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Influence of Micro additives on Macrostructure of Autoclavedaerated Concrete

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Abstract: *Autoclaved aerated concrete (AAC) is a construction material with porous macrostructure prepared of finely milled siliceous aggregate, lime, water and Portland cement with the addition of aluminium powder and being steam-cured under the pressure in an autoclave. Today AAC (light construction material) is a widely used in the constructions as a material that is compared to concrete or conventional stone material, which has a high insulation properties and fireresistant. Previous studies have shown that application of microadditives in AAC enhances physical-mechanical properties of AAC samples. Most of these changes are duo to an even distribution of pores. The influence of such additives, as amorphous SiO_2 (AS) and carbon fibres (CF), reduced to microsizeparticles, on formation of AAC macrostructure was investigated. The investigations were carried out with AAC forming mixture where 10% lime was replaced by equivalent content of Portland cement. Research results have showed, that the optimal replacement of sand by AS was 1.0% and by CF – 0.1%, and resulted in an equal distribution of pores and optimal macrostructure formation (higher amount of small pores, lower amount of merged pores) which leads to highest compressive and flexural strengths of modified AAC samples.*

Keywords: *autoclaved aerated concrete, amorphous SiO_2, milled carbon fiber, microadditive, macrostructure, area of pores, perimeter of pores, roundness of pores, compressive strength, flexural strength.*

I. Introduction

The energy consumption and the resulting CO_2 emission during the use of buildings are much greater than those involved in the production of the building materials. [1] It was recently estimated [2] that the energy consumption during the operation of buildings accounts for more than 40% of the total energy consumption of the country, which corresponds to approximately 13% of global CO_2 emissions. Considering global CO_2 and resource conservation issues, the demands to reduce the energy consumption in buildings have become greater. For example, since 2013, the heat transfer coefficient of exterior walls in residential buildings in South Korea has strengthened from 0.27 $W/m^2 \cdot K$ to 0.16 $W/m^2 \cdot K$. [3]To minimize cooling and heating loads in buildings, AAC blocks are widely used as a wall material. [4]

AAC is a special kind of concrete, where Portland cement, lime, water, sand and a blowing agent are mixedtogether. [5] The reaction triggered by the addition of the blowing expansive agents causes microscopic hydrogen bubbles to form, expanding the concrete to about two times its original volume within 3 h. After evaporation of the hydrogen, aerated concrete is cut to size and form and it is steam-cured in a pressurised chamber, known as autoclave. [6] After curing we get a non-organic, non-toxic, airtight, non-combustible, fireresisting material, characterised by its fine cellular structure, with air pores ranging from 0.1 to 2 mm. [6]

AAC was born in the 20s in Sweden as an alternative material used for constructions. Subsequently, it spread worldwide as an eco-friendly material and with a negligible environmental impact because it is completely mineral and produced with greatly abundant components. [7]

AAC is a lightweight material in which a uniform cellular structure of air voids distributed throughout a matrix of AAC of mortar. [8] With extremely low density (500 kg/m^3) and thermal conductivity (0.1 $W/m \cdot K$), AAC is an ideal material for thermal insulation and sound-proofing. [9, 10] Porosity of AAC is the most interesting feature, whereby this material presents- lightness, ease of transport and installation, ductility, thermal insulation properties, acoustic insulation properties, transpiring properties. [5]

Various types of additives were used to improve the structure and properties of AAC. [11, 12] Two microadditives – amorphous SiO_2 and milled carbon fibreswere used to modify macrostructure of AAC in this paper.

