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Influence of addition of calcium sulfate dihydrate on drying of autoclaved aerated concrete

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Abstract: The quality of the autoclaved aerated concrete (AAC) strongly depends on the chemical composition of the raw materials, as well as on the process of the hydrothermal reaction during autoclaving. Performance parameters depend on material structure: fine micron-scale matrix porosity generated by the packing of thin tobermorite plates and coarse aeration pores arising from the foaming of wet mix. In this study the binder varied in calcium sulfate dihydrate ($CaSO_4 \cdot 2H_2O$) content. Five series of AAC specimens were produced, with gypsum content 0; 0.55; 1.15; 2.3 and 3.5% of dry mass respectively. AAC units were produced in UNIPOL technology. The study presents experimental results of AAC moisture stabilization. The initial moisture content was determined directly after autoclaving. Slower drying process was observed for samples containing over 2% of gypsum. Whereas other performance parameters, compressive and tensile strength, as well as water absorption and capillary rise, were significantly better comparing to the reference AAC samples.

Keywords: autoclaved aerated concrete, calcium sulfate dihydrate, water absorption, stabilized moisture content

1 Introduction

In any construction process, it is important to promptly stabilize the moisture content of built-in materials to obtain the declared properties of the products as quickly as possible. Numerous masonry materials have increased moisture content as a result of wet production processes and often this condition adversely affects the parameters of building partitions. It is a common practice to built-in damp materials because of improper storage or due to moisture after the manufacturing process.

AAC is widely used for masonry wall construction in many countries, particularly in Europe. AAC products have different humidity during the masonry works, as well as the dynamics of their moisture stabilization is varied. Cellular concrete units after autoclaving process have moisture content of about 50% and they are often built-in with a moisture content exceeding 40%. The constructed wall may have water content of about 15%.

AAC is a porous material and absorbs moisture from the environment when the relative humidity exceeds 70%. The time needed to stabilize the moisture in the cellular concrete partition is between 1.5 and 2 years, and under extremely unfavorable conditions 2 to 3 years. The first figures in both cases refer to AAC produced using siliceous sand and the second figures to units with fly ash. The examination of AAC walls in buildings after 30 to 40 years has confirmed that the moisture content of partition from sand AAC was about 2.5% while partitions from fly ash AAC - about 4.5%. At such a moisture content, the partitions constructed from AAC units show good thermo-insulating properties [1]. European standard EN 771-4 permits operating moisture of AAC units at 4-8% [2].

Compressive strength is dependent on the moisture content of the material and decreases with moisture growth. There is a close dependency of compressive strength, water absorption and other physical properties on material porosity and the pore size distribution. Moisture is the key feature that determines strength, shrinkage, carbonation and thermal conductivity [3]. Particularly unfavorable is the effect of humidity on compressive strength and coefficient of thermal conductivity λ [4–6]. Authors [7] describe the problem of determining the coefficient of thermal conductivity under various humidity conditions, based on the standard ISO 10456 [8] and experimental studies. It has been found by laboratory tests that within a range of moisture content up to 5% the standard dependency (exponential function) is correct. Above 5% the course of changes is close to linear.

"White" AACs are generally produced from a mixture of finely ground silica sands, Portland cement, lime, gyp-

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sum (calcium sulfate dihydrate) and water. Adding aluminum powder develops its cellular or foamed character.

AAC is produced in a variety of technologies, and the proportions of binders in concrete are also different. The C-S-H phase, which binds the sand particles, plays a major role in the structure formation of cellular concrete and its compression strength [9]. A typical process for the AAC production includes hydrothermal treatment of concrete mixture at high temperatures (typically 180-200°C) under a saturated steam pressure. During the hydrothermal treatment platy-shaped tobermorite crystals ($5CaO \cdot 6SiO_2 \cdot 5H_2O$) are formed as a major component of hardened AAC [9–13].

