenomenon determined with surface TDR probe

The conducted research confirmed high absorptivity of water by the red ceramic bricks. At the bottom layer of the bricks, water appeared soon after the experiment had started. Water content quickly rose, which is clearly visible at the lowest bricks. At bricks 2A, 2B and 2C of the second level, water appeared about one or two days later and after 8 days of the process the water content value reached more than 20% vol., which was close to saturation. It must be underlined that no increase in water content was observed at layers 3, 4 and 5. This may be caused by several reasons. One of them is low permeability of cement mortar for water, which prevents water from rising to the higher layers. Another reason for that phenomenon may be the drying process, parallel to the capillary uptake. Compared to the other experiments [35], the model wall was not insulated from external environment, and higher layers of the wall could be not available for water due to its desorption processes.

Data obtained using the TDR technique, compared to capacitive results seems to be more stable. Capacitive readouts are fluctuating for about 4% vol. and the TDR readouts fluctuations do not exceed 2% vol. On the other side, it is possible that the TDR moisture readouts may be underestimated for that material which maximum moisture, according to previous determinations, may reach 25% vol.

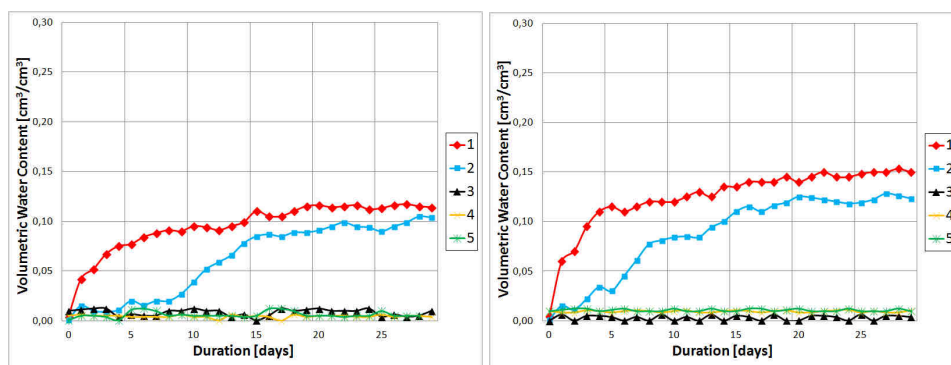


Fig. 7. Model wall made of the standard brick - capillary rise phenomenon determined with surface TDR probe (left - P1, right - P2)

The capillary rise process for the impregnated (P1 and P2) model walls was only examined using the TDR method. Diagrams presented in Figure 7 depict moisture readouts in the particular bricks of the wall. Compared to the capillary uptake processes by the standard bricks there are visible smaller moisture readouts. No increase of moisture was observed above the second level of the bricks. Only bricks 1A, 1B and 2A, 2B and 2C revealed moisture inside their pores. In case of the P1 impregnating agent the maximal moisture increase within the research duration (30 days) achieved only about 10% vol., which was significantly lower than the maximum moisture available for this material. Furthermore, the velocity of moisture increase was lower than in the compared standard brick wall. In case of the model wall impregnated using the P2 product, moisture levels reached by the lowest bricks (1A and 1B) were higher than in the P1 wall and reached about 15% vol., which may mean worse hydrophobic properties. However, it must be stressed, that it is still much less than in the representative standard wall.

Both determinations conducted with the use of the surface TDR probes proved that application of water-based hydrophobic preparations P1 and P2 improved hygric parameters of the model walls and significantly decreased the level of capillary rise phenomenon, which should be considered as positive for renovation processes.

Microstructural analysis using Scanning Electron Microscopy (SEM)

Together with reflectometric determination of the moisture parameters of the measured walls the analysis of the hydrophobic layer continuity was conducted with the use of Scanning Electron Microscopy method (SEM). The research was conducted using FEI Quanta 250 FEG microscope. Low vacuum and low beam energy were applied to avoid the presence of surface defects during the SEM analysis. Resin texture on brick fracture is presented in Figure 8, which is visible in the center and the right photograph. In the left photograph, representing the sample without impregnation, resin structure is not visible.

Macromolecular siloxanes formed a thin and uniform layer in the brick structure. Coating of the methylsilicone resin (preparation P1) is formed by the small globules, whose distribution did not cause suitable sealing effect. This feature of silicone resin guarantees good vapor permeability and hydrophobization effectiveness. Water-based preparation P2 also has formed a visible, uniform coating, which covered the brick structure. Anyhow, the differences between the structure of the standard and the impregnated samples are not significant, which confirms good vapor permeability important for hydrophobization.

