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Bound water content measurement in cement pastes by stoichiometric and gravimetric analyses

Piotr Prochoń^{a,*}, Tomasz Piotrowski^a

^a Department of Building Materials Engineering, Faculty of Civil Engineering, Warsaw University of Technology, Al. Armii Ludowej 16, 00-637 Warsaw, Poland

Abstract

The cement hydration is one of the most complex processes creating the cement matrix. The degree of hydration is a quantity of Portland clinker with gypsum, that as a whole react with water. For purposes of this analysis it was found to be directly proportional to the bound water in the cement paste specimens. Using the gravimetric method the bound water content was determined in samples by calculating the quantitative loss of the dry specimens weight after igniting to 1000°C and keeping the temperature for 5h. Obtained results were collated with theoretical stoichiometric analysis based on the Bogue and Taylor equations and XRD testing for Portland cement, CEM I 42.5. Obtained outcome has confirmed the relationship between the degree of hydration and the bound water content in cement paste. The influence of the w/c ratio and cement oxide components on bound water quantity were also noticed.

Keywords: Bound water; cement paste; Bogue equation; hydration

1. Introduction

Water-cement or water-binder ratio is one of the most important characteristic of cement pastes and mortars. It affects the rheological and mechanical properties of the blend, its permeability and durability (Aitcin and Flatt 2015). The increase in w/c ratio contributes to the porosity of the hardened paste and its reduction leads to incomplete cement hydration. It is recommended to choose w/c ratio as low as possible retaining optimum cement matrix plasticity (Peukert 2000; Jamróży 2009).

1.1. Water in cement matrix

Water in the hardened cement paste can be in chemically bound (constitutive) or free (unbound chemically) state. Constitutive water is closely bounded with the crystal phase and its separation may occur only by calcining the material at high temperatures or by chemical reactions. The unbound chemically water includes water vapor, physico-mechanically and physico-chemically bound water (pellicular, sorption). Pellicular water occurs on the developed pores surface or in cement paste capillaries and with fixed water vapor relative humidity in the pores, it remains at a constant level. The physico-mechanically bound water emerges as a result of the material

* Corresponding author. E-mail address: p.prochon@il.pw.edu.pl

supersaturation with water vapor (Witakowski 2008). Tamtsia and Beaudoin (2000) proposed a more detailed partition. They distinguished water chemically bound within the tobermorite-like crystallites and four types of evaporable water:

- pore water in the capillary and gel voids, outside the range of van der Waals adhesion forces,
- water adsorbed in layers not more than 2 molecules deep on the crystal surface,
- intercrystalline adsorber water,
- intercrystalline zeolitic water.

1.2. Cement hydration

Cement hydration is a very complicated process, involving in cement technology set of reactions and physical processes occurring after mixing cement with water (Czarnecki et al. 1994; Kurdowski 2014). The main Portland cement phases participating in the process of hydration are: alite (C_3S), belite (C_2S), celite (C_3A), brownmillerite (C_4AF) and gypsum (Nocuń-Wczelik 2003). The silicate hydration forms a gel of calcium silicate hydrate (C-S-H) and a crystalline phase called portlandite (calcium hydroxide). Aluminates and calcium sulfate react with water creating products of two different phase families: trisulfoaluminoferrite hydrates (AFt), with the most important phase, ettringite, and monosulfoaluminoferrite hydrates (AFm) composed of calcium and aluminum platelets with positive charge in an octahedral coordination with oxygen (Aitcin and Flatt 2015). The AFm phases differ among each other by its counter-ions intercalated between the platelets: monosulfoaluminate ($4CaO \cdot Al_2O_3 \cdot SO_3 \cdot 12H_2O$) monocarboaluminate ($3CaO \cdot Al_2O_3 \cdot CaCO_3 \cdot 11H_2O$) or Friedel's salt ($3CaO \cdot Al_2O_3 \cdot CaCl_2 \cdot 10H_2O$) (Aitcin and Flatt 2015; Gambal 2013).

Calcium silicates have a dominant role in the hydration process with approx. 60% content in the clinker. Their hydrates affect both the development of early and final strength. C_3S reaction with water is a complex heterogeneous process, and can be divided into five to six stages, due to hydration temperature rate change to time of hydration (Aitcin and Flatt 2015; Kurdowski 2014). The hydrolysis of calcium silicates are shown in Table 1.