In AAC technologies the source of sulphates, besides cement, is usually calcium sulphate in the form of gypsum or anhydrite. The most commonly used gypsum dihydrate (hydrated calcium sulphate) as one of the components of the concrete binder. Sulphates in an aerated concrete mixture regulate the setting of the mixture. The release of hydrogen in the cast aerated concrete mass is slower and the concrete microstructure is better. Sulphates influence the shape of tobermorite crystals - they are larger and flatter [13].

This additive has been used in AAC technology for years to improve the properties of this material. Reducing the sulfate content in the AAC mixes leads to increased shrinkage and reduced compressive strength [14]. Calcium sulphate in AAC during hydrothermal treatment accelerates the formation of tobermorite and C-S-H phase, which in turn increases the strength of the final product [12]. As a result, the structure of the concrete becomes tighter and the sorption of concrete is reduced directly after hydrothermal process as the gypsum content in the mix increases. However, it is important to remember that built-in AAC units often have a water content of up to 40% and, as the concrete tightness increases, the process of drying of AAC walls slows down. The aim of this study was to evaluate the drying process of AAC specimens depending on the content of calcium sulphate in the binder.

2 Description of the production method and composition of AAC specimens

AAC specimens were prepared in a prefabrication plant using UNIPOL sand technology, which uses the following raw materials: cement, lime, gypsum, quartz sand, aluminum powder. In this method one of the technological processes is a combined dry grinding of cement, lime and quartz sand in a ball mill to a specific surface of 400-600 m^2/kg [9]. Quartz sand is introduced into the mill after screening the oversize grains. During the grinding of the components the temperature is 80-90°C, which is the result of friction and exothermic reaction of water from damp sand with active calcium oxide CaO contained in burnt lime.

For research purposes, five concrete compositions with different gypsum content were designed and produced. The designed dry density of all AAC series was 520 kg/m^3. Portland cement CEM I, medium-burnt lime and quartz sand containing over 90% of silica SiO_2 were used for the production. Table 1 shows the composition of particular AAC series.

The classical hydrothermal treatment method (without vacuum in the first phase) was used in the manufacturing process. The whole hydrothermal process lasted 12 hours. Samples for the experiment were taken directly after autoclaving.

3 Experimental methods

The dry density of concrete specimens was determined according to [15] and the compressive strength according to [16]. The tensile strength test was carried out in accordance with the standard procedure [17] on dried 100 mm cube specimens prepared in the same manner as for compressive strength tests - cut from masonry units in the three zones: top, middle and bottom. The individual samples were loaded perpendicular to the direction of mass growth by applying a load through 2.0 cm wide steel bars located in the middle of a cube wall. Moisture content test was performed according to [18].

Capillary water absorption rate data were obtained using the standard procedure [19] in which cubes 100×100×100 mm of initially dry material were placed in contact with water (5±1 mm) in a shallow tray. The side faces were sealed with epoxy resin. Water was absorbed through the 100×100 mm bottom face. The weight gain of the sample was measured after 10, 30 and 90 minutes. Water absorption coefficient of masonry units, due to capillary action, is determined from Equation:

$$C_{ws} = \frac{m_{so,s} - m_{dry,s}}{A_S \sqrt{t_{so}}} \bullet 10^6 \ \ [g/(m^2 \times s^{0,5})] \qquad (3.1)$$

where: C_{ws} – water absorption coefficient of masonry units due to capillary action, $[g/(m^2 \star s^{0,5})]$; $m_{dry,s}$ – dry mass [g]; $m_{so,s}$ – mass after saturation time t [g]; A_s – area of contact surface [m^2]; t_{so} – saturation time [s].