AS is a very effective pozzolanic material. As an aggregate, AS powder is in particular suitable for modern building industry. It was used at construction since 1994 in New Zealand and with each year, its usage grew. AS is a by-product of ferrosilicon and silicon metal production and can be used in shape of very fine powder. [13] In production of concrete with AS, the pozzolanic reaction is running when SiO_2 with specific surface, which can be as high as 200000 cm^2/g, and with high content of amorphous silica (usually about 90%) [14]links $Ca(OH)_2$ present in solution, in the same way as active pozzolanic additives containing amorphous SiO_2 of opal

origin. This additive defines the process of cement hardening and modifies the microstructure of concrete by making it more homogenous and by reducing big pores in number, while the total porosity remains same as that of concretes without additives, as well as decreases conductivity of water and water vapour and increases strength and life of concrete. [15] CF in concretes are mostly used as a reinforcing additive for improvement of their strength properties, in particular those under flexure and compression. Furthermore, the sources [16, 17] maintain that this additive adheres pore walls and thus ensures the transfer of load through hollows. As an additive for AAC, CF was used since long and many sources [11, 16, 17] state that this additive enhances strength properties of AAC. In all abovementioned sources [11, 16, 17, 18] CF was used as a reinforcing additive without considering its possible impact when reduced to microsize particles. The production of AAC is rather complicated and the properties of its outcome are subject not only to AAC composition, but also to parameters of forming mixture, what affects the formation of the macrostructure. The purpose of this work was to estimate the impact of microadditives AS and CF on the macrostructure of AAC parameters such as: cross-section area, perimeter, roundness of pores and evaluate there impact to compressive and flexural strengths.

II. Materials and Methods

The composition of AAC forming mixture was the following: binding material, lime 90%, and 10% lime replaced by equivalent amount of Portland cement according to the formula:

$$K = \frac{44 \cdot C}{A_0}, \qquad (1)$$

where: *44* – a coefficient of recalculation at 22% activity of mixture, *C* – amount of Portland cement replaced by lime, kg, A_0 – activity of lime, %. [19]

The AAC forming mixtures were prepared using the mentioned below raw materials. Ground lime from JSC "NaujasisKalcitas" (Lithuania), corresponding to the requirements of standard EN 459-1:2010, activity 90%, duration 5 minutes, temperature 55 °C. Portland cement CEM I 42.5 R from JSC "AkmenėsCementas" (Lithuania), with specific surface of 3190 cm^2/g, determined by the Blein device (Germany), and volume stability (expansion) of 0.5 mm, determined by the Le-Chatelier device (France), corresponding to the requirements of standard EN 197-1:2011. Mineral composition of clinker (in %): C_3 – 58.54; C_2S – 15.29; C_3A – 10.40; C_4AF – 10.17. The chemical composition of clinker is provided in Table 1.

Table 1. Chemical composition of raw materials and additives

Raw materials and additives	Composition (in mass %)										Other, %
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	SO_3	R_2O	C	
Lime	4.05	2.21	0.88	89.24	2.38	-	-	-	0.37	0	0.87
Portland cement	20.76	6.12	3.37	63.50	-	1.0	0.3	0.8	-	-	4.15
Quartz sand	97.89	0.60	0.46	0.72	-	-	-	0.06	0.10	-	0.17
AS	98.00	-	0.05	0.25	0.4	1.2	0.1	0.35	-	0.6	0.00
CF	3.61	0.13	-	0.26	0.09	-	-	-	-	95.80	0.11

Quartz sand from JSC "AnykščiųKvarcas" (Lithuania), grounded in ball mill up to fineness of 2766 cm^2/g. The fineness was determined by Blein device according to the requirements of standard EN 196-6:2010.As a gas-generating agent in AAC forming mixtures, aluminium paste (Al) "AlboSchlenkDeg 4508/70" (Czech Republic) was used, specific surface 18000 cm^2/g, pure aluminium content in paste 70%.The following microadditives were used in the work. AS having pozzolanic properties "RW-Füller" (Germany), the chemical composition of which is provided in Table 1. It has been used 0.5, 1.0 and 1.5% counting of sand mass. CF ground to microparticles, selected in consideration of its resistance to alkaline medium and temperature. The grounding of CF went on for 10 hours in ball mill "Fritsch Pulverisette 7" (Germany) using agate balls of 16 mm diameter, speed 600 rpm. It has been used 0.05, 0.1 and 0.2% counting of sand mass. For distribution of mentioned additives in AAC forming mixtures, the surfactant "Ufapore TCO" (Norway) was used in content of 0.003% counting of solids mass. The compositions of AAC forming mixtures were selected basing on the methodical requirements. [20] The activity of mixture expressed by content of active CaO and MgO was 22%. The W/S ratio was 0.54, content of Al 0.18% (counting of solids mass). The components of forming mixtures were mixed in the vertical propeller stirrer of four litters by speed of 700 rpm. The duration of component mixing is presented in Table 2.

