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## **ANNEX 2b**

### **Summary of professional accomplishments Description of scientific output and achievements in English**

(Summary of professional accomplishments for the application to initiate habilitation proceedings in the field of technical sciences in Civil Engineering )

Lublin, November 2016

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### 1. Given name and surname

Danuta Barnat-Hunek

### 2. Diplomas and degrees held

5.07.2000 **Master of Engineering**, Lublin University of Technology, Faculty of Civil and Sanitary Engineering, specialization: construction, specializing in building and engineering constructions

Title of master's thesis:

*Project of cafeteria at Construction School Complex in Stalowa Wola"*

Supervisor: Dr.Eng. Andrzej Kowal

8.05.2008 **Doctor of Technical Sciences** in Civil Engineering, Lublin University of Technology, Faculty of Civil and Sanitary Engineering

Title of doctoral dissertation:

*Evaluation of effectiveness of hydrophobising calcareous rock walls on basis of humidity parameter analysis*

Supervisor: Prof. Dr. hab. Eng. Bogusław Szmygin

Reviewers: Prof. Dr. hab. Eng. Jerzy Jasieńko, Prof. Dr. hab. Eng. Stanisław Fic

### 3. Professional record and work in scientific units

#### Employment record in scientific units

1.10.2002 - 30.09.2008 Assistant in the Faculty of Civil Engineering and Architecture of Lublin University of Technology, Institute of Civil Engineering, Division of Construction, and then after reorganization in the Department of Construction

1.10.2008 to present Assistant Professor in the Faculty of Civil Engineering and Architecture of Lublin University of Technology, Division of Construction, and then after reorganization, in the Department of Construction.

26.09.2011 to present Full-time senior lecturer at Pope John Paul II State College in Biała Podlaska, Faculty of Economics and Technology, Department of Technical Sciences, Division of Construction.

#### Professional record

2001 - 2002 PPUP Elektrotechnika in Warsaw, implementation engineer

05.2001 - 12.2001 Lurgi Energie und Entsorgung - Construction of CCGT EC Lublin - Wrotków, position: assistant coordinator of construction works

2004 - 2005 STRIX GP Design Services Office in Lublin, designer (contract for specific work)

2007 - 2014 Center of Municipal Engineering Techniques 'Ekotechnika' in Warsaw, designer (contract for specific work).

#### **4. Indication of achievements under Art. 16 para. 2 of Act of 14 March 2003 on Academic Degrees and Titles and on Degrees and Titles in Art (Journal of Laws No. 65, item. 595, as amended)**

##### **4.1. Attained scientific title**

The basis for applying for the degree of doctor habilitatus in technical sciences in the discipline of Construction is the monograph entitled:

Danuta Barnat-Hunek, „**Surface free energy as a factor affecting hydrophobisation effectiveness in protection of building construction**” Publishing House of Lublin University of Technology, Lublin 2016, IBSN 978-83-7947-216-1, 230 pages.

##### **4.2. Discussion of scientific objective of said work and results achieved, together with discussion of their possible use**

After defending the doctoral dissertation entitled ‘*Evaluation of effectiveness of hydrophobising calcareous rock walls on basis of humidity parameter analysis*’, the motivation for further study was the conclusions derived from the work regarding the insufficient efficacy of hydrophobising stone non-resistant to corrosion, and the premises in literature concerning the lack of effectiveness, or even harmful effects of impregnation, resulting in compounded corrosion. This subject has long been known but not examined in detail, especially in terms of corrosion resistance, and the adhesive properties of hydrophobic coatings (SFE) seemed to be so interesting that the main direction of my scientific research embraced these issues.

Undoubtedly, the information about adhesive properties is one of the bases to predict the resistance of construction materials to moisture, frost and water-soluble salts.

The author is aware that hydrophobisation involves introducing a hydrophobising formulation to the material surface, which penetrates the pores and capillaries but does not form a continuous coating. However, for the purposes of this monograph a modification of the definition of hydrophobisation has been introduced. According to PN-EN 1504-2: 2006, it is not a protective coating, but from the point of view of this work in order to facilitate analysis and discussion of the results, it implies the presence of a hydrophobic coating in the structure of the material subsurface. The term to be used in this work - hydrophobic coating, will mean silane particle clusters or polysiloxane resin which to a lesser or greater degree fill the pores of the material.

In assessing the physicochemical characteristics of solid surfaces, the key parameter is surface free energy (SFE). According to the literature, the interaction between the surface material and the hydrophobic agent affects the degree of adhesion. Depending on the characteristics of the impregnating formulation, one can affect a reduction or increase in SFE, thus the surface tension of

materials, causing their wettability, which is connected with, for instance, resistance to chemical corrosion and frost resistance.

In the literature, an absence of studies has been observed or they are insufficient in relation to predicting or determining hydrophobisation effectiveness, considering the adhesion properties of the construction surfaces.

The phenomenon of interfacial interactions between different liquids and hydrophobic materials, especially the mathematical relationships describing the dependence of these effects are not yet fully known. The various methods of calculating the SFE value of materials based on the measured contact angles have been developed under different assumptions. For these reasons, the SFE values of a given material determined by other methods, and by using different liquids for measurement are not equal.

Verification of the methods for calculating SFE is necessary due to the confirmed significant differences in the obtained results presented in scientific articles. Not every method is suitable for the type of material, and its use causes high uncertainty in the results. Depending on the type of surface layer, in this case hydrophobic, the chemical composition and material structure change, which in turn cause changes in the interfacial interactions, and thereby alter the SFE value calculated by various methods. The determined SFE empirical values are the basis for formulating the relationship between SFE and the results of experimental studies on the physical and mechanical properties.

Based on analysis of the state of the topic, research issues associated with hydrophobising construction surfaces regarding their adhesion properties were formulated. The monograph is supplemented with knowledge in the areas of: the effectiveness of hydrophobising building materials, among others, protection against frost and sulphate corrosion by means of the characteristics of surface free energy as a factor influencing the effectiveness of construction surface hydrophobisation.

**The aim of the study** is to determine the applicability of organosilicon compounds in the renovation of buildings and in newly designed materials aimed at developing substantial resistance of these materials to water and corrosive environments. The studies present the effect of organosilicon compounds on the adhesion properties and corrosion resistance of construction surfaces. An important aim of the research work is to determine the effectiveness of the hydrophobisation of materials exposed to corrosive environments.

The additional objective of the work associated with achieving the scientific objective is to analyze the methods of calculating and determining the SFE of building material surfaces based on the results of contact angle measurements, including SFE occurring at interfaces, with particular emphasis on organosilicon compounds. The aim is to better understand the physical processes at solid-liquid interfaces and select the optimal hydrophobisation methods to be used in protecting building materials against corrosion.

Within the presented problems related to the topic of the monograph, **specific objectives of the work** were formulated:

1. Determine the effect of humidity on the properties of hydrophobised building materials.

2. Evaluate the effectiveness of hydrophobising the surface and subsurface of porous building materials i.e. ceramic bricks, plain concrete, high performance fiber-reinforced concrete, high performance concrete with industrial waste like slag and foundry sand, lightweight aggregate concrete with and without sewage sludge, thermal insulating mortar with lightweight aggregate, zeolite and perlite, including assessment of the effectiveness of surface hydrophobisation of corroded materials from buildings, including industrial ones such as brick and ceramic roofing tile.
3. Assess the resistance to freezing and sulphate crystallization of hydrophobised building materials.
4. Determine the adhesive properties of hydrophobic coatings by determining the surface free energy and work of adhesion, which are factors shaping the effectiveness of hydrophobisation.
5. Evaluate the usefulness of empirical models for determining the surface free energy of hydrophobised building materials.
6. Determine the correlations between the experimental results and the surface free energy determined using empirical methods.
7. Build a logistic regression model to analyze the corrosive factors affecting an increase in the odds ratio of damage or protection of the construction surfaces considered in the work. Calculate the odds ratios of effective protection of construction surfaces by hydrophobising, or to obtain wettability in the initial period of contact with water, in relation to their adhesive properties (SFE).

### **Hydrophobisation effectiveness research program**

The detailed scope of laboratory tests, together with the applied research methodology is described in Chapter 5 of the monograph. In subsections 5.2-5.4, the physical properties of the building materials adopted for testing and further analysis are characterized.

The proposed expanded hydrophobisation effectiveness research program is presented in sections 5.6.1-5.6.9. Selection of the research methodology was dictated by determining the impact of the interactions of the organosilicon compounds not only on the adhesive properties but also on the corrosion resistance of the construction surfaces. Such a choice of investigations enables the author to better grasp the specificity of hydrophobising materials that have been exposed to corrosive environments. In addition, it should be noted that studies on the effectiveness of hydrophobic formulations for protection against corrosion, according to the author's observations, are closely related to the adhesive properties characterized by surface free energy and the work of adhesion, as the factors influencing the effectiveness of hydrophobisation.

Within the presented issues related to the topic of the monograph, the following program of laboratory tests is proposed:

- 1) testing the physical properties of building materials before hydrophobisation

2) testing hydrophobic formulations: viscosity, surface tension

3) hydrophobisation effectiveness testing: water drop absorption rate, surface/mass absorbability, and water vapour diffusion capacity, water vapour permeability of hydrophobic coatings, watertightness, thermal conductivity, frost resistance: loss in mass after frost resistance testing, dynamic modulus before and after frost resistance testing, salt crystallization resistance, compressive strength, static contact angle.

A hydrophobic surface upon contact with a liquid should be impermeable to water and aqueous solutions, while ensuring the porosity and the ability of respiration, i.e. the free evaporation of water contained in the material. Accordingly, firstly tests were carried out on the water drop absorption rate, the absorbability of building materials and additionally concrete watertightness before and after hydrophobisation. In order to verify that the hydrophobisation of a material does not seal the pores and does not disturb the diffusion of gases and liquids, tests were carried out on the water vapour diffusion outflow ability, as well as the vapour permeability of the hydrophobic coatings.

The evaluation of hydrophobising effectiveness was expanded by investigating its effect on the thermal and physical properties of bricks as well as mortar in conditions of high humidity.

