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The Effectiveness of Hydrophobisation of Porous Building Materials by Using the Polymers and Nanopolymers Solutions

Stanisław Fic
Lublin University of Technology, Faculty of Civil Engineering and Architecture, 40 Nadbystrzycka Str., 20-618 Lublin, Poland
Email: s.fic@pollub.pl

Danuta Barnat-Hunek
Pope John Paul II State School of Higher Education in Biala Podlaska 95/97 Sidorska Str., 21-500 Biala Podlaska, Poland
Email: d.barnat-hunek@pollub.pl

Abstract—**The aim of the study was to assess the possibility of the use of formulations based on hydrophobic organosilicon compounds for the impregnation of porous building materials such as ceramic brick and natural stone - calcareous gaize. The process of the surface hydrophobization was analyzed. The study examined the effectiveness of two formulations differing in the degree of hydrolytic polycondensation, viscosity and concentration, as these are the factors that determine the final effect of hydrophobization. The following laboratory tests were performed: analysis of the physical characteristics of the tested materials, surface absorption, the analysis of characteristics and defects of silica gel nanofilm by using the SEM images.**

Index Terms—**hydrophobisation, ceramic brick, calcareous gaize, polymers and nanopolymers solutions**

I. INTRODUCTION

Polymers and for several years nanopolymers as solutions have been widely used for hydrophobisation of porous building materials. This group of materials includes natural rocks such as limestone, sandstone, and artificial rocks such as ceramic brick, ceramic hollow tile and concrete. Both natural rocks and artificial materials are characterized by a heterogeneous porous structure that determines their strength, durability, water absorption, corrosion resistance, etc. [1]. The structure consists of various kinds of defects in the form of: pores, capillaries, cracks formed in the technological process or formed by the influence of the nature in the natural rocks. The porous structure has the ability to capillary action of water moisture, the preparation of various salts, which results in the rapid destruction of the material.

One of the commonly used methods of protection against the ongoing destruction of building materials is hydrophobization [2]-[6]. In general, hydrophobization is the process of introducing the solution into the porous structure which, when hardened to different degrees, fills voids preventing or restricting the penetration of water or moisture. Organosilicon compounds or nanopolymers based on an aqueous or hydrocarbon solvent are used as the solution in the process of hydrophobization [7]-[10].

The silanes, siloxanes and silicone resins do not differ with each other in an activity, yet in the structure and particle size. The silanes are monomeric compounds, siloxanes - oligomeric, silicone resin - polymeric. The molecular weight of silanes is about 178, siloxanes about 400-600, and polysiloxanes about 2000-3000 [11]. The particle size of the organosilicon compounds affects the depth and speed of penetration into the structure of building materials. The smallest particles have the silicone microemulsions, larger ones - siloxanes. The largest particles have the silicone resins (Formulation A), which are about 100 times larger than the particles of siloxanes [12]. The pore diameters decide on the possibility of a chemical compound adsorption and the size of the critical particle [13] for instance, the critical particle diameter of several compounds: H_2O-0.260nm, C_4H_8-0.510nm, C_6H_6-0.560nm.

Critical diameter of some compounds such as molecular siliconates may be too large to penetrate the fine-pored structure of materials such as gaize. The primary objective of the submitted article is to evaluate the effectiveness of hydrophobization by using two polymer solutions. Moreover, the physical condition of nanofilm in capillaries of porous materials was investigated and analyzed on the basis of calcareous gaize and ceramic brick which are often embedded into the different parts of the building.

The process that precedes the shaping of protective nanofilm is very complex due to the arrangement of solution conformation at the phase boundary. The mechanism of self-assmebly formation of segments and nucleation is described by the thermodynamic Gibbs equation. It is assumed that the conformation of the

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polymer chain is composed of different structures [14], [15]. However, the macromolecule may be located in two energy states: at a solid surface or in the depths of the solution [15]. The conformation of the polymer chain is an important parameter, dependent on the construction of macromolecules [14]-[16]. By [14]-[16], three types of structures are formed by segments of the chain: trains, loops and tails. Each of trains elements has a contact with a surface of solid body - a porous material. Loops elements have only a contact with the solvent, while the tails have the branches from the surface to trains. The segments of the polymer chain are shown schematically in Fig. 1 [17], [18].