Table 2. Sequence of components dispensing and duration of mixing for production of AAC samples

Components	Duration of mixing, min
1. Water + sand	5.0
2. Water + sand + Portland cement	1.0
3. Water + sand + Portland cement + lime	1.0
4. Water + sand + Portland cement + lime +Al	1.0
5. Water + sand + Portland cement + lime +Al + AS or CF (mixed with surfactant)	1.0
	Overall: 9.0

For even distribution of microadditives and Al in the mixture, they were dispersed separately by ultrasonic disperser "UZDN-21" (Russia), frequency 22 kHz, capacity 480 W. The dispersion was carried out after mixing microadditives with water and surfactant, the duration of process 1 min. Al after mixing with water was dispersed for 3 min. For production of AAC, the equal W/S ratio (0.54) was used. The spreadability was tested by "Suttard" viscosimeter (Russia) according to the methodical requirements. [21] Macrostructure's investigations wascarried out by examiningcross-sectionalsurface of theAAC sample. Formed AAC Samples werecut, sanded, treated with compressedairfrom dust. Photo images were taken using Optical microscope "Motic" which magnified 12 times. The flatarea of the sample was examined by "UTHSCSA Image tool" and "Pixcavat or Image Analysis" software's. Poreroundness of theAAC sample'scross-section, whenaround pore is equal to 100%, as well as theperimeter, area, amount of pores was determined. All components amount for 0.01372 m^3 volume shown in Table 3.

Table 3. Amount of components AAC forming mixture in grams for 0.01372 m^3 volume

Components	Amount (in grams)						
Lime	195	195	195	195	195	195	195
Portland cement	19.5	19.5	19.5	19.5	19.5	19.5	19.5
Sand	580.3	577.4	574.5	571.6	579.9	579.5	578.7
AS	-	2.90	5.80	8.70	-	-	-
CF	-	-	-	-	0.40	0.80	1.59
Al	1.43	1.43	1.43	1.43	1.43	1.43	1.43
Surfactant	0.02	0.02	0.02	0.02	0.02	0.02	0.02
Water	430	430	430	430	430	430	430

The prepared AAC forming mixtures were poured into molds of (70×70×70) mm for compressive and (40×40×160) mm for flexural strengths. The expanded forming mixture was kept in molds for 3.0 h, then, after cutting off the heap of forming mixture, the molded AAC samples were hardened in the laboratory autoclave of 100 liters capacity, following the specified mode of hardening (5+10+5) h, at water vapor temperature of 176 °C what corresponds to pressure of 0.8 MPa. After hardening, the AAC samples were dried to constant mass in the conditioning oven at temperature of 105 °C ±5 °C. The compressive and flexural strengths of AAC samples were determined according to the requirements of EN 679:2005 and EN 1351:1996. The press "TINIUS OLSEN H200KU" was used. The loading rate of samples during compression was 60.0 N/s, and bending – 14 N/s. 3 samples of each batch were subjected to testing. As actual densities of samples from various batches varied, the obtained actual compressive and bending strength values were recalculated according to the technique [22] for selected density 450 kg/m^3.