Table 1. Simplified hydration progress of portland cement phases (Czarnecki et al. 1994).

Portland cement phases	Hydration
$C_3S = 3CaO \cdot SiO_2$	$2C_3S + 6H \rightarrow C_3S_2H_3 + 3CH$
$C_2S = 2CaO \cdot SiO_2$	$2C_2S + 4H \rightarrow C_3S_2H_3 + CH$
$C_3A = 3CaO \cdot Al_2O_3$	without gypsum: $C_3A + 6H \rightarrow C_3AH_6$ (hydrogarnet) with gypsum (when $C\hat{S}H_2/C_3A = 3$): $C_3A + 3C\hat{S}H_2 + 26H \rightarrow C_6A\hat{S}_3H_{32}$ (ettringite) with gypsum (when $C\hat{S}H_2/C_3A = 1$): $2C_3A + C_6A\hat{S}_3H_{32} + 4H \rightarrow 3C_4A\hat{S}H_{12}$ (monosulfoaluminate) or $C_3A + C\hat{S}H_2 + 10H \rightarrow C_4A\hat{S}H_{12}$ with gypsum (when $C\hat{S}H_2/C_3A < 1$): $C_3A + C_4A\hat{S}H_{12} + CH + 12H \rightarrow 2C_3A(C\hat{S},CH)H_{12}$
$C_4AF = 4CaO \cdot Al_2O_3 \cdot Fe_2O_3$	without gypsum: $C_4AF + 7H \rightarrow C_3(A,F)H_6 + C(A,F)H^*$ with gypsum: $C_4AF + 3C\hat{S}H_2 + 27H \rightarrow C_6(A,F)\hat{S}_3H_{32} + C(A,F)H^*$ after depleting gypsum: $C_4AF + C_6(A,F)\hat{S}_3H_{32} + 2CH + 2H \rightarrow 3C_4(A,F)\hat{S}H_{12}^*$

* (A,F) indicates aluminum with variable substitution of iron

Tricalcium aluminate (C_3A) is a clinker phase with quick reaction with water and the greatest impact on the rheology of cement paste. As a result of its hydration, hexagonal hydrates are formed, which, at temperature above $30^\circ C$ creates a solid, regular cement phase (C_3AH_6). Such rapid reaction is not conducive to the strength of hardened cement, impeding the hydrolysis of calcium silicates. In order to inhibit C_3A reaction with water gypsum additive is used (Kurdowski 2014). The course of its hydrolysis reaction with and without participation of gypsum is presented in Table 1.

Calcium aluminoferrites reaction with water is much more slower than with C_3A , probably due to its lower heat of hardening, and in result lower hydration temperatures. During those reactions hydrates $C_4(A,F)H_{19}$, $C_4(A,F)H_{13}$, C_3FH_6 are formed. C_3FH_6 is formed directly from ferrite at temperature above $50^\circ C$, without any hydrates in intermediate hexagonal form. C_4AF hydration is inhibited in the saturated liquid phase with $Ca(OH)_2$ and gypsum (Czarnecki et al. 1994; Kurdowski 2014). A simplified hydration progress is shown in Table 1.

1.3. High temperature long-term influence on cement composites

Primarily, cement based composites subjected to long-term temperature impact result in drying. This process is gradual and water is released depending on the energy that binds it to the solid phase. Subsequently, the free water evaporates first, further capillary and physically bound water, and finally, at the very end, water chemically bound in the cement hydrates. The loss of bound water has a significant effect on the decrease in the mechanical properties of the cement pastes, which depend, in about 50%, on cohesive forces and bonding presented in C-S-H. Heating of concrete causes an increase in aggregate volume and manifestation of shrinkage in the cement matrix surrounding the aggregate weakening the cohesion between both phases. This leads to formation of cracks and damages on the surface of the cement based composite materials (Hager 2013).

Table 2 shows changes in concrete under high temperature. With the temperature increase from room temperature to 1000°C occurs a gradual degradation of the material, especially cement paste, resulting in a significant change in the chemical composition and microstructure of the hardened cement paste. At low temperatures, these reactions are mainly connected with dehydration and water release. At 100°C the evaporation of water accelerates as a result of subpressure created by vapour water in the cement based composite. At temperatures above 105°C all free water evaporates. From 120°C to approximately 500°C transpires evaporation of physically bound water contained in the small pores and capillaries. At the same time, from 150°C (stability temperature of C-S-H gel) begins the process of dehydration. Portlandite decomposes at 460°C-540°C. Between 600°C and 700°C a decomposition of the second phase of C-S-H and formation of β -C₂S occurs. Above 600°C limestone aggregates decomposes into CaO and CO₂. However, this process depends on the content of SiO₂, temperature and pressure. At 1200°C some concrete components melts, and from 1300-1400°C cement based composite exists only in a liquid state (Hager 2013; Bednarek et al. 2009; Naus 2005).