The analysis of the surface after the surface glazing, shown in Fig. 1, is included later in this article.

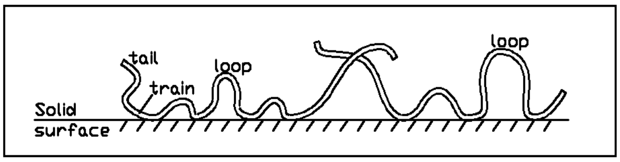

Figure 1. The segment of the polymer chain in the system: tail - train - loop.

Another important parameter describing the interaction of macromolecules in the absorption process in the final result, is the degree of coverage of material with coating, which was considered and analyzed for two types of building materials. This quantitative parameter was used to assess the durability of glazing area and was associated with water absorption. The analysis of the glazing state is useful for building materials while assessing the impact of strain - stress, water absorption, frost resistance, the thermal conduction coefficient λ.

The degree of glazing area P_s, marked as the ratio of the surface which was not glazed (as the surface defect) p_1 to p_2 - unit area expressed in [%].

II. MATERIALS USED IN RESEARCH

Six samples of calcareous gaize (silicate - lime natural stone) and six samples of ceramic brick made of burned clay were chosen to carry out the research. Hydrophobization effect of building materials depends on the structure of the organosilicon compounds, their viscosity and concentration of the active ingredient. The type of solvent and the degree of hydrolytic polycondensation, on which depends the particle size of the resin, decide on the above factors. Consequently, the formulations differing in the degree of hydrolytic polycondensation, viscosity, and concentration were applied to the research. The second criterion was the kind of solvent (carrier) - water and hydrocarbon.

For hydrophobization by using the immersion method, two formulations commonly used in the market were employed:

- Polymer formulation - methyl-silicone resin in the potasium hydroxide based on aqueous solvent (marked A),
- Alkyl-alkoxy-silane fine particle formulation based on hydrocarbon solvent (B).

The first preparation belongs to a group of siliconate that are alkaline, aqueous solutions of sodium and potassium methylsiliconate. Siliconates pass gradually through the condensation process under the influence of carbon dioxide from the air. Together with the increase of molecules, the hydrophobic properties of the material grow.

The second formulation belongs to a group alkyl-alkoxy-silanes which are less active and are more slowly hydrolyzed. Therefore, for a catalyst, pre-hydrolysis and polycondensation are carried out, yet to hydrophobization, solutions of obtaind oligomers are used [19].

Basic characteristics of the materials used in the studies are presented in Table I and II. Viscosity coefficient η was determined by measuring the flow time of solution in an Ostwald viscometer. The study was conducted at ambient temperature of 22.5 ℃. The surface tension was determined by the ascent of liquid in the capillary at the temperature of 22.5 ℃. Consequently, five measurements were taken as meaningful results [1].

TABLE I. AVERAGE BASIC DATA OF GAIZE AND CERAMIC BRICK

Type of material	Bulk density [kg/m^3]	Total porosity [%]	Absorbability [%]	Compressive strength [MPa]
Calcareous gaize	1.45	45	27	20
Ceramic brick	1.69	33.6	12	10

TABLE II. BASIC CHARACTERISTICS OF HYDROPHOBISING FORMULATIONS AND WATER [1]

Type of formulation	Viscosity η [Pa s ·10^{-3}]	Surface tension σ [N/m ·10^{-3}]	The quotient of the surface tension and viscosity σ/η	Density at 20 ℃ [g/cm^3]
A	1.099	67.92	61.73	1.26
B	1.479	23.11	15.65	0.80
Water	0.89	72.00	80.90	0.99

Calcareous gaize, used in the study, is a natural sedimentary rock. The main mineral component of stone is calcite, whose content varies from 45-52.5%, and minerals from the group of silica: opal, chalcedony (39-45%). Some varieties of gaize differ in relation to each other in terms of carbonates, silicates, clay minerals contents and the remains of the organic origin. Silica whose content can be up to 40% SiO_2 occurs in a dispersed form or as an admixture of terrigenous quartz variety, contained in the organic remains and in the binder.