Hydrophobisation, causing a decrease in water as well as saline solution absorption, should be one of the elements of proper material protection from frost and salt crystallization. For this reason, tests were carried out on the resistance to freeze-thaw cycles as well as salt crystallization cycles to demonstrate whether hydrophobisation raises frost resistance and resistance to sulphate corrosion.

The use of hydrophobising additives in concrete mixes may change not only the physical parameters of materials such as porosity, absorbability but also the mechanical parameters. For this reason, compressive strength tests of mortar and concrete were proposed, in which hydrophobising additives were used to demonstrate whether hydrophobisation of the mass reduces these parameters, important for these materials.

The assessment of hydrophobisation effectiveness was supplemented by microstructural analyses of the polysiloxane coating on the material surfaces.

Then, based on the static contact angle measurements, the surface free energy as well as work of adhesion were calculated, the methods for calculating surface free energy were compared, a mathematical and experimental model of hydrophobised construction surfaces using Statistica (linear regression model, logistic regression - adjusted odds ratio) was formulated and the work of adhesion was calculated.

### **Research subject**

An assessment of the effectiveness of surface and structural hydrophobisation of porous building materials, i.e. ceramic brick, plain concrete and high performance concrete, high performance fiber-reinforced concrete, high performance concrete with industrial waste like slag and foundry sand, lightweight aggregate concrete with and without sewage sludge, thermal insulating mortar with

lightweight aggregate, perlite, zeolite including evaluation of the effectiveness of hydrophobising corroded materials from buildings, among others, brick and ceramic roofing tile.

The research subject is based on hydrophobic formulations of organosilicon compounds. Classification of the formulations was performed based on literature and the author's own research.

The effectiveness of the construction surface hydrophobisation depends on the construction of the organosilicon compounds, their viscosity and active ingredient concentration.

Hydrophobising formulations with the following abbreviations used in the work qualified for surface hydrophobisation testing:

– formulations with an aqueous solvent:

ZMW – 20% methyl silicone resin solution in potassium hydroxide (1:6)

KK – a hydrophobic silicic acid compound

– formulations with an organic solvent:

AAS – alkyl alkoxy siloxane, of which the carrier is aliphatic hydrocarbons,  $R_n\text{-Si- (OR) }_{4-n}$ , where:  $R_n$  - derivative of alkyl alkoxy siloxane

ZMO – methyl silicone resin in an organic solvent (petroleum spirit),  $R\text{-O-Si- [O-Si] }_n\text{-O-Si-R}$ , where R - methyl derivative

Two types of hydrophobisation were applied:

– surface (double-brushing, or by dipping the material in the formulation for 10 sec)

– in mass (use of the formulation as an additive to mortar and concrete).

For structural hydrophobisation testing, two hydrophobising additives were selected - in the form of powders and emulsions. The materials used in the mass are indicated in the work as DC and DE, used for hydrophobising structural concrete and mortar.

### **Test and analysis results**

Testing of the hydrophobising efficacy of porous construction materials has been presented in detail in sections. 5.6.1-5.6.10 of the monograph. In Section 4 of this summary of professional accomplishments, only general conclusions of these studies are provided since the main purpose of the monograph was to determine the surface free energy.

In this summary of professional accomplishments, the adhesive properties of hydrophobic coatings by determining the surface free energy and work of adhesion, which are factors influencing the effectiveness of hydrophobisation, are presented.

### **Surface free energy**

Based on the measured contact angles, SFE was calculated using various calculation methods, namely Owens-Wendt, Neumann, Wu and Fowkes. On this basis, the author has shown that by using the selected methods, conflicting results are obtained.

Therefore, the author assessed the usefulness of the selected empirical models for determining the SFE of hydrophobised porous building materials. It is assumed, supported by statistical analyzes, that



the SFE of building materials can be reliably estimated using a method that gives results closest to the arithmetic mean of the results obtained by the Owens-Wendt, Neumann, Wu and Fowkes methods.

Table 4.1 presents the state equations and used measuring liquids for the four selected methods employed in the work to calculate SFE.

**Table 4.1.** State equations used to calculate SFE

Method	State equation	Liquid properties	Used liquid
Neumann	$\gamma_s = \frac{\gamma_s^d \gamma_l^d + \gamma_s^p \gamma_l^p}{\gamma_l}$	One liquid with a high polar component	Distilled water
Fowkes	$\gamma_s = \frac{\gamma_s^d \gamma_l^d + \gamma_s^p \gamma_l^p}{\gamma_l}$	One liquid with a high polar component and one liquid component with a high dispersive content	Distilled water, diiodomethane
Owens – Wendt	Dispersion component of SFE $\gamma_s^d = \frac{\gamma_s(\cos\theta_g + 1) - \gamma_l(\cos\theta_w + 1) \sqrt{\frac{\gamma_g^d}{\gamma_w^d}}}{2 \left( \sqrt{\frac{\gamma_g^d}{\gamma_s}} + \sqrt{\frac{\gamma_w^d}{\gamma_s}} \right)}$ Polar component of SFE $\gamma_s^p = \frac{\gamma_s(\cos\theta_g - 1) + \gamma_l(\cos\theta_w - 1) \sqrt{\frac{\gamma_g^d}{\gamma_w^d}}}{2 \left( \sqrt{\frac{\gamma_g^d}{\gamma_s}} - \sqrt{\frac{\gamma_w^d}{\gamma_s}} \right)}$	Two liquids with a high polar component	Distilled water, glycerin
Wu	$\gamma_s(\cos\theta) = \frac{\gamma_s^d \gamma_l^d + \gamma_s^p \gamma_l^p}{\gamma_l}$	Two liquids with a high polar component	Distilled water, glycerin

The following test results were obtained:

- ceramic brick

For standard samples, the SFE value (Wu method) was the highest and at the beginning of the study amounted to 94.43 mJ/m<sup>2</sup>. This demonstrates the high wettability of the bricks. The lowest SFE value was obtained by the AAS formulation - 19.58 mJ/m<sup>2</sup>. After 40 minutes, the hydrophobicity decreased by 7-15%, while the reference brick was completely hydrophilic. The SFE calculated by the Fowkes method was for all the measurements lower than the SFE determined by Wu. The differences are justified by the use of various measuring liquids as glycerin and diiodomethane, resulting in the different contact angles of these liquids.

- lightweight aggregate concrete

The smallest SFE value (weakest adhesion properties) was obtained by the lightweight aggregate concrete hydrophobised with alkyl alkoxy siloxane in an organic solvent (16.23 mJ/m<sup>2</sup>). Moreover, it can be seen that in all the cases, the dispersive component of SFE (15.45-127.49 mJ/m<sup>2</sup>) is by far the larger share of the total  $\gamma_s$  value than the  $\gamma_s^p$  polar component (0.05-4.17 mJ/m<sup>2</sup>).

Considering the changes in time, it was noted that with the passage of time (40 minutes), the  $\gamma_s^d$  component dispersion value and the total SFE value increase by 11.5% for the ZMO formulation, 44% - ZMW, 120% - AAS. Lightweight aggregate concrete without hydrophobisation has a very high SFE

value, which indicates high adhesive properties and lack of resistance against corrosive compounds with water in the porous structure of the material. The introduction of organosilicon compounds to the subsurface zone of the concrete causes, depending on the formulation chemical structure, a reduction in SFE and surface tension of concrete. This has an impact on reducing the penetration of corrosive substances into the concrete structure and thus affects its durability.

*- concrete with industrial waste*

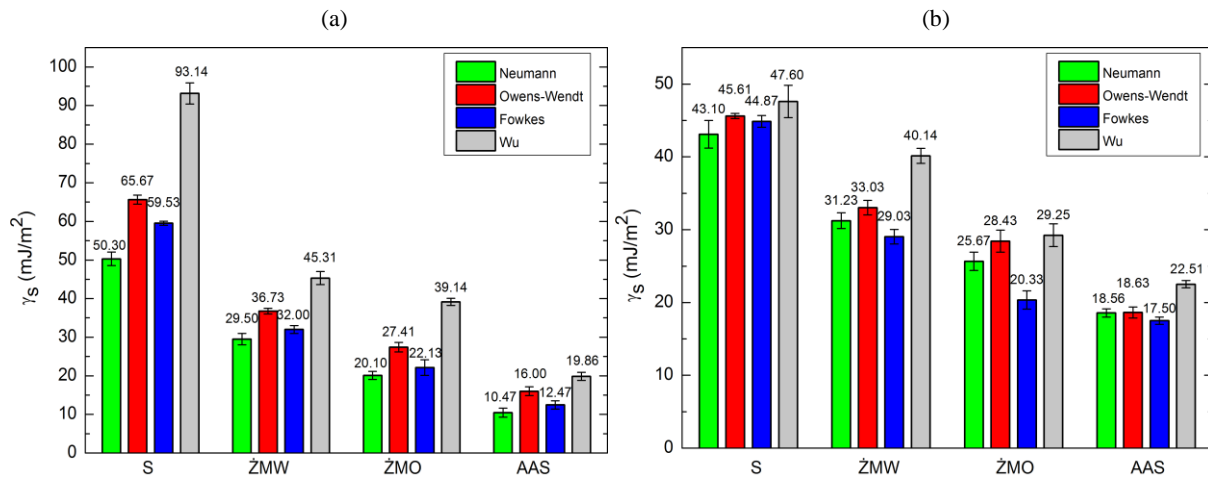
The SFE values are comparable irrespective of the test duration. The lowest SFE values  $\gamma_s = 62.35$  mJ/m<sup>2</sup> were obtained for the concrete without waste, which means the weakest adhesion properties of the investigated concretes. All the concrete surfaces were characterized by high wettability and good adhesion properties. At the beginning of the study, the SFE of the reference concrete was lower by 9.3% than the highest SFE value of the concrete containing 30% slag. The SFE values result from different concrete porosities, depending on the content of slag and sand from foundry processes.