Table 2. The list of changes in concrete during heating (Hager 2013).

Temperature range	Changes
20-200°C	slow capillary water loss and reduction in cohesive forces as water expands; 80-150°C ettringite dehydration; C-S-H gel dehydration; 150-170°C gypsum decomposition; physically bound water loss;
300-400°C	350°C break up of some siliceous aggregates; 374°C critical temperature of water;
400-500°C	460-540°C portlandite decomposition $\text{Ca(OH)}_2 \rightarrow \text{CaO} + \text{H}_2\text{O}$;
500-600°C	573°C quartz phase change $\beta - \alpha$ in aggregates and sand;
600-800°C	second phase of the C-S-H decomposition, formation of β -C ₂ S;
800-1000°C	840°C dolomite decomposition; 930-960°C calcite decomposition; $\text{CaCO}_3 \rightarrow \text{CaO} + \text{CO}_2$, carbon dioxide release; ceramic binding initiation which replaces hydraulic bonds;
1300°C	total decomposition of concrete, melting.

2. Experimental

This study concentrates on determining the content of chemically bound water in the cement pastes. In the case of free water it is considered to be released at 105°C. Samples should be heated in stable environmental conditions till receiving its constant dry weight. It is recommended to do the Loss on Ignition analyze (LOI) for tested cements in order to determine its original moistures (Fagerlund 2009).

Chemically bound water is strongly bound to the solid-state structure of cement paste. Therefore, its loss may occur only under certain conditions. In this study authors used a gravimetric method, consisting in heating the samples to 1000°C and leaving them at this temperature for 5h. Presented above method is consistent with research of other authors (Dillshad 2011; Fagerlund 2009; Mouret et al. 1997; Bentz and Stutzman 2006; Quincot et al. 2011).

2.1. Stoichiometric analysis

In most studies Bogue equations are basis for calculating cement phase composition. This method bases on a number of assumptions like: limiting cement composition only to the most essential phases and having equilibrium state in the clinkerization process and that $A/F = 1$ in C_4AF (Czarnecki et al. 1994). These assumptions led to the development of four equations:

$$C_3S = 4.07 \cdot CaO - 7.6 \cdot SiO_2 - 6.72 \cdot Al_2O_3 - 1.43 \cdot Fe_2O_3 \quad (1)$$

$$C_2S = -3.07 \cdot CaO + 8.6 \cdot SiO_2 + 5.07 \cdot Al_2O_3 + 1.08 \cdot Fe_2O_3 = 2.87 \cdot SiO_2 - 0.75 \cdot C_3S \quad (2)$$

$$C_3A = 2.65 \cdot Al_2O_3 - 1.69 \cdot Fe_2O_3 \quad (3)$$

$$C_4AF = 3.04 \cdot Fe_2O_3 \quad (4)$$

Other author (Taylor 1997) presents opinion that Bogue equations give too small values for alite and celite and too big for belite. He introduces some amendments taking into account the impurities content (Na_2O , K_2O) in the clinker phase. Additionally Taylor defines conditions that must be fulfilled in order to get close to the real phase content of cement. The oxides analysis with unbound calcium content need to be free of errors. The composition adjustments must be made for the four main oxides owing to the fact that a part of them is not considered in composition of the main phases of a cement. This applies both to adjustments associated with the free silica and sulfur phases as well as insolubility of some silicon and aluminum oxides. Taking that into consideration the equations take form (Taylor 1997, Czarnecki et al. 1994):

$$C_3S = 4.51 \cdot CaO - 8.59 \cdot SiO_2 - 6.90 \cdot Al_2O_3 - 1.51 \cdot Fe_2O_3 \quad (5)$$

$$C_2S = -3.61 \cdot CaO + 10.08 \cdot SiO_2 + 5.17 \cdot Al_2O_3 + 1.03 \cdot Fe_2O_3 \quad (6)$$

$$C_3A = 0.13 \cdot CaO - 0.40 \cdot SiO_2 + 3.65 \cdot Al_2O_3 - 3.96 \cdot Fe_2O_3 \quad (7)$$

$$C_4AF = -0.05 \cdot CaO + 0.008 \cdot SiO_2 - 0.83 \cdot Al_2O_3 + 5.63 \cdot Fe_2O_3 \quad (8)$$

For cement chemical compositions listed in Table 3 the contents of cement phases were calculated (eq. 1-8) and summarized in Table 4.