Calcareous gaize was primarily used in parts of the walls of historic buildings. Analyzed ceramic brick is made of light atoms: oxygen, carbon, silicon, aluminum, whose structure is densely packed.

III. SUGGESTION OF THE APPLICATION OF OWN STRUCTURAL MODELS TO THE EXAMINED BUILDING MATERIALS

The presentation of glazing defects can be considered at different levels of structural heterogeneity: from the submicro level in crystals to a macro level, as a more relevant to the heterogeneous construction materials. It is related to the possibility of diffusion, displacement and maintenance of moisture in the material, freezing of water, evaporation, etc. In the building materials different division of pores, capillaries are met, depending on their size. In the study, the following values of pores size were assumed: normal pores with a diameter >10μm; large capillary pores with a diameter of 0.05-10μm; small pores 10-50nm.

In the paper, own models of defects in the form of pores, capillaries, scratches, cracks are subject to analysis. These defects are represented by the wedge model, separated from the volume of material. The modelled wedge is described by using the length of the material "l", the width of the estuary "a" and the angle between the edges "α", as shown in Fig. 2. It is assumed that the pores, capillaries in building materials are small, so that the force of gravity is negligible in comparison with capillary forces and can be ignored especially that gravity is of no importance to the horizontal flow.

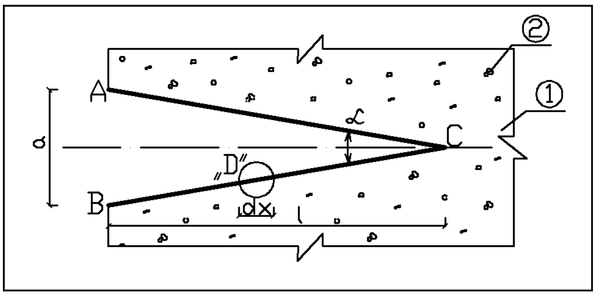

Figure 2. Model of crack (capillary) wedge-shaped filled with gas, 1 - porous material, 2 - pores

For the description of physical model which takes into account the formation of defects to the time of glazing, the following restrictions were assumed:

- In the area of ABC wedge in the initial stage, there is only gas (air),
- Temperature in the solid material and the solution during the hydrophobization process is t=const,
- The load (stresses) of the material are constant F=const,
- The process of hydrophobization is done without the application of pressure,
- The humidity of the material surroundings is below 55%.

In the case of the contact of the polymeric solution (Formulation A or B) with the material under the influence of capillary forces (Fig. 3a), the space of wedge is filled at the time t_1. Subsequently, after some space is filled, the state of equilibrium is followed between the liquid and the imprisoned "gas cushion" that formed around the top. Thus, the balanced system occurs at the equalization of pressure in the solution and in the "gas cushion" (Fig. 3b). The next steps refer to the stabilization of the system, nucleation and evaporation of the solvent from the solution. In the A formulation, the solvent was water, and in the B formulation, the solvent was aliphatic hydrocarbons. The evaporation process continues until t_2 (A formulation) and t_3 (B formulation). Finally, in the capillary, the crystalline nanofilm was formed with amorphous inclusions which include the edges of the crack, as shown in Fig. 3c.