Table 1: Composition of AAC per 1 m^3 of the mixture

Series	AAC0	AAC1	AAC2	AAC3	AAC4
Gypsum (calcium sulfate dihydrate) [% of dry mass]	0	0.55	1.15	2.30	3.50
Cement + lime [kg]	143.0	145.0	141.0	136.0	133.0
Quartz sand [kg]	348.0	350.0	347.0	345.0	340.0
Added gypsum [kg]	0	2.7	5.6	11.1	16.6
Gypsum in cement [kg]	3.7	3.8	3.7	3.5	3.5
Total gypsum content [kg]	3.7	6.5	9.3	14.6	20.1
Aluminum powder [kg]	0.42	0.42	0.42	0.42	0.42
water/dry components ratio	0.49	0.50	0.50	0.51	0.50

The procedures described in the standard [17] was applied for determination of the maximum water absorption and capillary rise.

Concrete maximum water absorption test was performed according to the following test procedure:

- the samples were initially, for 24 hours, put in water at 1/3 of the sample height;
- after 24 hours, the water table was increased to 2/3 of the sample height for another 24 hours;
- then the samples were completely immersed in water for another 24 hours;
- after the total time of 72 hours the maximum water absorption was determined.

The state of full saturation simulates the situation when material is totally submerged in water, for example, because of floods or long-term effects of rainwater.

For capillary rise test the prisms 100 × 100 × 200 mm were dried to constant mass at 105±5°C, and then after cooling they were placed in contact with water (30 mm). Water level was maintained constant throughout the experiment. Measurements of capillary rise were made after 1, 7, 24, 48 and 72 hours. The rise heights were measured from the water line at the center of each of the four prism side faces of the sample. The capillary rise of individual samples was determined as the arithmetic mean of the measurements on the four side faces of the samples. The samples were prepared from the whole masonry units, three samples from each unit in the same way as for density and strength tests.

The temperature and relative humidity (RH) during the concrete drying process were chosen assuming that under operational conditions the heated rooms for permanent residence have the temperature 20±2°C and the average RH is 50-65%. Testing of the drying rate from the moisture residue after the autoclaving process was carried out in laboratory conditions at temperature 22±1°C and RH in the range 50-55%.

Because of the significant moisture content in concrete as a result of the full saturation of its pores with water, the drying test was carried out under laboratory conditions at 25±1°C and RH 30±1% in a forced air circulation laboratory oven. Samples were dried until the stabilized moisture content of about 6% was reached.

4 Experimental results and discussion

The results of the performed examinations are presented in Table 2. The relationship between gypsum content and the compressive strengths of AAC specimens is given in Figure 1.

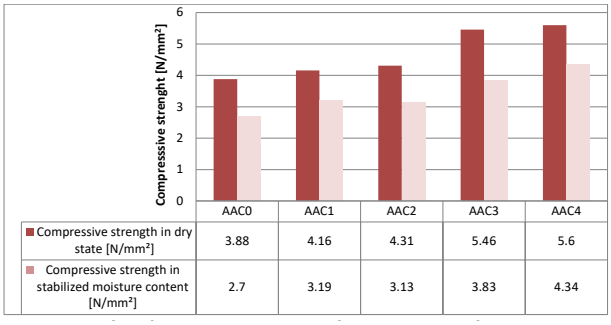

Figure 1: Dependency between gypsum content and compressive strength

The compressive strength tested on both dried and stabilized moisture specimens as well as tensile strengths increases with the increase of gypsum content. This increase is significant when gypsum content exceeds 2% of dry weight.

There was no significant effect of sulphate addition on the maximum water absorption of AAC samples after