*- ultra-high performance concrete with or without steel and polypropylene fibers*

The SFE calculations carried out for the seven types of UHP concrete with fibers constituted an extensive and substantial part of the work. The SFE value is highest and amounts to 76.23 mJ/m<sup>2</sup> for SPC1 (granodiorite aggregate) and 75.16 mJ/m<sup>2</sup> for SPC2 (granite aggregate) in the case of non-hydrophobised concrete. With respect to non-hydrophobised concrete, the SFE value was 5.5 times higher in the case of the granodiorite aggregate concrete (C1) and 3.7-fold higher for the granite aggregate concrete (C2) than that of the same after impregnation. The lowest SFE values  $\gamma_s = 11.02$  mJ/m<sup>2</sup> (by the Owens-Wendt method) were obtained for the C1 concrete hydrophobised with alkyl alkoxy siloxane AAS. Reference concrete (C2) attained the highest hydrophobicity with the methyl silicone resin organic solution ( $\gamma_s = 12.5$  mJ/m<sup>2</sup>). The concrete with the highest content of polypropylene fibers (PC) was characterized by about 20% higher SFE values as compared to the concrete without the fibers (C2). This indicates its higher wettability, which was confirmed by absorbability tests. A similar dependence can be observed in the tests of concrete with steel fibers. Depending on the method used, the SFE of concrete (C1) is 24% higher than the SFE of concrete (SC) with 1% steel fibers.

Considering changes over time, it was observed that with the passage of time (after 5 and 35 minutes) the SFE value increases. In the next part, the impact of 180 freeze-thaw cycles on the SFE value of standard concretes was examined. After the frost resistance test, all the SFE values were higher than prior to testing, up to 15% (SC). The results were interpreted based on SFE before and after the freeze-thaw cycles.

Subsequently, four methods for calculating SFE were compared in the work. In order to more accurately illustrate the occurring differences in the results obtained by the particular methods, Fig. 4.1 shows the SFE results for the various materials considered in the work.



**Fig. 4.1.** Comparison of SFE of ceramic bricks and high performance concrete with steel fibers before and after hydrophobisation (a) ceramic brick, (b) SC concrete

It was found that different results were obtained using the various calculation methods. The results obtained by means of the method based on liquid vapour depends on the nature of the liquid. By using two liquids, the SFE values are higher by about 7-20% than in the Neumann method using a single liquid. This involves appropriate selection of the second measuring liquid because the angles between the material and the second liquid have a significant impact on the SFE. In all the calculated cases in the work, the maximum SFE was obtained using the Wu method (up to 35%, and in the case of standard bricks 45%). The calculated SFE values are different and depend on the calculation method and the type of measuring liquid.

In order to reach conclusions regarding selection of the most reliable methods for calculating the SFE of porous building materials before and after hydrophobisation, the author compared (Tab. 4.2-4.3) the deviations from the mean SFE value, the average deviation and standard deviation calculated for the four methods adopted in the work with each other. The SFE of the selected materials, i.e. UHPC with/without steel fibers and/or polypropylene fibers, ceramic brick and lightweight aggregate concrete, were analyzed.

Analyses of the average deviation from the mean SFE and standard deviation confirm that the SFE results obtained using the Wu method deviate significantly statistically from the average obtained by the other methods. In the case of the Wu method, the average deviation is over 4 times greater than in the Owens-Wendt method in the case of UHPC and 13 times higher in the case of lightweight aggregate concrete, and 10 times more regarding ceramic bricks. The most reliable results (the lowest mean and standard deviation) for all the analyzed materials were obtained using the Owens-Wendt method. The accuracy is similar to some of the tests described in the literature. The Owens-Wendt method is the most recommended method for calculating the SFE of polymer surfaces and plastics. The analyses performed in the work confirmed the possibility of using this method in the case of porous building materials before and after hydrophobisation.

**Table 4.2.** Deviation from average SFE value of UHPC

Concrete type	Formulation type	Mean SFE [mJ/m <sup>2</sup> ]	Deviation from mean [%]			
			SFE calculation method			
			Neumann	Owens-Wendt	Fowkes	Wu
C1	S	50.09	8.96	6.98	7.76	23.70
	ZMW	33.67	1.11	4.73	14.17	20.01
	AAS	17.43	13.32	2.37	6.50	22.19
	ZMO	12.64	3.21	2.57	4.23	4.87
SC	S	45.21	4.68	0.71	0.46	4.43
	ZMW	31.87	0.54	3.85	8.70	5.39
	AAS	19.30	3.76	3.71	9.30	16.77
	ZMO	25.76	0.94	9.81	20.43	11.56
SPC1	S	69.12	13.63	2.65	0.69	10.29
	ZMW	33.37	0.58	1.24	14.30	16.11
	AAS	17.86	1.96	2.63	8.17	12.77
	ZMO	35.34	3.09	0.67	9.46	11.88
SPC2	S	66.02	11.97	0.44	2.31	13.84
	ZMW	31.71	1.85	1.66	10.75	14.26
	AAS	22.81	7.87	3.66	0.90	5.11
	ZMO	38.57	4.71	1.21	6.13	12.04
SPC3	S	56.10	9.91	0.23	3.03	12.71
	ZMW	28.39	3.14	3.43	4.20	10.77
	AAS	16.24	6.60	2.69	2.72	6.63
	ZMO	39.16	7.57	4.28	2.18	5.48
PC	S	61.94	10.49	0.36	5.07	15.21
	ZMW	23.86	5.09	1.78	3.58	10.46
	AAS	20.24	2.71	1.82	6.11	10.64
	ZMO	22.33	1.59	1.23	9.97	10.32
C2	S	54.77	5.45	4.68	3.96	4.73
	ZMW	22.57	4.50	3.08	3.83	11.41
	AAS	15.39	11.80	2.83	1.20	10.17
	ZMO	13.92	1.72	1.58	10.20	10.34
Mean [%]			<b>5.46</b>	<b>2.75</b>	<b>6.44</b>	<b>11.57</b>
Standard deviation s [%]			<b>4.08</b>	<b>2.10</b>	<b>4.75</b>	<b>5.10</b>

An equally accurate method of calculating the SFE of high performance concrete is the Neumann method (the average deviation was 5.46%, standard deviation 4.08%). For materials of a higher porosity than high performance fiber-reinforced concrete, such as ceramic brick and lightweight aggregate concrete, according to the analyzes the second valid and accurate method for calculating SFE is the Fowkes method. However, in the case of lightweight aggregate concrete the standard deviation was higher in this method than in the Neumann method. The major advantage of the Neumann method is the possibility of using only one liquid, for example distilled water to determine the angle. This significantly reduces possibility of errors related to experimental studies providing input for further calculations.

**Table 4.3.** Deviation from average SFE value ceramic bricks and expanded lightweight aggregate concrete

Concrete type	Formula-tion type	Mean SFE [mJ/m <sup>2</sup> ]	Deviation from mean [%]			
			SFE calculation method			
			Neumann	Owens-Wendt	Fowkes	Wu
Ceramic brick	S	67.16	25.10	2.21	11.36	38.71
	ZMW	35.89	17.80	2.43	10.83	26.32
	AAS	14.70	28.80	8.85	15.17	35.11
	ZMO	27.20	26.10	0.80	18.62	43.90
Mean [%]			<b>24.45</b>	<b>3.57</b>	<b>14.00</b>	<b>36.01</b>
Standard deviation s [%]			<b>4.70</b>	<b>3.59</b>	<b>3.69</b>	<b>7.40</b>
BK1	S	107.97	6.08	0.18	5.03	11.41
	ZMW	69.68	3.85	1.49	0.98	3.33
	AAS	16.23	5.74	0.02	5.13	13.15
	ZMO	81.29	2.82	0.16	1.59	4.56
BK2	S	128.01	2.35	0.02	0.79	3.12
	ZMW	32.04	6.34	0.37	3.25	9.24
	AAS	33.27	7.11	3.02	7.14	19.47
	ZMO	57.26	5.69	0.40	3.94	10.03
Mean [%]			<b>5.01</b>	<b>0.71</b>	<b>3.48</b>	<b>9.29</b>
Standard deviation s [%]			<b>1.75</b>	<b>1.05</b>	<b>2.26</b>	<b>5.60</b>

The author has noted that errors are possible not only during laboratory measurements of the contact angle with a second measuring liquid, but also at the stage of adopting the data for calculations. Namely, different values for measuring liquids and their components can be found in the literature. Various SFE component values can be found not only for water but also other liquids, for example, diiodomethane or glycerin adopted in the work. The author broadly addressed this issue in Subsection 2.3.2 of the monograph. It should be noted that this has an influence on obtaining various numerical values when calculating the SFE of material surfaces. This is another reason for the discrepancy of calculation results when using several methods and various measuring liquids.

In the case of the Fowkes method, the results are similar to the those obtained using the Owens-Wendt method on the surfaces of ceramic bricks and lightweight aggregate concrete, as in the mathematical sense, these methods are similar but the course of calculating SFE is different. It should be noted for safety reasons that diiodomethane is used as the dispersion liquid to measure the contact angle in the Fowkes method. It is a poisonous toxic substance and specific protection measures should be adopted in contact with it. This fact to some extent supports the use of equally accurate methods, but using safe, commonly available liquids like water or glycerin.

Regarding the largest discrepancies obtained by the Wu method, it should be recalled that it uses a complicated method of harmonic mean using two highly polar component liquids such as water and glycerin. The equation derived by Wu takes the form of an equation with four unknowns, which is usually solved numerically. Due to its very complex and complicated character, the Wu concept did not play a special role in the development of research on the wettability and SFE of polymeric materials. The author of the work confirmed the lack of usefulness of this method in measuring the SFE of porous building materials due to the too large deviation from average SFE values.

An even more difficult situation can be encountered if the surface of the material is modified with a chemical substance, in this case a hydrophobic formulation. Depending on the chemical composition modification, surface layer texture or roughness, it causes changes in interfacial interactions. This is crucial when calculating the SFE value using various methods. For these reasons, the subject of the comparative analysis of SFE values may be only the results obtained by means of the same method and using the same measuring liquids.

Based on the conducted calculations and analyses in the work as well as literature analyses, for further consideration the Owens-Wendt method which provides the most credible SFE results of hydrophobised building materials was adopted.

The determined empirical SFE values are the basis for formulating the relationship between SFE and the experimental results, which are presented in detail in Section 5.8 of the monograph.