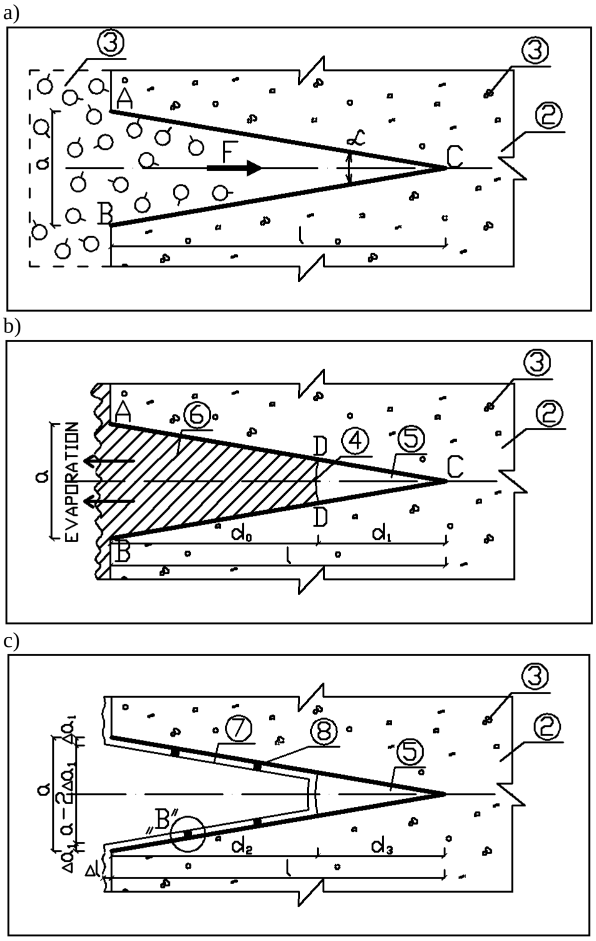

Figure 3. The model of crack filled with polymer (capillary) and formation of polysioloxane gel film: a - influence of capillary forces F and the graduał filling of space, b - equilibrium pressure in the capillary and in the "gas cushion", the arrows show the process of solvent evaporation, c - stage of creation of crystalline nanofilm with amorphous inclusions at the edges of the wedge, d - "B" detail - formation of defects at the surface of nanofilm; 1 - polymer of nanopolymer molecules, 2, 3 - the material and the pores, 4 - meniscus of the solution, 5 - "gas cushion", 6 - solution in the capillary at the phase boundary, 7 - amorphous film surrounding the edges of the crack, 8 - defects arising in hardened nanofilm.

IV. The Analysis of the Results

The results were analyzed on the basis of the assumption that the roughness of the edges in the model segments AC and BC (Fig. 2) are similar to the surface of the material (gaize, brick).

According to Mac Bain [20], [21], mechanical adhesion on the phase boundary between the polymer and solid, causes the penetration of solution into the micropores of the material and creates mechanical anchoring. Roughened surface determines the strength with the ground, and above all, affects the process of self-assembly of molecules in the process of diffusion and adhesion. The distortion of surface makes the process of self-assembly difficult on the rough surface by weakening the viscous force as a result of “hitching” molecules on uneven surface. In the present case, the process of self-assembly in A solution becomes more random in comparison with the solution B which in turn illustrates how the packing occurred, and the final shape of the film on the surface of the solid (Fig. 4). The main reason for this phenomenon can be found in the physico - chemical properties of both solutions A and B i.e. viscosity, surface tension, and the used solvent. A fine particle B formulation more easily and accurately penetrated through rough surface depressions of both gaize and brick than a macromolecular A formulation based on methyl-silicone resin. Fine particle alkyl-alkoxy-silane (B formulation) covered the rough surface with more uniform film which looked like a coating on the surface of gaize and limestone. In comparison with the A formulation, in this case the shape of the film is more uneven with protruding growths, spatial pillars, and spheres (Fig. 4). A possible reason for the formation of such protruding parts was the arrangement of chains in the polymer solution of A formulation, which is schematically illustrated in Fig. 5.