III. Results

3.1. Macrostructure

Established, that AAC with different amount and different type of microadditives had affect to AAC macrostructure. Examined area – 49 mm^2 (7 mm x 7 mm). A small amount of the AS additive (up to 0.5%) had no significant effect on structure (Fig. 1, b) comparing to AAC without additive. Using 1.0% AS in AAC samples has created equal, small, closed pores (Fig. 1, c) and addition up to 1.5% of this additive began to change structure, formed various sizes merged pores (Fig. 1, d).It is considered, that adding more than 0.1% of very fin (specific surface $20 \cdot 10^3$ m^2/kg) and active AS, and without changing the W/S ratio, was accelerated reaction of cement hydration, which increases the temperature and expansion of the formation mixture.An increase of high specific surface area of the AS particles in the same W/S of the mixture decreased plasticity. Due to this reason (Al intense reaction) disintegrated the macrostructure during extension (formed merged pores), so in this case, the optimal amount of AS would be 0.1% (counting of sand mass), comparing to AAC without additives.Adding CF into AAC (Fig. 1, e-g), had no significant visual changes of AAC macrostructure, only one could see, that distribution of the pores were more equal and their diameter more uniform, comparing with the AAC without additives (Fig. 1, a).

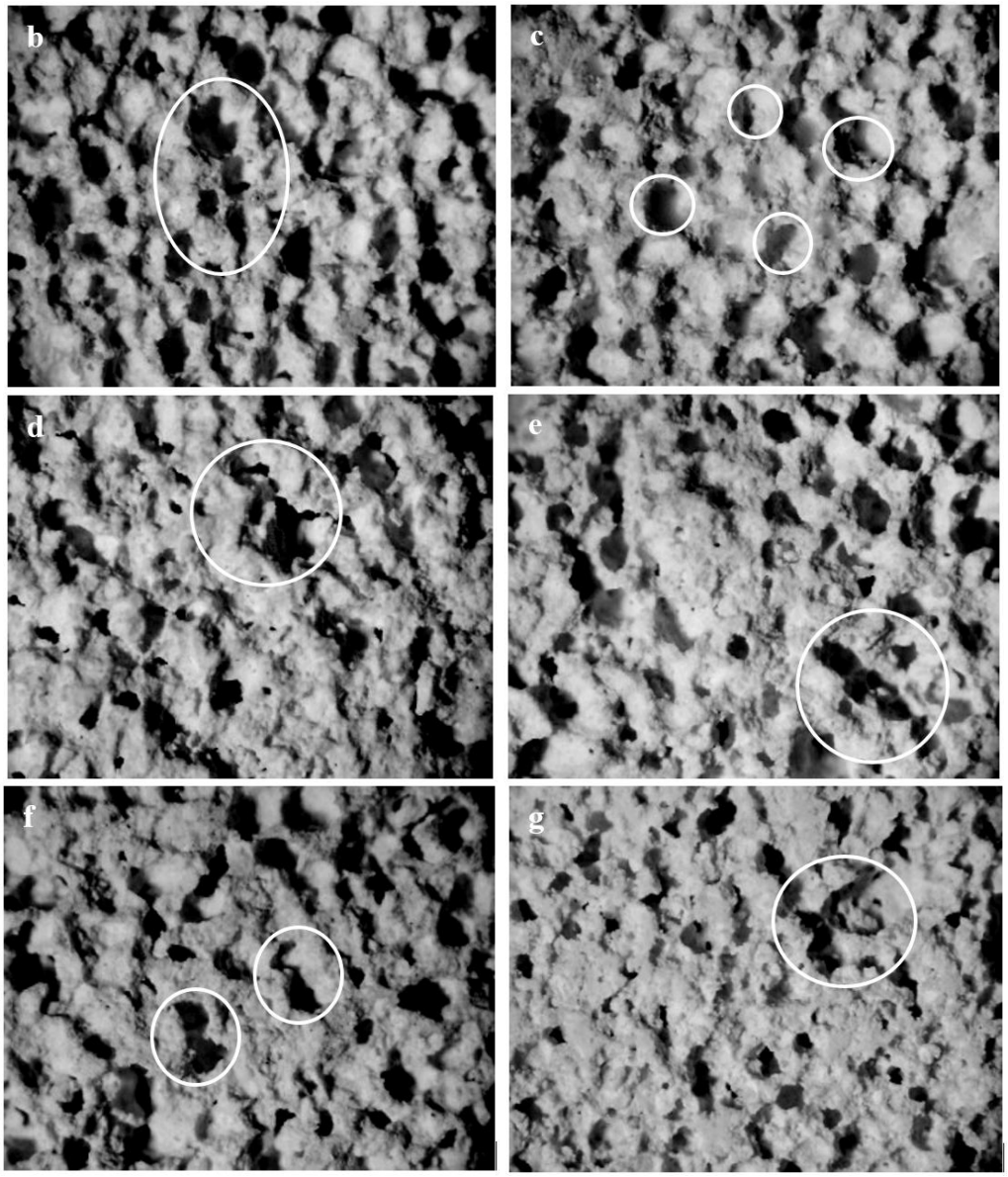

Fig. 1. Pore distribution in AAC samples at the content of additive by %: a – 0; b – 0.5 AS; c – 1.0 AS; d – 1.5 AS; e – 0.05 CF; f – 0.1 CF; g – 0.2 CF. Magnification ×12