### **Formulation of mathematical and experimental model of hydrophobised construction surfaces**

#### **Linear regression models with one or two output variables**

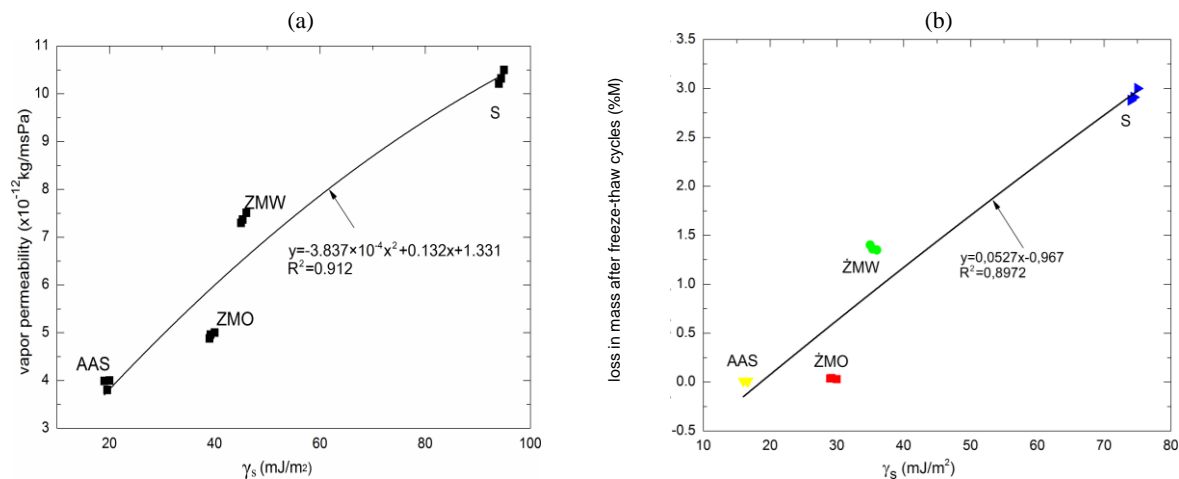
Due to the greater possibility of using the SFE calculation results obtained by the Owens-Wendt method, mathematical and experimental models with one input variable were designated. These models in the present work are set in the form of quadratic functions. In formulating the mathematical and experimental models, the time factor was not considered, therefore these models are static. The quality of matching the models to the experimental results was assessed by determining the  $R^2$  determination coefficients. The high values of this ratio confirm the very good match of polynomial functions to the experimental data. The SFE value is one of the most important factors that determines the adhesion properties of the material before and after hydrophobisation, as well as being a factor that determines the effectiveness of hydrophobisation in the protection of construction surfaces against corrosion.

First, the simplest mathematical and experimental models with one input variable are set - the surface free energy. The relations of the physical characteristics of the construction materials to their adhesion properties (SFE) are presented.

The impact of SFE on the water vapour permeability of ceramic brick was observed. Fig. 4.2a presents the application of the Owens-Wendt method to determine the second-degree curve, which describes the dependence of water vapour permeability on surface free energy. The high determination coefficient  $R^2 = 0.912$  confirms the existence of a strong relationship between these properties.

The results are clearly grouped according to the type of hydrophobisation. The highest values of SFE and water vapour permeability were obtained by the S bricks, which means good wettability and water vapour diffusion of the material. Differing properties were obtained for the bricks hydrophobised with alkyl alkoxy silanes. In this case, the ratio of SFE to water vapour permeability is almost three times smaller than that of the standard bricks.

In the work, it was demonstrated that SFE is in close relationships with other physical properties of ceramic bricks before and after hydrophobisation. One example of such a correlation is the linear function of SFE calculated with the Owens-Wendt method and the loss in mass after freeze-thaw cycles (Fig. 4.2b). In this case, the results are also grouped according to the type of hydrophobisation. The best protection against the influence of freeze-thaw cycles was assured by the alkyl alkoxy siloxane ceramic surface.



**Fig. 4.2.** Relationship between SFE and water vapour permeability (a) and loss in mass after freeze-thaw cycles, (b) ceramic brick

Table 4.4 lists the mathematical and experimental models with one input variable – the SFE of bricks before and after hydrophobisation.

**Table 4.4.** Mathematical and experimental model of reference and hydrophobised bricks

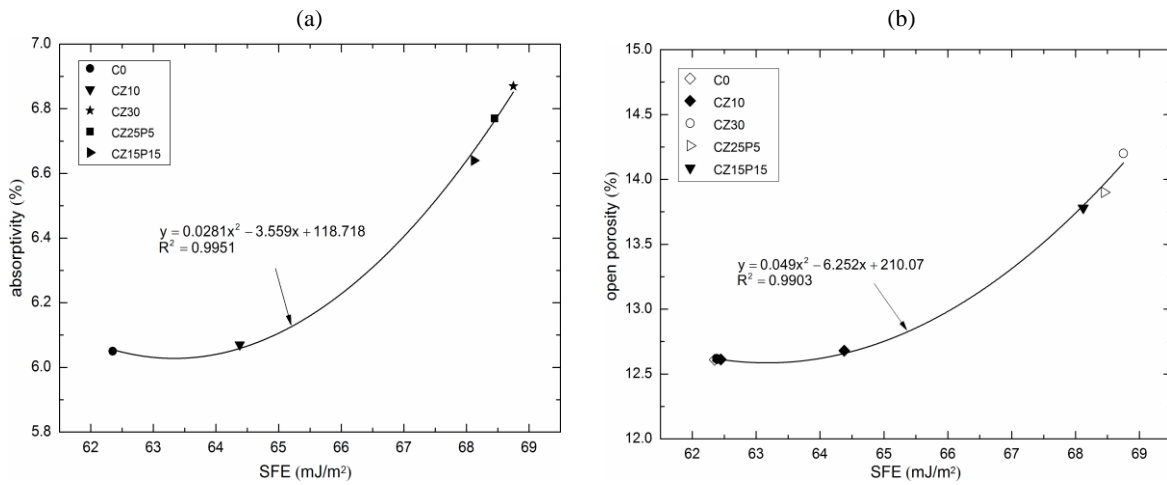
Property	Dependence on one empirical variable $x - \gamma_s$	Coefficient of determination $R^2$
Absorbability [%]	$y = -0.0175x^2 + 3.299x - 48.1954$	0.8896
Vapour permeability [ $x10^{-12}kg/msPa$ ]	$y = 3.837x10^{-4}x^2 + 0.132x + 1.331$	0.9120
Loss in mass after frost resistance test [%]	$y = 0.0527x - 0.967$	0.8972
Loss in mass after salt crystallization test [%]	$y = 0.0524x - 1.2112$	0.9260

In all the analyzed scenarios, a significant impact of the hydrophobisation itself and the type of formulation on the physical properties of bricks was observed. In each case, the lowest SFE values and the lowest absorbability, water vapour permeability, loss in mass after frost resistance and salt crystallization testing was obtained by brick hydrophobised with alkyl alkoxy silane (AAS). The highest values of all the parameters considered in the work were obtained by standard bricks. The adhesive properties of the bricks remain in close correlation with the above-mentioned physical characteristics, as evidenced by the high coefficients of determination  $R^2$ .

Other building materials analyzed in the work also are characterized by strict relations between SFE and their physical parameters.

For five of the analyzed high performance concretes with waste, the correlation between the total SFE value and absorbability and open porosity is presented. Fig. 4.3 shows the application of the

Owens-Wendt method to calculate the curves describing the dependence of absorbability and open porosity on the SFE value.

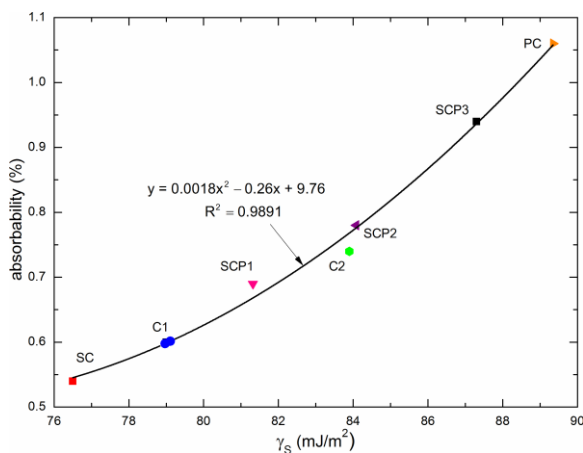


**Fig. 4.3.** Relation between absorbability and SFE (a) and open porosity (b) of concretes

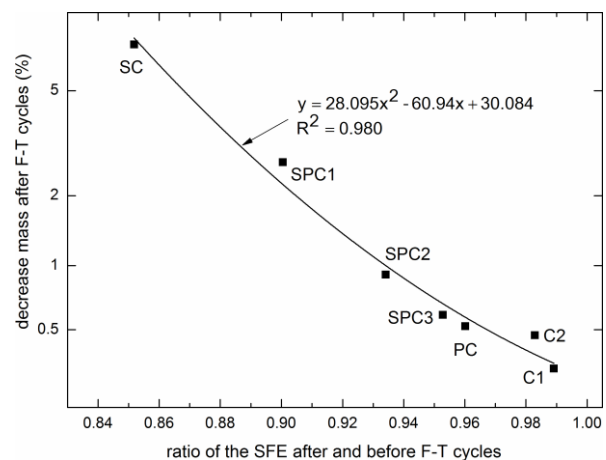
The highest values SFE, absorbability and open porosity were obtained by concrete CZ30 with the highest boiler slag content, while concrete C0 without waste has the lowest values of the mentioned parameters. It was found that the SFE value corresponds exactly to the absorbability and porosity, regardless of the test duration. Functions of the second degree are characterized by good matching as the correlation coefficient in the first case amounts to 0.9951 (Fig. 4.3a), and 0.9903 in the second (Fig. 4.3b) and relatively low deviations from the curve.

Fig. 4.4 shows the relationship between absorbability and SFE values calculated using the Owens-Wendt method for all the tested standard UHP fiber-reinforced concretes.

The highest SFE and absorbability values were obtained by concrete PC with the highest content of polypropylene fibers, while the lowest SFE values and absorbability were achieved by concrete SC with the highest steel fiber content. It was found that the SFE value corresponds exactly to the absorbability, regardless of the test duration. The second-degree polynomial obtained by the Owens-Wendt method has a very good coefficient of determination  $R^2 = 0.9891$ .