Table 3. Cements chemical compositions.

	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	SO ₃	Cl	Na ₂ O	CaO _w
	%	%	%	%	%	%	%	%	%
CEM I 42.5 R	63.16	18.92	5.06	3.14	1.32	3.22	0.049	0.19	0.41
CEM II/A-S 42.5 R	59.63	22.33	5.30	2.49	3.35	2.85	0.039	0.00	0.47
CEM II/B-S 42.5 N	51.26	30.78	7.22	2.95	4.36	2.39	0.073	0.00	1.15
CEM II/A-LL 32.5 R	60.31	18.23	5.21	2.60	2.52	2.95	0.073	0.00	0.47
CEM III/A 42.5 N	48.83	31.09	7.77	2.15	6.26	2.07	0.093	0.85	0.84
CEM V/A(S-V) 32.5 R	42.75	32.97	10.04	3.27	4.20	2.61	0.069	0.00	1.33

Table 4. Phase contents of testing cements.

	C ₃ S		C ₂ S		C ₃ A		C ₄ AF	
	Taylor	Bogue	Taylor	Bogue	Taylor	Bogue	Taylor	Bogue
	%	%	%	%	%	%	%	%
CEM I 42,5 R	62.71	63.93	8.08	6.03	6.10	8.10	10.28	9.55
CEM II/A-S 42,5 R	25.67	23.78	48.69	46.10	7.98	9.84	6.48	7.57
CEM II/B-S 42,5 N	-100.22	-89.53	175.77	155.80	8.66	14.15	7.86	8.97
CEM II/A-LL 32,5 R	64.09	57.87	4.80	8.63	8.94	9.41	7.18	7.90
CEM III/A 42,5 N	-114.02	-102.15	187.76	166.21	13.46	16.96	3.01	6.54
CEM V/A(S-V) 32,5 R	-178.86	-161.57	244.68	216.43	15.66	21.08	7.74	9.94

Values obtained on the equations basis (Table 4) may be compatible with the actual phase configurations in cements only for pure portland cement: CEM I 42.5. Other types of cement obtained too low or even negative phase values and cannot be considered as probable. To validate the results X-ray diffraction analysis (XRD) was taken for CEM I (phase composition: 58.6% alite, 13.9% belite, 6% celite and 9.9% brownmillerite). In accordance with those results the reliance on values calculated from the formulas of Taylor and Bogue is insufficient and can lead to wrong assumptions. That is why further theoretical analysis of the bound water content is performed only for the phase composition of CEM I based on XRD examination and hydrolysis reactions (Table 1). For the purposes of the study some assumptions were made. The gypsum content in cement is 3%. In the hydrolysis reactions reacts whole cement and gypsum reacts with C_3A and C_4AF , in ratio 2:1 according to the speed of those reactions (faster for C_3A).

The content of bound water was calculated from the molar ratio of cement phases and water counted for 100g of cement. In celite and brownmillerite reactions there was calculated additionally amount of water needed for the hydrolysis reactions without the assistance of gypsum. All obtained results are presented in Table 5.

2.2. Gravimetric analysis

In this study six types of cements were used, including four classes of 42.5 (CEM II/A-S, CEM II/B-S, CEM III, CEM I), and two 32.5 (CEM II/A-LL, CEM V). Beside CEM III, which has ordinary early strength (N) others characterizes by high early strength (R). In addition, CEM V belongs to a special group of cements with low heat of hydration (LH). Their element composition given as oxides is shown in Table 3.

All samples were molded in cylindrical forms with dimensions of 50 x 50 mm and prepared in accordance with normative guidelines (EN 206; EN 196-1). For each cement types six series of samples of different ratio w/c (0.23, 0.25, 0.275, 0.30, 0.35) were prepared. After demoulding samples were placed in constant environmental conditions (relative humidity 65%, temperature $22 \pm 2^\circ\text{C}$) for 90 days.