It is visible in the Figure 8 that the applied preparations sealed the sub-surface pores, especially P2 preparation. Such structure of silicone gel caused a small porosity decrease which was also confirmed by moisture transport research.

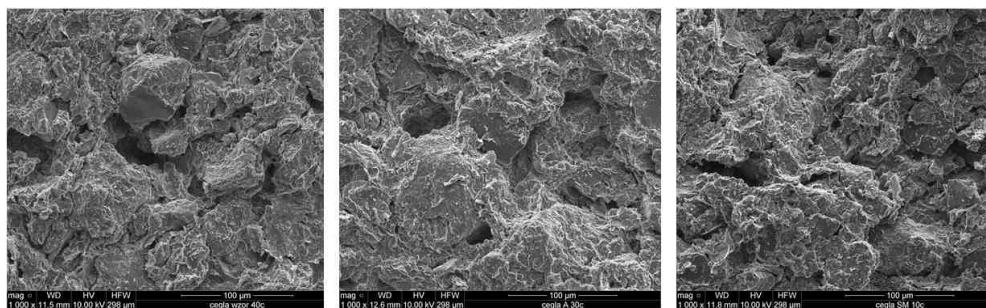


Fig. 8. Water-soluble preparations P1 and P2 in the structure of ceramic bricks with $\times 1000$ enlargement (left - standard, middle - P1, right - P2)

Conclusions

- According to both methods of moisture estimation, water uptake process was confirmed, especially in the bottom layers of the walls, strongly exposed on water presence.

- Research results of hydrophobization efficiency of the model red brick walls revealed the decrease in water absorptivity compared to the non-impregnated brick, which confirms the relevance of hydrophobization.
- The best protection of red ceramic brick against water influence among low VOC water preparations is provided by the macromolecular silicone resins.
- Hydrophobic preparations seal the surface of the porous media, which prevents water vapor diffusion from the materials and is negative for its hygric features. However, the analysis of the polysiloxane gel distribution inside the brick structure proved that water vapor permeability can slightly decrease. This was also revealed by the research presented in [10] article.
- The experiment confirms the potential of the TDR technique for moisture processes monitoring in building materials. The application of the surface TDR probes enabled the quantitative determination of moisture changes in the material, without the necessity of invasive probes installation, which may be successfully used *in-situ* experiments.
- The dispersion of moisture readouts for the capacitive sensor was greater than for the TDR measurements, which can be explained by the salinity influence on the capacitive measurement.

Acknowledgments

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ZABEZPIECZENIA PRZEGRÓD BUDOWLANYCH PRZED WILGOCIĄ WOLNE OD LOTNYCH ZWIĄZKÓW ORGANICZNYCH

Wydział Inżynierii Środowiska, Politechnika Lubelska
Wydział Budownictwa i Architektury, Politechniki Lubelska
Wydział Budowlany, Politechnika Czeska w Pradze

Abstrakt: W artykule przedstawiono parametry materiałów budowlanych, które wpływają na skuteczność stosowania preparatów hydrofobowych. Należą do nich porowatość, nasiąkliwość i stan powierzchni. Podkreślono również ekologiczne aspekty stosowania dostępnych na rynku budowlanym hydrofobowych preparatów wolnych od lotnych związków organicznych. Pierwsza część pracy jest omówieniem problemów wilgotnościowych w przegrodach budowlanych. Duży nacisk położono na elektryczne techniki detekcji wilgoci ze szczególnym uwzględnieniem metody TDR. Druga część ma charakter eksperymentalny. W celu zbadania zjawiska podciągania kapilarnego przygotowano trzy modelowe ścianki z cegły ceramicznej pełnej. Omawiany proces był monitorowany za pomocą czujników pojemnościowych oraz powierzchniowych sond TDR. Uzyskane wyniki pozwalają na śledzenie procesu podciągania kapilarnego w modelowych ściankach z cegły ceramicznej różniących się od siebie rodzajem zastosowanego preparatu hydrofobowego i potwierdzają możliwości sondy powierzchniowej TDR w pomiarach wilgotnościowych murów. Równoległe do badań za pomocą technik elektrycznych wyznaczono podstawowe parametry fizyczne cegły wykorzystanej do wymurowania ścianek, wykonano również zdjęcia za pomocą skaningowego mikroskopu elektronowego (SEM) w celu przeanalizowania ciągłości warstwy hydrofobowej.

Słowa kluczowe: podciąganie kapilarne, cegła ceramiczna pełna, powierzchniowe sondy TDR, hydrofobizacja, wolne od LZO preparaty, lotne związki organiczne, skaningowy mikroskop elektronowy