**Fig. 4.4.** Relation between SFE and absorbability of UHP fiber-reinforced concrete



**Fig. 4.5.** Relation between loss in mass and SFE index before and after freeze-thaw cycles

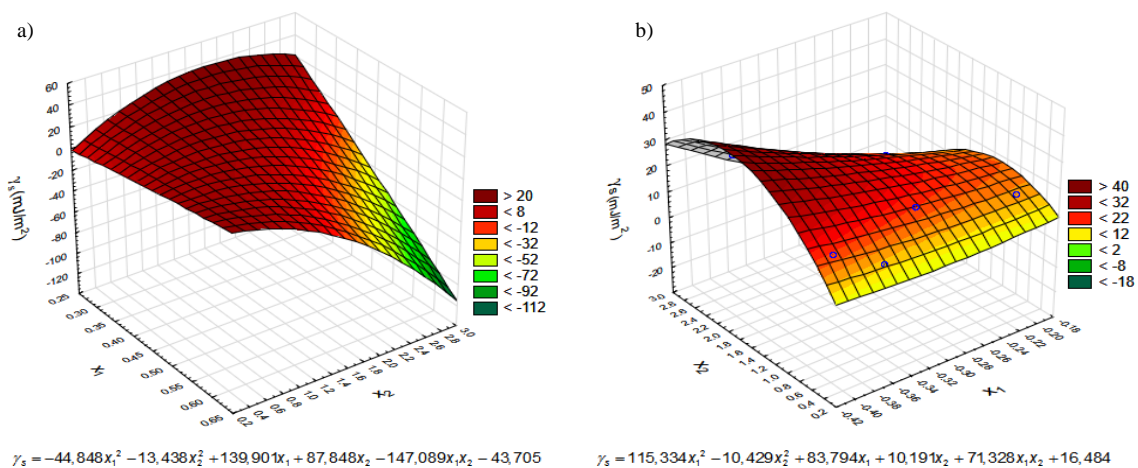


For all the UHP fiber-reinforced concretes, the correlation between the loss in mass and the SFE index before and after the frost resistance test was determined (Fig. 4.5). This relationship can be described by the equation  $y = 28.095x^2 - 60.94x + 30.084$ , which is characterized by an excellent coefficient of determination  $R^2 = 0.98$ . The results for the concrete with 1% steel fibers (SC) were significantly different from the other results. This concrete has the highest loss in mass in the frost resistance test and the highest difference between SFE before and after the test. It was found that the steel fibers have an adverse effect on the corrosion resistance, whereas the concrete without fiber has the highest resistance to frost.

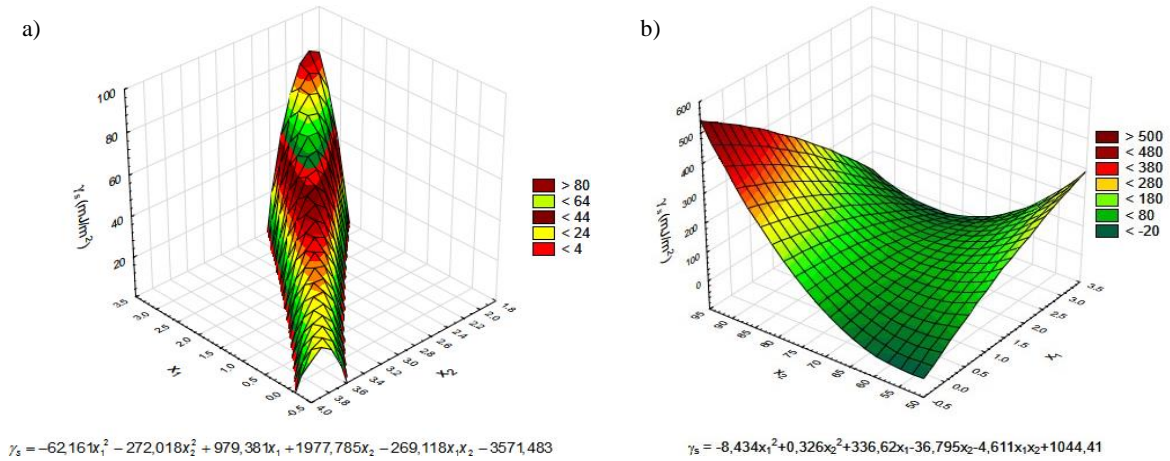
Simple mathematical and experimental models with one input variable confirmed that SFE can be a measure of various physical characteristics of building materials, such as a measure of porosity, wettability and frost resistance of high performance concrete with waste materials such as slag and foundry sand or high performance fiber-reinforced concretes with varying fiber content. The existence of such a strong relationship between these characteristics will eliminate additional long-term laboratory studies on absorbability, porosity, and especially frost resistance.

In the following part of the paper, a more complex mathematical and experimental model with two input variables is proposed. The second-degree model function was determined by means of the Statistica software method of least squares. Complex models are aimed at better understanding the adhesion properties of the material after hydrophobisation and its impact on the physical characteristics of the material.

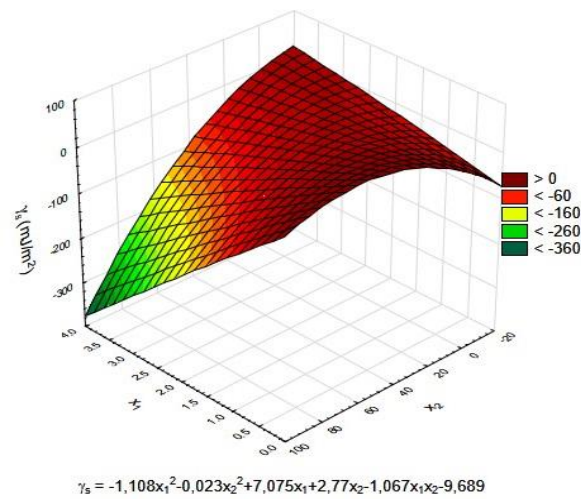
The models presented below (Table 4.5 and Figs 4.6-7.9) represent to what extent the properties of the material affect the adhesive properties and thus the wettability of the surface, which indirectly defines the corrosion resistance of the material.



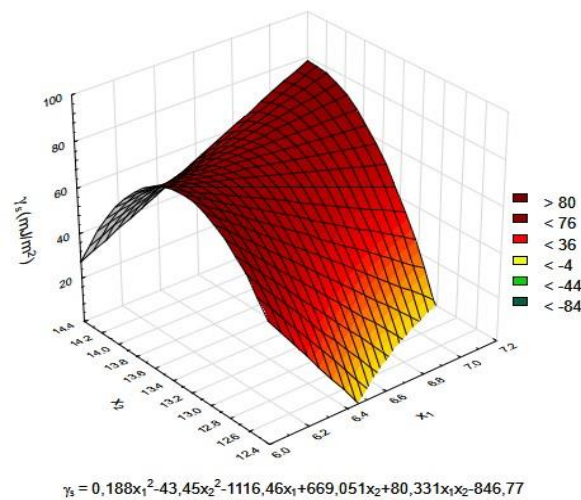
**Fig. 4.6.** SFE dependence on: (a) absorbability and frost resistance, (b) resistance to salt crystallization and frost resistance of concretes hydrophobised with AAS



**Fig. 4.7.** SFE dependence on: (a) loss in mass and dynamic modulus after frost resistance test (b) loss in mass after salt crystallization test and rate of water vapour diffusion after 7 days, of bricks before and after hydrophobisation



**Fig. 4.8.** SFE dependence on loss in mass after frost resistance and absorbability tests of hydrophobised lightweight aggregate concrete BK1 (ZMW, ZMO, AAS)



**Fig. 4.9.** SFE dependence on absorbability and porosity of reference concretes with waste (C0-CZ15P15)

**Table 4.5.** *Mathematical and experimental model with two reference and hydrophobised construction surface input variables*

Material	SFE dependence ( $\gamma_s$ ) on two input variables
	$x_1$ - absorbability, $x_2$ - loss in mass after frost resistance
UHPC - S	$\gamma_s = 84.977x_1^2 - 11.606x_2^2 - 114.725x_1 + 74.009x_2 - 47.304x_1x_2 + 71.749$
UHPC - AAS	$\gamma_s = -44.848x_1^2 - 13.438x_2^2 + 139.901x_1 + 87.848x_2 - 147.089x_1x_2 - 43.705$
	$x_1$ - loss in mass after salt crystallization, $x_2$ - loss in mass after frost resistance
UHPC - S	$\gamma_s = -1139.508x_1^2 - 0.3405x_2^2 + 139.281x_1 + 14.09x_2 + 71.69x_1x_2 + 38.609$
UHPC - AAS	$\gamma_s = 115.334x_1^2 - 10.429x_2^2 + 83.794x_1 + 10.191x_2 + 71.328x_1x_2 + 16.484$
	$x_1$ - loss in mass after frost resistance, $x_2$ - dynamic modulus after frost resistance test
Brick	$\gamma_s = -62.16x_1^2 - 272.02x_2^2 + 979.38x_1 + 1977.78x_2 - 269.12x_1x_2 - 3571.48$
	$x_1$ - loss in mass after salt crystallization, $x_2$ - rate of water vapour diffusion
Brick	$\gamma_s = -8.434x_1^2 + 0.326x_2^2 + 336.62x_1 - 36.795x_2 - 4.611x_1x_2 + 1044.41$
	$x_1$ - loss in mass after frost resistance, $x_2$ - absorbability
BK1	$\gamma_s = -1.108x_1^2 - 0.023x_2^2 + 7.075x_1 + 2.77x_2 - 1.067x_1x_2 - 9.689$
	$x_1$ - absorbability, $x_2$ - porosity
Concrete with waste	$\gamma_s = 0.188x_1^2 - 43.45x_2^2 - 1116.46x_1 + 669.051x_2 + 80.331x_1x_2 - 846.77$

The adhesive properties (SFE) remain in strict relations with other characteristics of the material. The knowledge of SFE can therefore be useful not only in practice when selecting a suitable hydrophobising agent, but can also form the basis for the design of the composition of materials such as mortar, lightweight, plain and high performance concrete.

#### Logistic regression - odds ratio

The aim of calculating the odds ratio is to analyze the corrosive factors having a bearing on increasing the odds ratio of damage or protection of the surfaces and structures of the building materials considered in the work.