In order to determine the content of bound water (BW) in the cement pastes first, samples were heated to 105°C , to get rid of the so-called free water. At this temperature tested samples were staying until gaining constant dry mass (m_s). The dry mass of a sample is defined as a last weight obtained by sample when the differences between consecutive measurements are $\leq 0.2\%$ (PN-B-06250). After that, examined entities were again placed in a furnace and heated to 1000°C . This temperature was maintained for 5 hours, and followed by self-cooling of the samples to 80°C . After that their weight was measured (m_{sc}). The content of bound water in cement pastes was calculated with Eq. 9. Obtained results and the trend lines are presented in Figure 1.

$$\text{content of bound water [\%]} = \frac{(m_s - m_{sc}) \cdot 100\%}{m_{sc}} \quad (9)$$

3. Results

Figure 1 shows the results of BW content in cement pastes for different water to cement ratios. It can be seen that for each cement type with the increase of w/c ratio there is a linear increase in BW with similar angular coefficient values for all cements. Therefore, the evaluation of tested samples can be performed for a selected single w/c = 0.35.

With the above presumption the highest BW content was found for the CEM II/A-LL (21.2%) and the lowest for CEM III (11.72%). Other values ranged between 13 and 17%. Such big difference between extreme results may indicate incomplete reaction of the cement components in the samples, lower content of hydrates and different hydrates by the reaction of the SCM (supplementary cementing material). It may be noted also that the trend function for CEM I and CEM V have the smallest angular coefficients, and because of that in those cements with the increase of w/c ratio the BW content will grow slower determining them as cements with the least BW content from examined.

Analyzing the chemical composition of cement (Table 1) there is a noticeable relationship between BW and calcium, silicon and aluminum oxides percentage in cements. The lowest water content represents cements with higher percentage of SiO_2 , Al_2O_3 and lower CaO. Both, the CEM V and CEM III, has their trend lines positioned at the bottom of the graph (Figure 1) and have less than 50% of CaO, more than 7% of Al_2O_3 and over 30% of SiO_2 . For comparison, CEM II/A-LL consists of 60.3 % of CaO, 5.21% of Al_2O_3 and only 18.2% of SiO_2 .

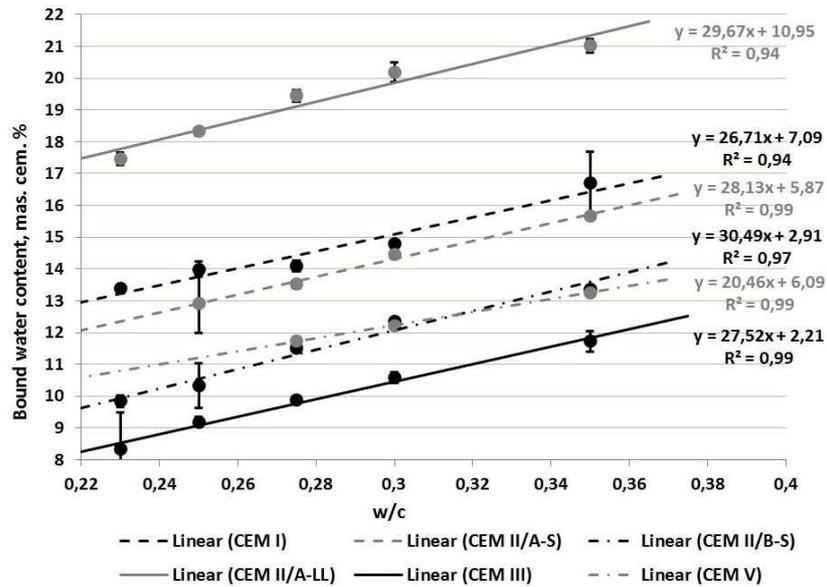


Fig. 1. Bound water content in cement pastes with different w/c ratio.

Comparing the molar ratios of the hydration components with the phase composition of portland cement CEM I 42.5R, in accordance with previously mentioned assumptions, it was considered that only 1/3 of the celite reactions is with gypsum. The remaining part of C_3A undergoes hydrolysis and form hydrogarnet. In case of brownmillerite hydration, only 1/10 of it reacts with gypsum and the rest directly with water.

