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Investigation of the Composite Material with Inclusions of Autoclaved Aerated Concrete Chips

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In production of autoclaved aerated concrete (AAC) units, after mechanical processing and hardening in autoclaves, the waste of hardened product is unavoidably generated. Till now this waste has no utilization. The investigations within this work showed that the crushed AAC waste, sized from 2.5 mm to 10.0 mm, can perform the function of a lightweight aggregate in development of the composite with density of ≤ 850 kg/m^3 and compressive strength up to 4.0 MPa. The matrix of this product is composed of hardened Portland cement and modifying additives (complex additive, CA). It was established that, thank to CA, in the structure of the composite, the AAC granules are coated by a thin layer of cement stone, 50.0 μm thick, and the properties close to those of AAC are ensured.

Keywords: AAC chips, Portland cement binding material, super plasticizer, air entraining additive (AEA), SiO_2 micro dust, operational properties.

INTRODUCTION

One of the promising and effective directions in the modern construction is ever widening application of various additives of organic and inorganic origin in production of concrete. An additive of one tenth or one hundredth per cent to cement mass can essentially change the chemical hardening of concrete, increase its mechanical strength, improve the whole spectrum of physical-technical properties, i. e. reduce water adsorption, increase resistance to frost and corrosion, decrease thermal conductivity etc. [1].

Namely this impact of chemical additives on properties of concrete accounts for increase in volume of production of concrete with additives all over the world. E.g. in USA and Japan it reaches 80 %÷85 %, in Germany and Austria 60 %÷75 % of total amount of concrete produced [2–5].

The European standards [6, 7] set forth the main requirements for various plasticizers, super plasticizers, air entraining additives, reducers of water separation and water adsorption, regulators of binding and hardening time, accelerators and retarders.

An ever increasing role is played by pozzolanic additives, which influence the process of hydration and hardening of Portland cement, as well as the properties of concrete. These additives are numerous, including natural rock (tripoli, opoka, diatomite) and industrial wastes (slags, ashes, other amorphous SiO_2 containing materials). Of wide use currently is the additive of SiO_2 micro dust for production of concretes. The most effective content of this micro dust is 15.0 % of cement mass, however, for its high price, as a rule, not more than 5.0 % of micro dust is added to concrete [8–11].The amorphous SiO_2 containing additives participate in the reaction with lime, which is generated in the course of cement minerals hydration, and the reaction with lime results in gel calcium hydrosilicates, as well as hydroaluminates, hydrogarnets and other hydrates [12]. These new formations increase the strength of concrete and simultaneously help to save Portland cement. The application of strength-increasing additives is especially urgent for concretes with aggregates of low mechanical resistance interconnected by a small quantity of cement binder.

The fractionated chips of hardened autoclaved aerated concrete (AAC) waste may serve as a mentioned aggregate. So far, this waste, which is generated after mechanical treatment and hardening of AAC, is not fully utilized. Only a part of it after crushing is returned back to the process of production, meanwhile the remainder is most often transported to damps. The scientists of Belarus propose to fractionate the waste and, subject to coarseness of fraction, to use it in the production of dry mixes, masonry mortars and lightweight concretes after preliminary hydrophobization of fractionated waste [13]. Of course, this additional technical operation increases the cost of product. Our proposal, which involves application of special additives, enables to obtain a lightweight concrete with similar properties, not employing hydrophobization of AAC chips.

The purpose of work is to develop a lightweight composite (with density of 850 kg/m^3 and compressive strength of up to 4.0 MPa), the matrix of which consists of cement stone and AAC fractionated granules, as an aggregate, and to investigate the operational properties of this composite.

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RAW MATERIALS AND INVESTIGATION METHODS

The composite material with inclusions of AAC chips was prepared from the following raw materials:

a) Binder, Portland cement, from the company AB "Akmenės cementas", complying with the requirements of CEMI-42.5 R and CEMI-52.5 mark N, standard LST EN 197-1:2001.The chemical and mineral compositions and properties of used cements are provided in Table 1.
b) AAC chips from the company UAB "Matuizų dujų silikatas". Coarseness from 2.5 mm to 10 mm, bulk density of 400 kg/m^3;
c) Plasticizing additives – polycarboxylates: liquid GLEN-30, powders FS-20 and FS-40;
d) Air entraining additive (AEA) "Ufapore" – white-colored powder, pH of aqueous solution is 8.0, percentage of active part, 94.0 %;
e) Pozzolanic additive, SiO_2 micro dust ("Silimac", Poland) containing amorphous SiO_2 of 88.0 % (complying with the requirements of EN 13263).