The chance of damage to samples of the selected construction materials subjected to freeze-thaw cycles is calculated below. The first group (A) constituted reference materials without hydrophobic protection, reference group (B) materials after hydrophobisation.

The odds ratio refers to situations where damage to materials (the occurrence of a particular phenomenon) is tested in two independent groups. It is expressed as the ratio of the chance of damage to the specimens in group A, or C (A), the chance of damage to the specimens in group B, or C (B). Table 4.6 summarizes the information about the tested samples, the chances of damage to the sample by frost and the odds ratio.

In the case of ceramic bricks and UHP fiber-reinforced concretes, the chance of damage to the samples by frost is respectively almost 5 and 4 times higher in the group of unimpregnated materials than in the bricks and fiber-reinforced concretes surface hydrophobised with organosilicon compounds. In the case of structural hydrophobisation (by mass) of ordinary concrete the situation is

reversed. The chance of damage to the concrete samples is 7.2 times higher among concretes hydrophobised structurally than of concretes without hydrophobic additives.

**Table 4.6.** *Chance and odds ratio of damage to materials during freeze-thaw cycles*

Material type	Group type	Number of samples			Chance of C(A) or C(B)	Odds ratio OR <sub>AxB</sub>
		damaged	undamaged	sum		
Lightweight mortar	A	21	33	54	0.636	2.802
	B	10	44	54	0.227	
Brick	A	7	13	20	0.538	4.847
	B	3	27	30	0.111	
Plain concrete	A	1	9	10	0.111	7.200
	B	8	10	18	0.800	
UHPRFC	A	8	34	42	0.253	4.016
	B	5	79	84	0.063	

Next, the chance of effectively protecting construction surfaces by hydrophobising (to obtain wettability at the initial stage of contact with water) with respect to their adhesive properties (SFE) was calculated. The first group (A) comprised materials without hydrophobic protection, the second group (B) materials after hydrophobisation. The considered parameter is the SFE values. It is understood that the material is wettable if the contact angle of the material with water is greater than 90 degrees. Then the SFE value for such an angle calculated by the Neumann method is 29.24 mJ/m<sup>2</sup>. In the work, for the analysis it was assumed that the material is wettable, or its adhesive properties are very low, if the SFE is less than 29 mJ/m<sup>2</sup>.

In Table 4.7, the calculation results of the logistic regression model - chance of obtaining wettability are compiled.

**Table 4.7.** *Logistic regression model - chance of wettability of building material surfaces after hydrophobisation*

Material type	Number of samples			Chance of C(A)
	$\gamma_s < 29$ [mJ/m <sup>2</sup> ]	$\gamma_s > 29$ [mJ/m <sup>2</sup> ]	sum	
Brick	8	12	20	0.667
Lightweight aggregate concrete	6	14	20	0.428
Plain concrete	10	10	20	1.0
UHPRFC	84	42	126	2.003
Concrete with waste	32	13	45	2.461

Based on the results, the materials are listed in order of increasing chances of obtaining wettability of the material surfaces by their hydrophobisation:



The lowest chances of effective hydrophobisation (wettability) have lightweight aggregate concrete. It was observed that with a decrease in porosity and absorbability of the material, the chances rise.

To compare the effectiveness of hydrophobising particular building materials with each other, in Table 4.8 the odds ratio is presented in the form of a matrix.

**Table 4.8.** Odds ratio matrix of building material hydrophobisation effectiveness

Odds ratio OR <sub>AxB</sub>	Brick	Lightweight aggregate concrete	Plain concrete	UHPFRC	Concrete with waste
Brick	-	1.56	1.50	3.00	3.69
Lightweight aggregate concrete	1.56	-	2.34	4.68	5.75
Plain concrete	1.50	2.34	-	2.00	2.46
UHPFRC	3.00	4.68	2.00	-	1.23
Concrete with waste	3.69	5.75	2.46	1.23	-

The greatest chance of effective hydrophobic protection of surfaces occurs in high performance concretes with boiler slag and foundry sand and high performance fiber-reinforced concretes with steel and polypropylene fibers. This chance is more than twice higher than in ordinary concretes with  $w/c = 0.45$  and more than five times higher than in lightweight aggregate concretes. Brick was more than three times less effectively protected against water than high performance concretes. The more porous and absorptive a material is, the less chance of attaining an SFE value  $< 29 \text{ mJ/m}^2$ , and therefore a wettable surface by its surface hydrophobisation. Analysis of the SFE results obtained after hydrophobisation of the materials indicates that the use of oligomeric alkyl alkoxy siloxanes significantly increases the chance of obtaining an SFE value  $< 29 \text{ mJ/m}^2$ , except for lightweight aggregate concretes, where the best SFE parameters were achieved using methyl silicone resin in an aqueous solvent. The application of macromolecular resin in the case of the other materials reduces the chance substantially. The risk of ineffective hydrophobisation can also be reduced by extending the hydrophobisation time, by applying more than two layers, employing compounds in organic solvents or in the form of creams and pastes.

A valuable benefit of the analyses obtained based on the odds ratio is the possibility of more accurate interpretation of hydrophobisation efficacy in comparison to other materials. The applied statistical method can evaluate the significance of this type of impregnation and estimate its chances of success.

### Work of adhesion

For selected construction surfaces, the work of adhesion  $W_{sl}$  was determined with the Dupré formula:

$$W_{sl} = \gamma_s + \gamma_l - \gamma_{sl} \quad (4.1)$$

where:

$\gamma_s$  - solid state SFE [ $\text{mJ/m}^2$ ],  $\gamma_l$  - liquid state SFE [ $\text{mJ/m}^2$ ],  $\gamma_{sl}$  - interfacial SFE (solid-liquid) [ $\text{mJ/m}^2$ ].

The work of adhesion calculation results are compiled in Table 4.9.

**Table 4.9.** *Work of adhesion of construction surfaces*

Material type	Work of adhesion $W_{st}$ [mJ/m <sup>2</sup> ]	Material type	Work of adhesion $W_{st}$ [mJ/m <sup>2</sup> ]
BK1	122.28	Brick	155.16
BK1-ZMW	68.42	C-ZMW	89.48
BK1-ZMO	36.12	C-ZMO	87.98
BK1-AAS	41.46	C-AAS	45.10
C1	119.64	C0	137.31
C1-ZMW	16.65	CZ10	144.45
C1-AAS	6.71	CZ30	151.80
C1-ZMO	89.24	SC	91.22

Based on the tests, it was observed that the greater the wettability of the material (smaller contact angle), the more work should be done in order to separate the analyzed bodies from each other. The more hydrophobic material was, the greater the contact angle, and the less work of adhesion. The smallest work of adhesion (6.71 mJ/m<sup>2</sup>) among the tested materials was obtained by UHPC C1 hydrophobised with alkyl alkoxy siloxane AAS.

### Summary

The results arising from the author's years of research have been gathered in the monograph "Surface free energy as a factor affecting hydrophobisation effectiveness in protection of building construction".

The basic task of the performed research and analyzes was to acquire and deepen the knowledge which is essential when selecting and performing the hydrophobisation procedure of construction surfaces with different properties and structures, increasingly used in a variety of structures operating in corrosive environments. For this purpose, a various number of experimental studies both in terms of determining the character of the tested construction materials and specifying their adhesion properties before and after hydrophobisation, was conducted. While implementing the scientific objectives, the efficacy of hydrophobising the surface of building materials i.e. ceramic brick, high performance concrete with/without fibers, plain concrete, lightweight aggregate concrete and lightweight mortars was evaluated. Hydrophobisation effectiveness largely depends on the physical properties of the treated surface - porosity, tightness, absorbability and the type of hydrophobising formulation.

In the study, the possibility to influence changes in the adhesion properties of construction materials was adopted, namely by applying a variety of organosilicon compounds on the surface of the materials.

The conclusions from the research presented in the work are as follows:

The difference in the effectiveness between formulations is noticeable, however, hydrophobisation with aqueous solutions usually leads to a small decrease in absorbability, especially of concrete, which is related to its high tightness. The resin obtained from macromolecular siliconates does not guarantee a hydrophobic effect in the long term, apart from lightweight concrete, as confirmed by the tests.

In addition, the water-soluble formulations caused the deposition of polysiloxane gel in the surface layer, significantly narrowing the width of the capillaries. This formulation does not penetrate the structure, particularly of concrete, but seals the pores of the substrate subsurface. It must be assumed that it limits the possibility of free movement of crystallized ice in the concrete structure, causing a decrease in strength and significant losses in mass of concrete hydrophobised with siliconates in relation to the reference concrete. Because of water penetrating the porous coating and frost corrosion, delamination at the interface between the substrate and the coating is possible. If the hydrophobic coating insufficiently protects the substrate against the ingress of water e.g., due to the lack of formation of a molecular hydrophobic layer or because of mechanical damage, scratches, cracks, the hydrophobisation process may result in increased destruction compared to the untreated material. The hydrophobisation of building materials shifts the ice crystallization zone deep into the material. Therefore, an important parameter to guarantee hydrophobisation effectiveness is the adhesion of coatings to the substrate.

The thin silicon film obtained from formulations based on VOC ensures hydrophobisation effectiveness. Due to its low molecular structure in the initial state, the formulation of low molecular weight alkyl alkoxy siloxane exhibits a very good ability to penetrate and chemically react in concrete in the presence of atmospheric moisture passing into the hydrophobic active substance, resistant to atmospheric agents - polysiloxane. The author agrees with the opinion that when creating a polymer coating, the geometry, the construction of the polymer molecules, rather than the chemical nature of the polymer compound are important, which also has been shown in the monograph. The hydrophobic coatings exhibited good water vapour permeability. Both the impregnated and reference samples obtained humidity comparable to that of pre-test absorbability, although the percentage drop in humidity is lowest in the case of the micromolecular hydrocarbon formulation. On this basis, it can be inferred that the hydrophobising agents do not impede the diffusion of water vapour from the impregnated material, or do so to a small degree.

Based on the results obtained while realizing the scientific achievements, the highest effectiveness of concrete and ceramic brick hydrophobisation is clearly indicated by using micromolecular alkyl alkoxy siloxane. In the case of highly porous materials as lightweight aggregate concrete, greater efficacy was achieved using macromolecular methyl silicone resins.