The application of A formulation resulted in the creation of surface defects in the form of gaps in the local coverage area, unfilled pores, and breaks on rough curves. A quantitative analysis of these defects was performed by using the computer software ImageJ 1.46r. Measurements of surface defects were made on a series of SEM images taken for gaize and bricks covered with A and B formulations. In the case of gaize degree of surface coating by using A formulation was 93.7%, and B formulation 96.26%, while surface coating of ceramic brick by using A formulation was 94.95%, B formulation 96.42%.

The measurements of medium-sized microcracks in polysiloxane gel nanofilm in the structure of gaize and brick were also performed, which is shown in Fig. 6.

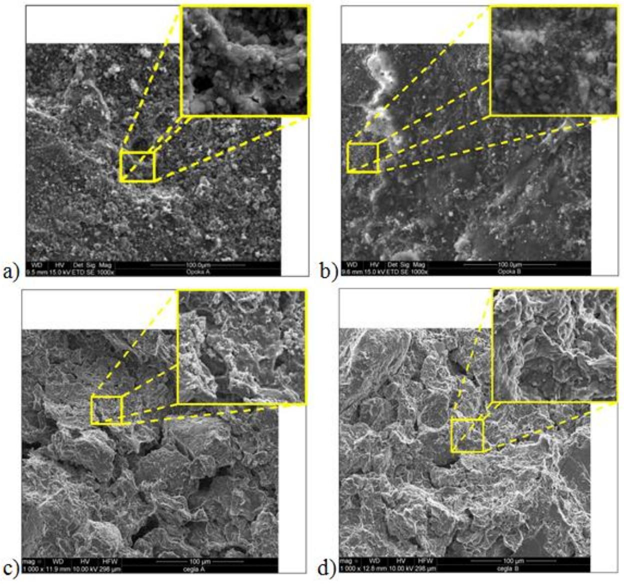

Figure 4. The microstructure of the samples surfaces viewed by SEM; a) calcareous gaize - A formulation; b) calcareous gaize - B formulation; c) ceramic brick - A formulation; d) ceramic brick - B formulation.

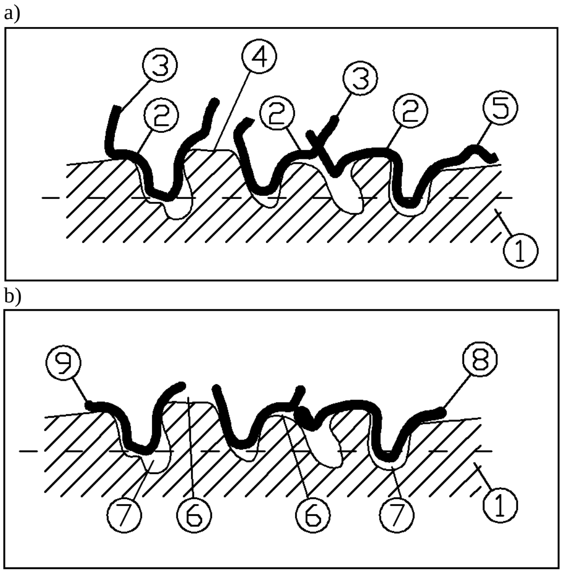

Figure 5. Scheme of discontinuity on the surface of rough gaize or brick in the case of using A formulation: a) polymer chains of A formulation before glazing process, b) scheme of nanofilm creation after glazing process by using A formulation. 1- rough surface; 2- trains; 3-tails; 4- surface not covered with polymer chain; 5- loops; 6- surface defect; 7- trapped air under nanofilm; 8- nanofilm – after glazing; 9-“growths” after glazing.

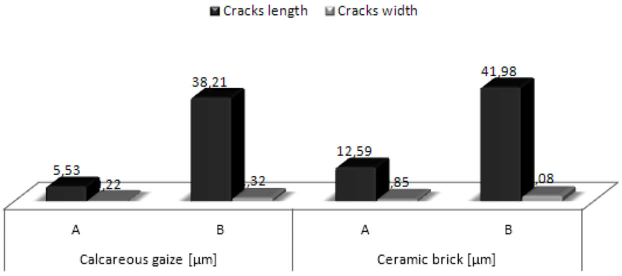

Figure 6. Average size of microcracks on the surface of materials.