Adding 0.05% (Fig. 1, e) or 0.2% (Fig. 1, g) CF formed quite a similar amount an shape of pores, however, it formed more rounder and merged pores comparing with AAC samples without additives.AAC porosity (merged pores, equal distribution throughout the sample) results mechanical properties (compressive and flexural strength) of AAC samples.

3.2. Mechanical properties of AAC samples

The results have showed that AS and CF additives have an effect on the compression and flexural strength of the AAC samples. Results were recalculatedaccording to the technique [22] for selected density 450 kg/m^3. The largest increase of compressive strength was using 1.0% of AS (Fig. 2a, column 3), compressive strength was 20% higher than AAC without additives (Fig. 2a, column 1). Using a larger AS amount (1.5%), compressive strength decreased by 8% (Fig. 2a, column 4), comparing to AAC with 1.0% AS (Fig. 2a, column 3). This led to the change of technological parameters of the AAC formation mixture, this is due to very fin AS additive and without changing the W/S ratio. In this case, it decreased compressive strength. The tests showed, that the biggest effects of the flexural strength of AAC samples was using the optimal amount of AS additive (1.0%), flexural strength increased by 31% (Fig. 2b, column 3) comparing with AAC without additives (Fig. 2b, column 1). Using larger amount of AS (1.5%), flexural strength decreased approximately 6% (Fig. 2b, column 4), comparing with AAC sample with 1.0% AS additive.