The most important scientific achievements are the results of the work on the effectiveness and resistance to frost and sulfate salts of materials which have not been or are seldom the subject of scientific research in this field. Such materials include lightweight aggregate concrete with sewage sludge, thermal insulating mortar with lightweight aggregate, ultra-high performance concrete with/without steel and polypropylene fibers, high performance concretes with waste materials such as slag from burning coal and foundry sand. A wide range of detailed analyses of commonly used materials such as ceramic brick was also performed. The hydrophobisation methods resulting the

author's own research and analyses of the literature, have been verified on elements coming from buildings and thereby their usefulness in practical applications was confirmed.

The adhesive properties were specified using surface free energy, determined by the four methods most commonly used for solids. Surface free energy is the most representative feature enabling one to determine the impact of adhesion properties on hydrophobisation effectiveness.

One of the results of the scientific achievements is the evaluation of the usefulness of empirical models for determining the surface free energy of hydrophobised building materials, and determination of the correlation between the experimental results and surface free energy.

It has been shown that the SFE values calculated using the Wu method deviate from the mean values obtained by other methods, indicating that it obtains the least reliable results. The most reliable results are obtained using the Owens-Wendt method. An equally accurate method of calculating SFE as the Owens-Wendt method is the Neumann method. The main advantage of this method is the possibility of using only one measuring liquid to determine the contact angle. This reduces the errors associated with experimental investigations and calculations.

The original outcome of a scientific nature is the determination of the correlation between SFE and such features as absorbability, porosity, water vapour permeability, loss in mass after the frost resistance test and the sulphate salinity test. In a few cases linear equations were obtained, and for the most part an equation of the second degree. A very good match of  $R^2$  data values, greater than 0.90 was obtained. With confidence, it can be stated that the constructed mathematical and experimental models showed a convergence of functions obtained during the modeling of the results obtained during the experimental investigations.

In many cases, the use of designated equations eliminates the execution of long-term experimental studies to determine the adhesion or corrosion resistance characteristics of hydrophobised and non-hydrophobised materials.

A logistic regression-odds ratio model was designed, with which analyses were performed of corrosive agents having an impact on an increase in the odds ratio of damage or protection of surfaces and structures of the building materials considered in the work. The chance of effectively protecting construction surfaces by their hydrophobisation, or obtaining wettability in the initial period of contact with water in relation to their adhesive properties (SFE) was also calculated.

The conducted study, testing and analysis work is an important contribution to the development of the discipline of civil engineering, in learning, deepening and complementing issues related to surface free energy as a factor shaping the effectiveness of hydrophobisation in the protection of construction surfaces.

It can be assumed that implementation of the scientific and research achievements described in the monograph will enable proper, effective hydrophobisation of building materials exploited for a long time in terms of their technical condition and corrosion resistance. Effective and durable hydrophobisation of the material protects against various types of corrosion to which it is exposed in



the external environment, allowing for long-term operation without the need for major maintenance or repairs.

Comparison of the experimental investigation results and conducted computational analyses together with the scientific objectives is the basis for stating that the research objectives have been achieved and verified by scientific methods.

The conclusions and observations resulting from the work presented in the studies suggest the need to intensify research work, particularly in the areas of studies on surface roughness, adhesion of the hydrophobic layer, the chemical properties of surfaces after hydrophobisation especially in a corrosive environment.

## **5. Other scientific-research achievements**

### **5.1. Prior to obtaining doctoral degree**

From 1.10.2002 to 30.09.2008. I worked as an assistant first at the Department of Civil and Sanitary Engineering and then at the Faculty of Civil Engineering and Architecture of Lublin University of Technology in the Institute of Civil Engineering in the Division of Construction, and then following reorganization in the Department of Construction.

The subject of my research work prior to my doctorate addressed the following issues [Annex No. 3]:

- problems and methodology of eliminating humidity and salinity from historic buildings [A1, A9, A11, A13-A15, A17-A19]
- assessing the effectiveness of hydrophobising porous structures of building materials, i.e. ceramics, stone [A7, A8, A10, A12]
- ecological constructions including asbestos removal [A16, A21, A22, A26, A27]
- modernization and thermo-modernization of residential buildings [A20, A23-A25]
- building materials [A28-A33]
- construction law [A2-A6].

Among the most significant scientific achievements of this period I include:

- Participation in development of the method of monitoring the moisture parameters of calcareous rock walls using TDR reflectometry techniques [A1, A9, A17].
- Carrying out analyses on the effectiveness of hydrophobising formulations in the preservation of historic ruins from calcareous rock [A8, A11-A14].
- The proposal to increase the insulation effectiveness of large block buildings, ways of modernizing the balconies in residential buildings from the late 1970s [A20, A24, A25].

Before my doctorate I published 21 articles in national journals, 4 chapters in scientific monographs and 8 papers in materials science and technology conferences. I participated in 6 national and 1 international scientific and technical conference.

## **5.2. Doctoral thesis**

The aim of the dissertation entitled "Evaluation of effectiveness of hydrophobisation of calcareous rock walls on basis of analysis of humidity parameters" was to determine the technological and conservation possibilities of using organosilicon compounds in the renovation of buildings of Kazimierz calcareous rock that has led to significant resistance of the rocks to water. The paper analyzes the effectiveness of protecting heterogeneous porous structures of calcareous rock from the harmful effects of rainwater through its hydrophobisation. Part of the work is to determine the rules of proceeding before making the decision to perform hydrophobisation treatment.

The studies present the influence and explain the interaction mechanism of hydrophobic silicone agents on the moisture parameters of Kazimierz limestone. Analysis was carried out on the properties of the produced hydrophobic coatings in the structure of five different rocks.

The subject of hydrophobisation is five types of calcareous rock of different parameters affecting the effect of the treatment, e.g. different orientation relative to the world (different insolation, frequency of freeze- thaw cycles), salinity, source of origin.

The material used for hydrophobisation are silicone compounds produced in Poland and Germany. Four formulations differing in the solvent, viscosity, concentration of the active substance, structure and particle size were selected.

The obtained results of the study made it possible to draw conclusions on the purposefulness and effectiveness of hydrophobic safeguards, of reconstructed and new walls of calcareous rock widely used in the construction industry area Lublin.

Based on literature analyses and the author's own research conducted in the work, the veracity of the proposed hypotheses was demonstrated. It means that:

- assessment of the hydrophobisation effectiveness of rock walls can be done depending on the humidity parameters of the stone
- the hydrophobisation effectiveness of rock walls can be predicted based on a set of calcareous rock tests.

## **5.3. After obtaining doctoral degree**

After obtaining my doctoral degree, in October 2008 I started working as an assistant professor at the Faculty of Civil Engineering and Architecture in the Department of General Construction Lublin University of Technology where I work to date. From 26.09.2011 to date I have also been working as a senior lecturer in a full-time position at Pope John Paul II State College in Biała Podlaska in the Faculty of Economics and Technology, in the Department of Technical Sciences in the Division of Construction.

### 5.3.1. Scientific publications

The leading direction of my interests is the continuation of the theme undertaken in the doctoral dissertation:

1. Evaluation of the effectiveness of hydrophobising porous structures of building materials, the resistance of porous building materials to corrosion, as well as surface free energy. Publications on this topic: B1-B5, B7-B9, B11, B12, B16, B18, B19 B20, B23, B24, B30, B33, B35, B36, B40, B48, B49, B52, B55, B56, B57, B59-B61, B63, B64, B66, C1, C2, C6, C7.

Considerable attention in my post-doctoral scientific work has been devoted to other issues related to civil engineering:

2. The development of construction objects regarding energy-saving technologies, ecological use of waste materials in construction, high performance concretes.

The original achievements attained in these research topics are documented by the following scientific publications: B6, B10, B13, B15, B17, B21, B25, B26, B27, B29, B32, B34, B41, B44, B47, C3-C5, C8-C10, C12.

3. The issue of moisture and salinity of historic buildings.

Publications on this topic: B22, B31, B38, B39, B43, B45, B46, B58, B62, B65.

4. Modernization and thermo-modernization of residential buildings.

Publications: B28, B37, B42, B50, B51, B53, B54, C11.

#### Ad. 1.

The most important scientific-research achievements in the scope of "Evaluation of the effectiveness of hydrophobising porous structures of building materials, the resistance of porous building materials to corrosion, as well as surface free energy", in addition to monograph [B1], discussed in section 4, I include:

- The publication of single author monograph [B63] which is the summary of several years of research within the scope of the doctoral dissertation on hydrophobising calcareous rock. The research results on the efficacy of hydrophobising calcareous rock are also presented in paper [B57].

- Analysis of the possibility of applying and evaluating the effectiveness of hydrophobisation with organosilicon compounds on other building materials, i.e. thermal-insulating mortars with perlite, lightweight aggregate and zeolite [B16, B18, B20, B33], lightweight aggregate concrete [B5, B9, B30], ceramic brick [B3, B7, B35, B40, B48, B55], corroded brick and ceramic tile [B24, B59, B64], plain concrete [B52]. Anti-graffiti agents on ceramic brick were also tested [B60]. Part of the work was devoted to compounds with a low content of volatile organic compounds (VOCs) [B11, B48].

The results of some of the works were presented at international conferences in Dubai [C7], Slovenia [C6], Kuala Lumpur [C1].

- Detailed analysis of the results of laboratory tests and to determine the correlation between the physical characteristics and the SFE of building materials, i.e. ceramic brick, lightweight aggregate concrete, mortar, high performance concrete with the addition of waste materials that cause higher wettability and lower resistance to corrosion, i.e. boiler slag from power plants, casting sand, waste sand from foundry processes and others, as well as high performance concrete containing steel and polypropylene fibers.

Novel accomplishments obtained in the research topic have been documented by scientific publications found among others in [B2-B4, B8, B9, B16, B23, B30], including those in the Journal Citation Reports database.

- Joint authorship of the development of production technology of lightweight concrete with the use of organosilicon compounds - polysiloxane, applied for in 2014 as a patent solution [B21] as well as the development of production technology of ultra-high performance concrete with/without steel and polypropylene fibers resistant to frost and sulphate corrosion, submitted to the Patent Office in 2016. (description in Subsection. 5.3.2).