The chemical and mineral compositions and binding time of Portland cement were determined according to the techniques [14 – 16], its specific surface by means of the Blaine device.

The bulk density and moisture of AAC chips were determined basing on the standard techniques [17, 18].

For this goal the balance "Kern EW4200-2NM" (expanded uncertainty ±6.0 mg) and 5 l metal vessel (internal diameter 182 mm) were used.

The components of concrete were mixed in the vertical mixer "MXP1602E" at rotation of 225 rpm. In the compositions of mixes the ratio of Portland cement mass to AAC chips mass was equal 1 : 1.75, W/C – 0.41, content of other additives: AEA – 0.001 % ÷ 0.005 % of solids mass, super plasticizer – 0.1 % ÷ 0.5 % and SiO_2 micro dust 2 % ÷ 4 % of cement mass. Procedure and duration of mixing: AEA + H_2O – 1 min; AEA + H_2O + cement – 1 min; AEA + H_2O + cement + SiO_2 micro dust + plasticizer – 1 min; AEA + H_2O + cement + plasticizer + SiO_2 micro dust + AAC waste – 3 min, i. e. the total mixing time – 6 min.

In case of absence of one or other component in the mix, the sequence of mixing was preserved.

The produced samples (100 mm × 100 mm × 100 mm cubes and 50 mm × 50 mm × 200 mm prisms) were hardened in the special hardening chamber, afterwards the density and compressive and bending strengths of concrete were determined from test of 6 samples according to the requirements of the standard [19]. The presses "Hounsfield H10KS" (expanded uncertainty ±0.15 %, for bending strength) and PSU-10 (expanded uncertainty ±0.23 %, for compressive strength).

For measurement of the impact of additives on hardening of cement matrix (by determination of the exothermal, effect of Portland cement minerals hydration) the technique [11, 20] was used. The same ratio of W/C (0.27) was taken in this experiment at all cases.

To determine the thermal conductivity coefficient (λ_{10}) the apparatus FOX-304 was used. Dry samples (300 mm × 300 mm × 50 mm) of composite material were tested at 10 °C temperature in accordance with [21] standard requirements.

Water vapour permeability was determined by standard [22] by climate regime "C": 23 – 50/95 (23 °C – ambient temperature, 50 % – relative air humidity, underneath the sample, 95 % – relative air humidity above the sample). The area of samples was 256 cm^2, thickness – 35 mm.

Table 1. Chemical and mineral composition, specific surface and binding time

Characteristics of Portland cement	Mark of Portland cement	
	CEM I 42.5 R	CEM I 52.5 N
Chemical composition, in %		
SiO_2	20.58	20.76
CaO	63.19	63.50
Fe_2O_3	3.48	3.37
Al_2O_3	5.60	6.12
SO_3	0.74	0.80
MgO	3.92	4.01
Others	0.23	0.23
Mineral composition, in %		
C_3S	56.04	58.54
C_2S	16.72	15.29
C_3A	8.96	10.40
C_4AF	10.59	10.17
Specific surface, in m^2/kg	383	375
Binding time, in h and min.		
beginning	3′45″	3′30″
end	4′30″	5′00″

For measurement of drying shrinkage of composite material samples (160 mm × 40 mm × 40 mm) by [23] standard digimatic indicator "Mitutoyo ID C 112B" whith accuracy ±0.003 mm was used.

The hygroscopic sorption properties were determined by [24] standard, using saturated solutions of four salts (K_2CO_3, NaBr, $(NH_4)_2SO_4$ and K_2SO_4) above which at temperature of 23 °C the values of relative air humidity reached 43 %, 58 %, 81 % and 94 % respectively.

For the phase analysis the X-ray diffraction analysis was employed using the diffractometer "Dron-2" (Cu anode, Fe filter).

The macro structure of samples was determined by optical microscope (magnification up to 100 times) connected to the computer, which fixed the zones of characteristic structure.