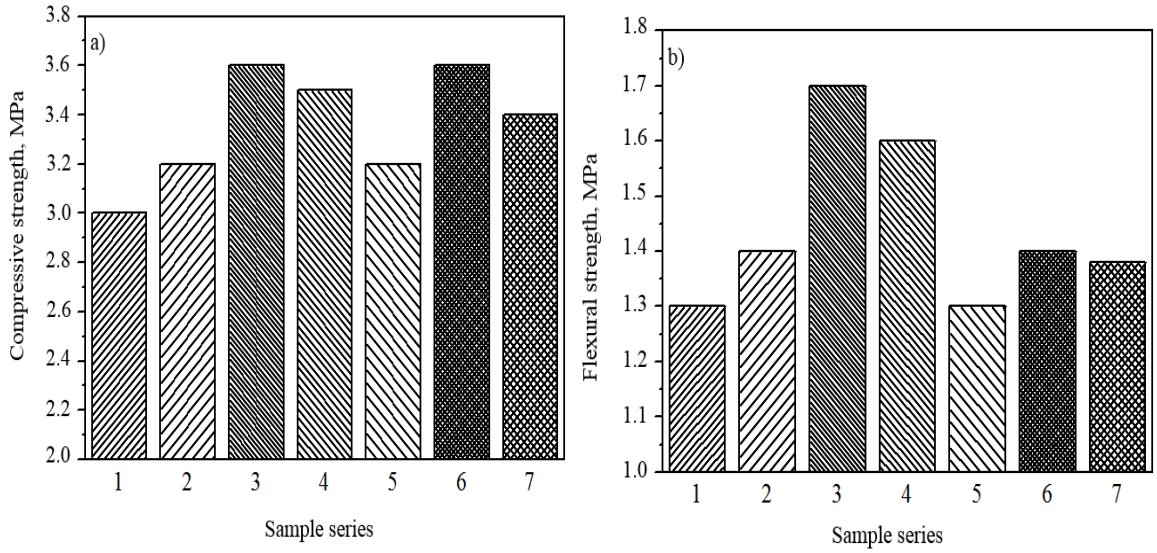

Fig. 2. Compressive strength (a) and flexural strength (b) of AAC samples at the content of additive by %: 1 – 0; 2 – 0.5 AS; 3 – 1.0 AS; 4 – 1.5 AS; 5 – 0.05 CF; 6 – 0.1 CF; 7 – 0.2 CF

Different amounts of CF effected different the compressive strength of AAC samples. Research results presented in Figure 2a, 5-7 columns. CF additive in AAC sample ranged from 0.05% to 0.2% (counting of sand mass), the highest compressive strength obtained with 0.1% CF, it increased by 20% (Fig. 2a, column 6) comparing with AAC without additives. With a larger (0.2%) (Fig. 2a, column 7) or lower (0.05%) (Fig. 2a, column 5) amount of CF additives in AAC samples compressive strength was not significant. Using 0.2% of CF additive compressive strength decreased approximately 6% comparing with AAC with 0.1% of CF additive.
Effects of different amount CF additive on AAC flexural strength presented in Figure 4b, 5-7 columns. With different amount of CF additive (0.05%, 0.1%, 0.2%) flexural strength evolved accordingly: 0%, 8%, 6% (Fig. 2b, columns 5-7) comparing with AAC without additives.
Compressive and flexural strengths for AAC samples with AS and CF additives increased duo to more even distribution of pores, it confirmed earlier results of submitted researches. This results confirmed researches of macrostructure, that optimal AS amount was 1.0% and CF – 0.1%.
Evaluated mechanical properties and macrostructure researches of AAC with AS and CF additives were carried out more detailed investigations in macrostructure such as amount, their area, perimeter and roundness of pores. The results presented in further research.

3.3. Cross-section area of pores of autoclaved aerated concrete samples

Investigations of AAC sample of the 49 mm^2 area shows the proportion takes by the voids and carcass. Area of pores of AAC without additives and with optimal amount of additives cross-section was calculated, because in previous researches (macrostructure and mechanical properties) other amount of additives had no significant effect. Structure of the AAC depends on size and amount of the pores. Point graphics in Figure 2 shows: Y-axis delayed area of pores, and X axis - amount of pores of the AAC samples. In the sample of AAC without additives (Fig. 3, curve 1) was dominated larger diameter pores, but the amount of pores was low (49 mm^2 is ~ 45 pores), a total area of pores of AAC sample was approximately 20 mm^2. It can be presumed that larger diameter of the pores leads to less dense carcass and deteriorating mechanical properties of AAC samples. Using 1.0% of AS additive in AAC sample dominated small pores (a pore of the largest area of the sample was up to 0.2 mm^2), and their amount increased about four times (165 pores) (Fig. 3, curve 3) comparing with AAC without additives.
We can assumed that the AS additive for regular, smooth surface of the spherical structure, during expansion, can reduce the friction between the larger particles in the forming mixture, so the size of the pores obtained smoother. Structure with the large amount of small pores leads to create denser carcass, which allows to obtained higher mechanical properties of the AAC samples. An assumption is that denser carcass of AAC sample leads to more equal stress distribution at the test (compressive and flexural strength). AAC with CF additive (Fig. 3, curve 6) shows that pores become about 30% smaller (in the 49 mm^2 area dominant 75 pores, the size varied from 0.1 to 0.7 mm^2) comparing with AAC sample without additives, but the macrostructure was not so equal as with AS additive.As AAC with AS additive, so with CF additive area of pores was 15% lower, although amount of pores increased, comparing with AAC without additives.