#### **Ad. 2.**

The most important scientific and research achievements as well as the main topics in the scope of "Shaping building objects including energy-saving technologies, ecological use of waste materials in construction, high performance concrete" are as follows:

- One of the topics in this area was to study the possibilities of using lime-hemp composites to erect walls or to make from them filling for the frame of a wood house. In the period from 01.03.2014 to 31.12.2014. I participated as expert No. 1 in the project "Development of technology for building ecological and energy-efficient houses with composite filling of wooden framework" carried out at Pope John Paul II State College in Biała Podlaska in the Poland-Belarus-Ukraine Cross-Border Cooperation Programme 2007-2013. Original achievements obtained in these research topics are documented in one scientific publication found in the Web of Science database [B26] and co-authored monograph published in three languages (Polish, English, Russian) [B29]. After completing participation in the project, the topic was expanded and continued at the Lublin University of Technology in the Faculty of Civil Engineering and Architecture. The study was extended, among others by limestone composites, concrete composites and lime-gypsum composites with linen and hemp fiber as well as with polypropylene fibers. This resulted in the 2016 foreign publication [B17] and two accepted for print in 2017 in journals on the Philadelphia list [B13, B14].

- Another topic concerned the development of production technology of aggregates and lightweight concrete with the use of sewage sludge. The original achievements in this area are documented publications in list A [B5, B6], the results also were presented at an international conference [C9, C10].

- The compositions of thermal insulating mortar with additions of natural zeolite, perlite, and lightweight aggregate were developed. Their suitability for use in energy-saving construction [B34]

was investigated in the wide range tests studying the impact of their hydrophobisation on frost and sulfate corrosion contained in the articles listed in Ad.1.

- Another topic is the continued participation in the development of research results, among others, of high performance concrete with polypropylene and steel fibers, UHPC containing boiler slag and casting sand from waste sand from foundry processes.

The original achievements in this topic are documented in co-authored publications in list A [B4] and [B10] presented at an international conference in Slovenia [C5] as well as [B23, B32].

#### **Ad. 3.**

Simultaneously, I continued the theme of scientific research involving questions of moisture and salinity in the masonry of historic buildings. As part of the most important works, among others the possibility was demonstrated of applying surface TDR probes in the non-invasive measurement of moisture in historic buildings [B46, B58, B62]. Research on the effectiveness of formulations based on orthosilicic acid to strengthen historic Lublin stucco mortar was conducted [B45].

From July 2013 to June 2014, I participated as a specialist in the research project "Development of an innovative model of cross-border use of zeolite tuffs" carried out at the Lublin University of Technology in the Poland-Belarus-Ukraine Cross-Border Cooperation Programme 2007-2013, Priority 1. Increasing competitiveness of the border area. Within this activity I was co-creator of the compositions and laboratory tests of several restoration plasters using, among others, zeolite. It is worth noting that the work realized in the framework of the grant is important in application terms because as a result of it a new plaster was developed, which can be applied on walls with high salinity. Within these issues a patent was developed [B22], described in detail in Subsection 5.3.2.

Novel developments in this area were documented in publications [B18, B25, B31, B38, B39]. The results of the work were presented in the form of a presentation at the scientific and technical conference: Modern Technologies of zeolite tuff usage in industry. Lviv Polytechnic National University, Institute of Civil and Environmental Engineering, Lviv, Ukraine 2014 [C8].

#### **Ad. 4.**

In the context of issues related to the modernization and thermo-modernization residential buildings, an assessment was carried out of the effectiveness of internal insulation on the example of a historic building [B37], as well as traditional housing [B54]. In domestic and foreign papers [B28, B50, B51, B53] the conducted diagnostic records, analysis of the results of laboratory tests are discussed and their suitability for further use is identified.

### **5.3.2. Patent applications**

1) Smarzewski P., Barnat-Hunek D., *Lightweight concrete*. Polish Patent Office's Official Gazette 44 (3) (2016) 13-13.

The subject of the patent application is a lightweight concrete with sediments coming from the treatment of potable water from city waterworks. In the composition of the concrete water-soluble organosilicon compounds – polysiloxanes were proposed.

- 2) Barnat-Hunek D., Klimek B., Franus M., *Restoration plaster*. Application no. (21) 410436 Polish Patent Office's Official Gazette 44 (13) (2016) 20-20.

The subject of the patent application is a restoration and primer plaster with the addition of natural zeolite, blast furnace slag or lightweight aggregate.

- 3) Smarzewski P., Barnat-Hunek D., *Ultra-high performance corrosion resistant concrete*. Patent application P.416800 dated 2016-04-11 (not yet published)

The subject of the invention is an ultra-high performance concrete with steel and polypropylene fibers resistant to frost and sulfates.

### **5.3.3. Management of international and national research projects and participation in such projects**

- 1) The project "Development of technology for building ecological and energy efficient houses with composite filling of wooden framework", Pope John Paul II State College in Biała Podlaska, Poland-Belarus-Ukraine Cross-Border Cooperation Programme 2007-2013. No: IPBU.02.01.00-06-704 / 11-00 **position of expert No. 1**. 03.01.2014 - 31.12.2014.
- 2) The project "Development of an innovative model of cross-border use of zeolite tuffs" Lublin University of Technology, Poland-Belarus-Ukraine Cross-Border Cooperation Programme 2007-2013, Priority 1. Increasing competitiveness of the border area. **Specialist in testing**. 07.2013 - 06.2014.
- 3) POIG project, Action 1.3: R & D project, support for entrepreneurs carried out by scientific entities WND-POIG.01.03.01-06-146 / 09 "Innovative technology for production of zeolite from fly ash" Lublin University of Technology, **executor** 2009.

### **5.3.4. National and international awards for scientific activities**

- 1) Individual award of the third degree from the Rector of Lublin University of Technology for achievements in scientific activity in the academic year 2007/2008.
- 2) Medal of the National Education Commission in 2016.

### **5.3.5. Participation in scientific work of University collective bodies**

- 1) Secretary of the Scientific Committee on Doctoral Procedures at the Faculty of Civil Engineering and Architecture of Lublin University of Technology.
- 2) Member of the Faculty Committee on Commercialization for the term 2016-2020.

## 6. Summary of scientific and research activities

Table 1 lists the total number of works published and other important achievements before and after obtaining the doctoral degree. In brackets the number of single author works is provided.

Table 2 presents the list of the *impact factor* and publications in the Web of Science database.

Table 3 shows the total number of citations and the Hirsch index.

**Table 1.** Summary of most important scientific and research achievements in terms of quantity and points

Type of publication	Number of publications before doctorate	Number of publications after doctorate	Total number of works
Original published scientific creative work, monographs, chapters in monographs, conference materials			
Articles in JCR database journals	-	11 +2	11 +2
Monographs in Polish	-	3 (2)	3 (2)
Chapters in monographs in Polish	3 (1)	9	12 (1)
Chapters in monographs in English	-	2	2
Articles in international journals	-	9	9
- in English, not included in JCR database	-	1	1
- In another language	-	-	-
Articles in journals with national coverage	22 (3)	23 (1)	45 (4)
Papers for international conferences in English	-	3(1)	3 (1)
Papers for national conferences	8 (1)	1	9 (1)
Posters at international conferences in English	-	7 (1)	7 (1)
<b>Total</b>	<b>33 (4)</b>	<b>69 +2 (5)</b>	<b>102 +2 (9)</b>
Participation in projects			
NCBiR	-	3	3
EU	-	2	2
<b>Total</b>	-	<b>5</b>	<b>5</b>
Patent applications			
National applications	-	<b>3</b>	<b>3</b>
Awards and distinctions for scientific, educational and organizational activities			
Rector's Award PL	-	2	2
NEC Medal	-	1	1
<b>Total</b>	-	<b>3</b>	<b>3</b>
Scientific supervision of students			
Supervised engineer's theses	-	93	93
Supervised master's theses	-	33	33
<b>Total</b>		<b>126</b>	<b>126</b>

After my doctorate, I published in total:

- 3 monographs including 2 single author monographs
- 11 chapters in monographs (2 in English)
- 11 articles in journals from the JCR database (+ 2 accepted for publication)
- 34 articles in other journals (including 10 in international journals in English, one in Ukrainian, 23 in Polish)
- 3 papers in international scientific conference publications.

The total number of points of all the publications is **644**, and after obtaining the doctoral degree **596**, according to the year of publication.

(+ 40 points for articles accepted for publication in 2016 and 2017. = **684**)

The total *impact factor* of my publications in the Web of Science database (Table 2) is 17.398 (18.382), the h-index according to the database Web of Science is equals 3 (Table 3).

**Table 2.** Publications in Web of Science database, and impact factor summary

Lp.	Journal	Year of article publication	Position in Annex No. 3	Impact Factor	MNiSW points in year of issue
1.	Construction and Building Materials	2016	B2	2.421	40
2.	Construction and Building Materials	2016	B3	2.421	40
3.	Construction and Building Materials	2016	B4	2.421	40
4.	Materials	2016	B5	2.728	35
5.	Environmental Monitoring and Assessment	2016	B6	1.679	25
6.	Energy and Buildings	2015	B7	2.973	40
7.	Materiali in Tehnologije	2015	B8	0.548	15
8.	Materiali in Tehnologije	2015	B10	0.548	15
9.	Ecological Chemistry and Engineering S	2015	B9	0.553	15
10.	Ecological Chemistry and Engineering S	2014	B11	0.553	15
11.	Ecological Chemistry and Engineering S	2011	B12	0.553	15
12.	Composites Theory and Practice	2016	B16	-	11
13.	Composites Theory and Practice	2015	B23	-	11
14.	Composites Theory and Practice	2015	B26	-	11
15.	Journal of Natural Fibers	2016	B13	0.492	20
16.	Journal of Natural Fibers	2017	B14	0.492	20
<b>Total</b>				17.398 18.382*	328 368*

Total points including articles accepted for publication (15.16)

**Table 3.** Summary of the total number of citations and an Hirsch index

Database name	Number of publications in the database	The total number of citations	Hirsch index
Web of Science	14	21	3
Scopus	11	21	3
Google Scholar	48	55	4