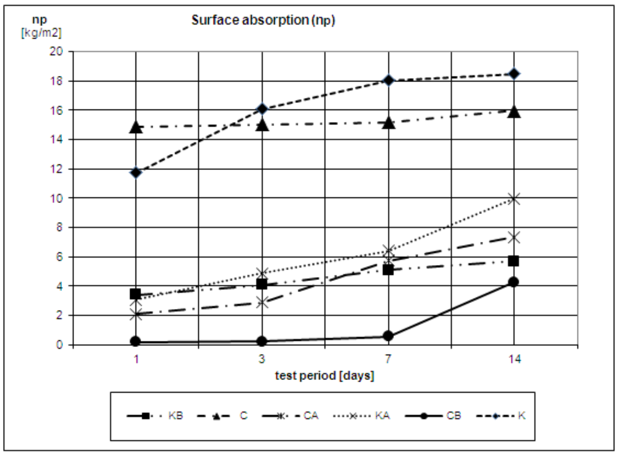

Figure 7. Average water absorption of brick and gaize before and after hydrophobization np [kg/m^2]; K - gaize, KA, KB - gaize with A and B formulation, C - ceramic brick, CA, CB - brick with A and B formulation.

Microcracks measurements showed that the reason for their arising was greater shrinkage deformation which formed in the nanofilm in the case of B formulation with hydrocarbon solvent, as compared to the A formulation with an aqueous solvent. In addition, the impact of the role of defects arising after hydrophobization was assessed in the test materials by measuring the water absorption on the surface.

The analysis of bricks and gaize absorption (Fig. 7) showed that the samples of both materials received the greatest resistance to water by using a dispersion solution of organic oligomers (B). The lowest water absorption after 14 days of the study achieved a brick secured by B formulation ($4.284kg/m^2$) and gaize ($5.68kg/m^2$). Hydrophobization effectiveness was respectively:

- Gaize - A formulation 46%, B formulation 70%;
- Brick - A formulation 54%, B formulation 73%.

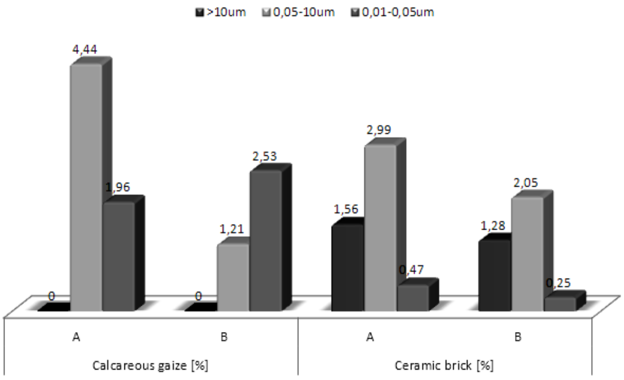

Figure 8. Diameter of defects on the surface of materials. The percentage of pores on the surface of the sample.