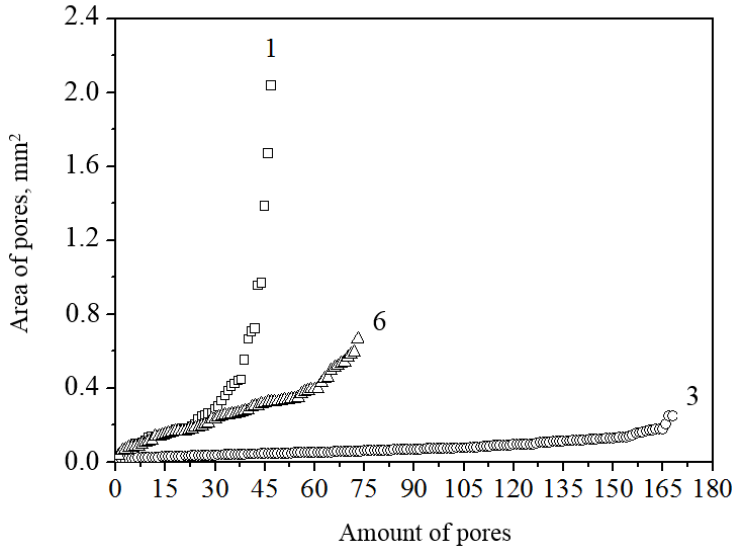

Fig. 3. Area and amount of pores in AAC samples at the content of additive by %: 1 – 0; 3 – 1.0 AS; 6 – 0.1 CF

Research has showed that the both additives increased amount and reduced the area of pores of the AAC samples, therefore it is concluded, that the use of additives produces a large amount of small closed pores. These results confirmed the visually research of the macrostructure. It is believed, that at very fine AS and without changing the W/S ratio increased AAC forming mixture expansion, thus changed the structure of the AAC samples.

3.4. Cross-section perimeter of pores of autoclaved aerated concrete samples

According to values of perimeter of pores may indicate the amount of the merged pores. If two pores of the same diameter were connected, than the perimeter of the individual pore will be less than two pores, but the area of two pores will be similar. Point graphic in Figure 3 shows, that one point is one perimeter of the one pore. It is estimated, that in investigated 49 mm^2 of AAC sample without additives cross-sectionarea were formed about 45 pores, with a total perimeter - 55 mm and a largest perimeter of pore was 3.6 mm, lowest - 0.18 mm.

Calculations of the perimeter of pores in investigated 49 mm^2 of AAC cross-sectionarea established, that adding 1.0% of AS additive density of the pores increased four times and total perimeter of pores − 171% (Fig. 4, curve 3), comparing with AAC samples without additives (Fig. 4, curve 1). Amount of the pores increased twice by the using 0.1% of CF in AAC, perimeter − 77% (Fig. 4, curve 6), comparing with AAC without additives.

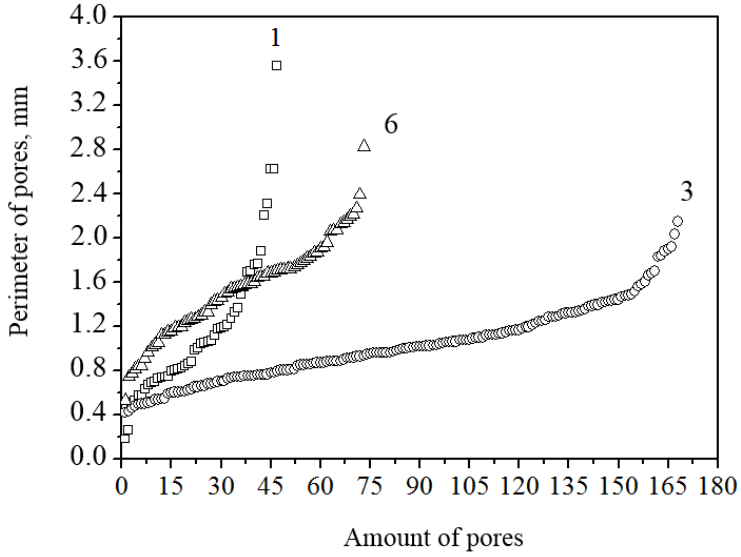

Fig. 4. Perimeter of pores and amount in AAC samples at the content of additive by %: 1 – 0; 3 – 1.0 AS; 6 – 0.1 CF

Addition of the AS or CF additives to the AAC samples, formed many smalls, rounder, equal distributed pores, with diameter from 1.0 mm to 0.06 mm. This confirmed results of the "Cross-section area of pores of autoclaved aerated concrete samples" section.