Table 2: Test results of AAC specimens

Series		AAC0	AAC1	AAC2	AAC3	AAC4
Dry density [kg/m^3]		548	520	510	514	541
Compressive strength in dry state [N/mm^2]		3.88	4.16	4.31	5.46	5.60
Compressive strength at stabilized moisture content [N/mm^2]		2.70	3.19	3.13	3.83	4.34
Tensile strength [N/mm^2]		1.1	1.1	1.3	1.4	1.6
Stabilized moisture content [%]		5.1	4.4	4.8	5.4	4.9
Time of moisture stabilization [h]		21.0	25.0	30.0	44.0	58.0
Maximum water absorption [%]		64.0	59.7	61.8	56.4	60.1
Water absorption [g/(m^{2}s$^{0.5}$)]	after 10‘	185.4	148.1	151.4	91.6	116.1
	after 30‘	150.8	113.5	120.3	77.9	94.3
	after 90‘	125.0	91.3	95.1	65.9	74.7
Capillary rise [cm]	after 1h	2.3	1.9	1.6	1.3	1.7
	after 7h	4.1	3.1	2.3	1.8	2.3
	after 24h	6.2	4.7	3.8	2.7	3.1
	after 48h	8.6	6.5	5.4	3.8	3.9
	after 72h	**7.4**	**10.0**	**7.3**	**4.4**	**4.4**
Change of moisture as a result of drying out from the moisture after the production process [%]	after 0 h	29.6	28.2	29.8	26.9	29.6
	after 24 h	19.1	20.4	21.8	22.3	24.8
	after 48 h	12.0	12.8	14.4	16.0	18.4
	after 72 h	9.2	9.9	11.5	13.1	15.6
	after 96 h	7.7	8.3	9.9	11.8	14.0
	after 120 h	6.1	6.5	8.1	9.9	12.2
	after 144 h	5.2	5.2	6.8	8.5	10.8
	after 168 h	4.4	4.4	5.6	7.3	9.6
	after 184 h	3.8	3.8	4.8	6.6	8.7
	after 200 h	3.6	3.4	4.4	6.0	8.2
	after 224 h	3.3	2.8	3.5	4.9	6.9
	after 264 h	**3.1**	**2.4**	**2.9**	**4.1**	**6.1**
Change of moisture as a result of drying out from maximum water content [%]	after 0 h	64.0	59.7	61.8	56.4	60.1
	after 6 h	59.2	54.4	57.7	52.4	55.5
	after 12 h	51.7	45.8	50.9	45.9	48.5
	after 18 h	44.3	38.2	45.1	39.8	42.1
	after 24 h	36.6	31.0	38.8	34.4	36.3
	after 30 h	28.4	24.4	32.1	28.8	31.5
	after 36 h	22.5	20.0	26.2	24.8	27.5
	after 42 h	17.7	16.9	22.0	21.8	24.5
	after 48 h	14.2	14.3	19.1	19.6	22.3
	after 54 h	**12.3**	**12.8**	**17.1**	**17.9**	**20.5**

72 hours, though all specimens containing gypsum have lower absorption compared to the reference concrete.

Effect of the gypsum addition is apparent in case of absorption after 10, 30 and 90 minutes and capillary rise. Figure 2 shows the experimental results of the absorption rate w^* (expressed as cumulative absorbed mass/unit inflow area versus $t^{0.5}$). For 90 minutes only the first stage of water sorptivity was tested - an early time stage where absorption rate w^* is linear in $t^{0.5}$. It is evident that even small amount of added gypsum reduces sorptivity of AAC. The curve constructed for the reference specimens is steeper and the total mass increase is almost two times higher than for the specimens with sulphate content exceeding 2% of dry mass.

There is a strong dependency between capillary rise and gypsum dosage (Figure 3). As with mechanical properties, the effect is more significant when gypsum is dosed above 2% of dry mass. Capillary rise after 72-hour test de-

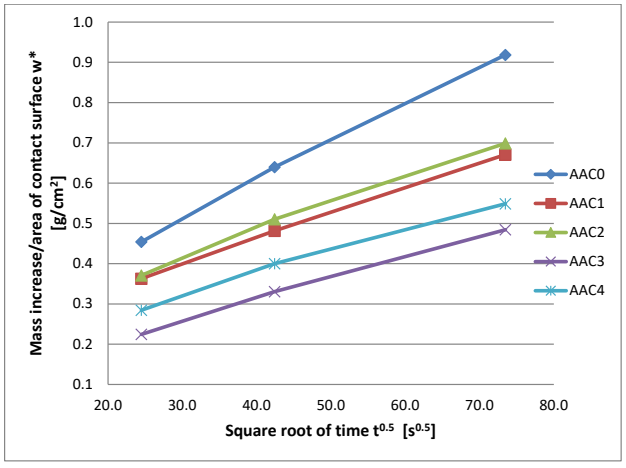

Figure 2: Capillary water absorption of AAC specimens (cumulative absorption w^* versus $t^{0.5}$) depending on gypsum content