The average theoretical content of bound water in cement paste samples, calculated on the basis of a stoichiometric analysis is 22.61% (Table 5). Comparing it with the test results (Figure 1 and 2) it is noticeable that BW content is on average lower by 8.03% in measured samples. The increase of w/c ratio narrows that gap (to 5.44%). This confirms the influence of w/c ratio on bound water content in the cement paste.

Table 5. Phase composition of cement paste with CEM I 42,5R and bound water calculation results.

Mass [g]	Water/cement ratio				
	0.23	0.25	0.275	0.3	0.35
Sample	118.9	144.4	183.07	182.27	171.17
Cement	96.67	115.52	143.58	140.21	126.79
Alite	56.65	67.69	84.14	82.16	74.30
Belite	13.44	16.06	19.96	19.49	17.62
Celite	5.80	6.93	8.62	8.41	7.61
Brownmillerite	9.57	11.44	14.21	13.88	12.55
Gypsum	3.57	4.33	5.49	5.47	5.14
Bound water mass for 100g of phase component:					
Alite	23.68				
Belite	20.93				
Celite	173.33 (after depleting gypsum: 33.33)				
Brownmillerite	100.00 (after depleting gypsum: 25.93)				
Chemically bound water [%]	23.97	22.63	22.44	21.92	22.15

The degree of hydration (Figure 2) in relation to the BW content has a correlation close to linear. A change of w/c ratio of 0.12 increases the degree of hydration of approx. 36% (Figure 1). Attempts to study and determine the accurate relationship between the bound water and the degree of hydration and its generalization for cement based composites can be useful, in particular, for the shielding materials since, as the authors (Mazgaj and Piotrowski 2015; Piotrowski et al. 2015a; Piotrowski et al. 2015b; Piotrowski and Skubalski 2014) point out, moisture and BW content have an influence on the shielding properties against ionizing radiation of neutrons.

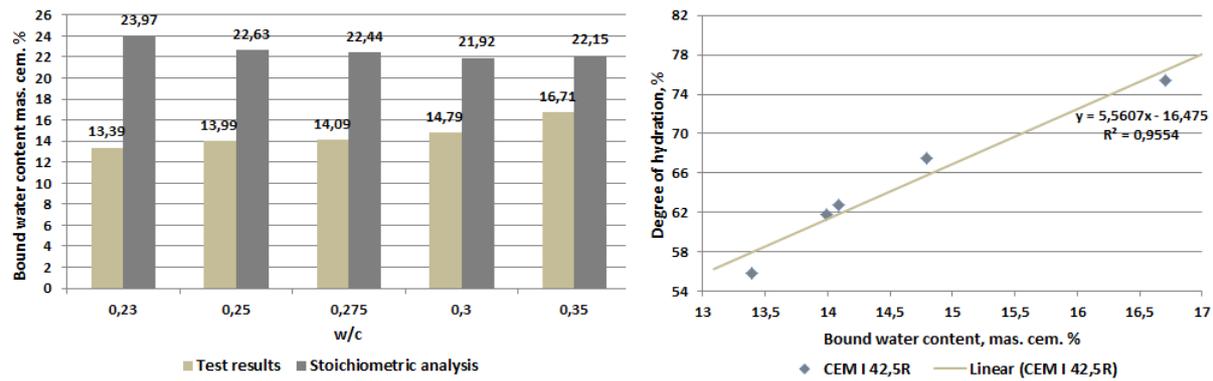


Fig. 2. Comparison of BW content from both analysis (left) and degree of hydration and BW content relation (right).

4. Conclusion

The w/c ratio change has a linear influence on the BW content in a cement matrix, in example, a w/c ratio increase of 0.1 causes an increase in the BW content from 2.6% to nearly 4.0%. The highest BW content among examined cement pastes characterized samples made with CEM II/A-LL. Other specimens with a higher content of Al_2O_3 and SiO_2 show lower values of this parameter. That is why it is recommended to take BW content according to the used type of cement. Those with addition of limestone should have a higher value of measured parameter, in example 25%, while others near 15%. Next studies, however, should verify if the numerical values of this parameter will not change after taking into account LOI of the individual components – cement and aggregates.

Using Taylor and Bogue equations is reasonable only for not modified cements and cannot be an exchange for experimental studies. For all cements the bound water content measured by stoichiometric analysis was higher than the results received in the experiment. With the w/c ratio growth the hydration degree increased in linear pattern, showing the importance of moisture for the hydration process. Further studies are in process for analyzing bound water content in concrete to find possible relations between water content in cement pastes and concretes.

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