V. CONCLUSIONS

As the research and analysis revealed, B formulation, based on gasoline solvent, showed improved surface coverage by far smaller share of the final surface defects. The number of defects when using the A formulation resulted in greater absorption of both materials in comparison with the B formulation. The best effect in securing the ceramic brick and gaize against the penetration of water was obtained by using B formulation based on petroleum solvent which increases the hydrophobicity of the bricks up to 73%, and gaize to 70%. The division of the measured defects on the surface of a film (Fig. 8) indicates the reduction of durability, since the share of pores >10µm, in an amount of 6.4% in the gaize, enables the free flow of water and freezing even at low negative temperatures or the ability to maintain constant water moisture in the volume of material. Similarly, in the case of capillary pores with diameters 0.05-10µm, (5.05% brick) in the defects, it is possible to maintain the free water which is relatively easy moving as a result of capillary action beyond the area of the film, in contrast to the other part of the material, where it was not possible to “access” the hydrophobic formulation. In the defects having a diameter of 10-50nm, there is strong surface tension ($67\text{-}72N/m \cdot 10^{-3}$), and the increase of the adsorption forces manifested by a tendency to condensation. This defect under normal conditions does not cause freezing of the water in them. Moreover, Raised voids of air beneath the surface of nanofilm can cause its destruction by vapor diffusion which is moving from other material volumes. In addition, A formulation based on an aqueous solvent is easily leached from the volume of material, which is confirmed by the studies of cyclic saturation and drying (low cyclic fatigue testing). Hydrophobic formulations based on organic solvents, fine molecule compounds of alkyl-alkoxy-silanes cause the most effective hydrophobization [22]-[25]. For practical reasons, it is recommended to use the B formulation as it is resistant to the external environment (durability of about 10 years), according to a manufacturer. Nevertheless, A formulation was evaluated for a 4 - 5-year period of durability.

This article presents some own research on the effectiveness of hydrophobization - nanofilm created in building materials, such as calcareous gaize and ceramic brick. It is evident that on phase boundary between polymer and solid, the connection and the chemical bonds were significant factors and for this reason they are described in another paper.

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Stanisław Fic. Bachelor of Science and Master of Science from civil engineering at Lublin University of Technology, Poland in 1977. In 1984 he received PhD in the field of Civil Engineering at Warsaw University of Technology in Poland. Title of Associate Professor in 2004 at Odessa State Academy of Civil Engineering and Architecture in Ukraine. He works as an ASSOCIATE PROFESSOR and the HEAD of The Department of General Construction at the Faculty of Civil Engineering and Architecture, Lublin University of Technology in Poland. Since 2010 the HEAD of Institute of Civil Engineering in John Paul II State School of Higher Education in Biala Podlaska, Poland. In 2006-2010 DEPUTY MAYOR of Lublin in Poland. In 1995-2002 VICE DEAN of Engineering Department at Lublin University of Technology. In 1990-1995 a DESIGNER at a private construction design company. In 1989-1990 an EMPLOYEE in "M&M Construction Company" New York, USA. In 1985-1991 a DESIGNER and SUPERVISOR DESIGNER in Lublin state construction design company "Inwestprojekt". He is co-author of the opinions and technical expertise in the field of building materials and construction.

Main publications:

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His area of research includes the use of new building materials and nanocomposites in construction.

He is a member of Polish Union of Building Engineers and Technicians and Polish Academy of Sciences. He received many awards. Award of President of Lublin University of Technology in 2005 and 2013. Personality of the Year 2012 in Poland in the field of engineering innovations - Innovatica 2012 - a statue and certificate by National Chamber of Commerce. The first award of Forbes magazine in Poland 2010 for business activity titled "Lublin miastem przyjaznym dla inwestorów" (The City of Lublin - Investors Friendly City). The leader of Polish ecology 2009 - award of Minister of Environment.

Danuta Barnat-Hunek. Bachelor of Science and Master of Science from Civil Engineering with a specialization in building and engineering structures at the Faculty of Civil Engineering at Lublin University of Technology, Poland in 2000. In 2008 she received PhD in the field of Civil Engineering at Lublin University of Technology in Poland. She works as an ASSISTANT PROFESSOR in the Department of General Construction at the Faculty of Civil Engineering and Architecture, Lublin University of Technology in Poland. Additionally she works as an SENIOR LECTURER in the Institute of Civil Engineering in John Paul II State School of Higher Education in Biala Podlaska, Poland. She is co-author of the opinions and technical expertise in the field of building materials.

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Her area of research includes materials engineering, energy-saving construction, maintenance of historic buildings, methodology in moisture and salinity decommissioning in buildings with particular emphasis on hydrophobization of the walls made of stone.

She received two awards of President of Lublin University of Technology in 2008 and 2011.