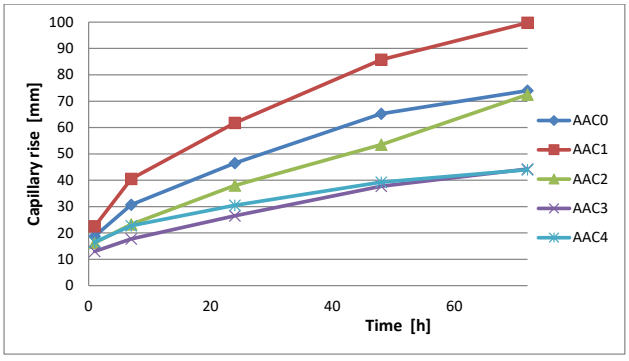

Figure 3: Capillary rise of AAC specimens depending on gypsum content

creased by approximately 40% compared to the control samples.

It was observed, however, that the rate of drying from moisture residue after production process of the concretes containing hydrated calcium sulphate was slower compared to the reference samples (Figure 4). Moisture content in samples containing 3.5% of gypsum was about twice as high as in the control concrete after 264 hours of drying at 22±1°C.

Moisture content in the specimens with 3.5% of gypsum was over 60% higher than in the reference series (20.5 versus 12.3%) after 54 hours of oven drying from the maximum water absorption, though the starting point was lower (60.1 versus 64.0%).

It is known that the crystallinity of tobermorite largely affects the physical properties of AAC. The differences in microstructure of the tested AAC specimens, resulting in different performance features, are due to the binder composition. Evidently addition of calcium sulfate dihydrate modified the material mictrostructure.

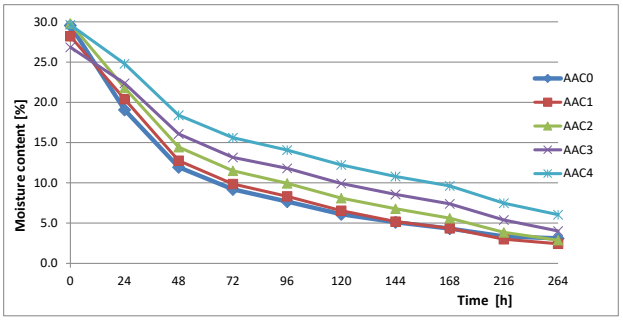

Figure 4: Influence of gypsum dosage on the drying process of AAC at 22°C from the moisture resulting from the production process to the stabilized moisture content

5 Conclusions

AAC that was the subject of this study was manufactured on an industrial scale, which minimized possible errors in the process of dosing, mixing of components or hydrothermal treatment. The whole process was automatically monitored. By modifying the composition of concrete binders, we can modify the properties of cellular concrete and hence the properties of the masonry elements intended for building partitions.

AAC specimens in which calcium sulfate dihydrate content exceeded 2% of dry mass had, compared to gypsum-free specimens:

- compressive strength higher by approximately 40% in the dry state and 60% higher in the stabilized moisture content;
- tensile strength higher by approximately 40%,
- lower water absorption,
- capillary rise lower by approximately 40%.

It was observed, however, that when the gypsum content was higher than 2% of dry mass, the drying from the moisture residue after manufacturing process was slower and after 264 hours the moisture content was still higher than 4%. Calcium sulphate affects the process of crystallization of tobermite and C-S-H phase, which is confirmed by the results of strength tests. As a result, AAC3 and AAC4 specimens are characterized by a different porosity structure compared to the reference concrete.

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