3.5. Cross-section roundness of pores of autoclaved aerated concrete samples

Roundness as a perimeter of pores have showed amount of the merged pores in the AAC sample. If the pores are merged, so there roundness are smaller than separate pores. Examining cross-section of AAC samples roundness of the perfectly round shape pore would be equal to 100%.

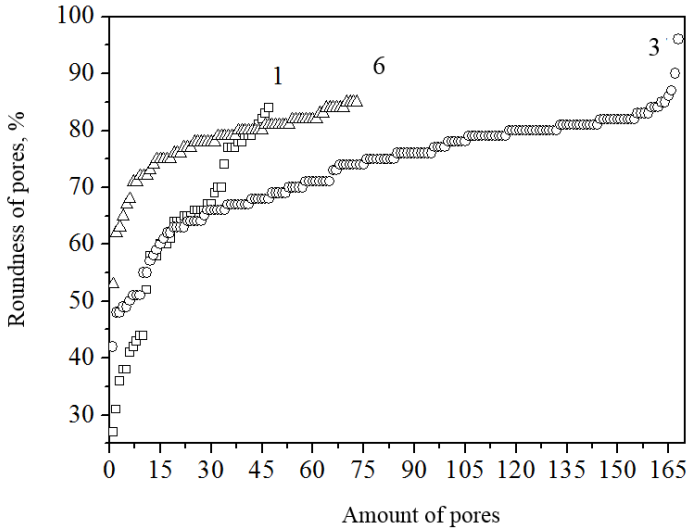

Fig. 5. Pores roundness % (where round pore equal to 100%) and amount in AAC samples at the content of additive by %: 1 – 0; 3 – 1.0 AS; 6 – 0.1 CF

Average of the pores roundness of AAC samples without additives was equal to 63% (Fig. 5, curve 1). It was determined that using 1.0% of AS in AAC, rounder pores were received and roundness average of the pores were 60% higher than AAC samples without additives, while perfectly round roundness of pore was even 96% (Fig. 5, curve 3). Using 0.1% of CF in AAC roundness average of the pores were 38% bigger than AAC samples without additives, while perfectly round pore roundness − 84% (Fig. 5, curve 6). Changes in macrostructure were greater in AAC with AS than CF additives. Using AS additive formed smaller and less merged pores. These results confirmed of previously submitted cross-section pores area and perimeter of autoclaved aerated concrete samples results. It was determined that formation of less communicating pores in the AAC samples was duo to usage of very fin AS and CF additives. Cross-section amount, area, perimeter and roundness of pores confirmed earlier researches (macrostructure and mechanical properties). It can be presumed, that higher amount of merged pores leads stress distribution in lower quantity of cross-section elements.

IV. Conclusions

The macrostructure analysis has showed, that the optimal amount of very fine AS additive is 1.0%, because higher (1.5%) amount of this additive has damaged AAC macrostructure, duo to formation of AAC more merged pores have been formed and it had influence to mechanical properties. Small amount (0.5%) of AS additive had no significant effect on the macrostructure and mechanical properties of AAC samples.

Using CF additive, its optimal amount is 0.1%, using this amount in AAC samples, it increased mechanical properties and improved macrostructure. The higher (0.2%) or lower (0.05%) amounts of CF additive had no significant effects to mentioned characteristics.

Optimal amounts of AS or CF additives have improved the macrostructure of AAC samples, formed smaller, equal distributed, closed pores, which formed dense carcass, what leaded to incensement of compressive and flexural strengths of AAC samples.

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