RESULTS AND DISCUSSION

In development of the new composite out of crushed AAC waste bound by cement paste, the quantity of Portland cement was not greater than 300 kg/m^3.

During the investigations it was established that 1 m^3 of concrete takes about 1.2 m^3 of chips. Therefore, the structure of molded concrete becomes more compact and the density of samples may reach 820 kg/m^3 – 850 kg/m^3. The results of investigations showed that the mixing of Portland cement paste with AAC chips without using plasticizers is problematic. At lower ratio W/C (up to 0.5), the even coating of grains by cement paste is not achievable, while at higher ratios W/C in the forming mix the separation of layers takes place. The added small quantity of super plasticizers helped to prepare mixes of quality at W/C = 0.4.

The impact of investigated super plasticizers on compressive strength of composite material is provided in Fig. 1.

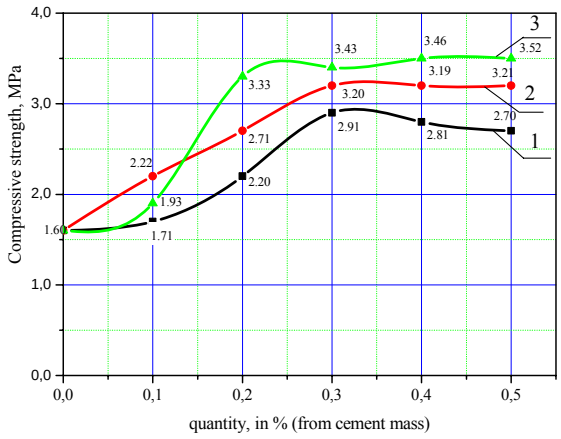

Fig. 1. Impact of quantity of super plasticizers on compressive strength of samples: 1 – FS-20; 2 – GLEN-30; 3 – FS-40 (test variation coefficient was less than 7.2 %)

The obtained results demonstrate that the strength of samples is most effectively influenced by the super plasticizer FS-40. When its content in the mix amounts to about 0.2 % of Portland cement (CEMI 42.5 R) mass, the compressive strength of samples reaches the value of 3.3 MPa (Fig. 1, curve 3). Greater quantities of this super plasticizer (up to 0.4 %) influence the strength of samples insignificantly, but almost twice retard the hardening of samples. When concrete contains 0.2 % of plasticizer FS-40, the samples can be demolded after 9 h ÷ 10 h, when 0.4 %, the demolding can be performed only after 19 h ÷ 21 h what affects the efficiency of production process.

It is known that small quantities of AEA (0.01 % ÷ 0.06 % of solids mass) in forming mixes serve for modification of inclusions surface what ensures an improved contact of inclusions with matrix of the composite. However, even such small AEA quantities make pores in the matrix and decrease the mechanical strength of the composite [25 – 28]. Having this in view, the impact of AEA in quantities of 0.005 % ÷ 0.005 % on properties of the composite was investigated. The impact of this additive on bending and compressive strengths of the composite is provided in Figs. 2 and 3.

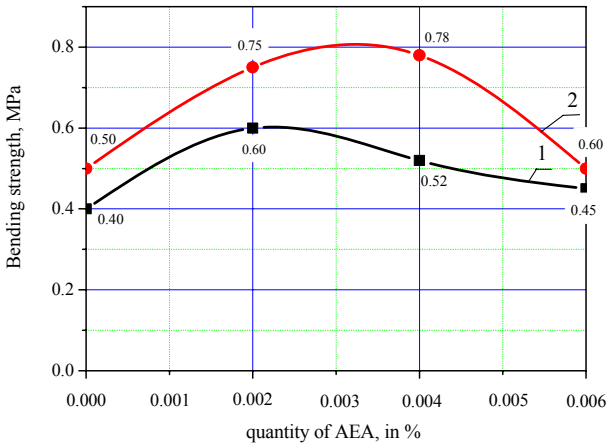

Fig. 2. AEA impact on bending strength of samples in the presence of the following binders: 1 – CEM I 42.5 R; 2 – CEM I 52.5 N at equal content of super plasticizer FS-40, 0.2 % of cement mass (test variation coefficient was less than 4.3 %)

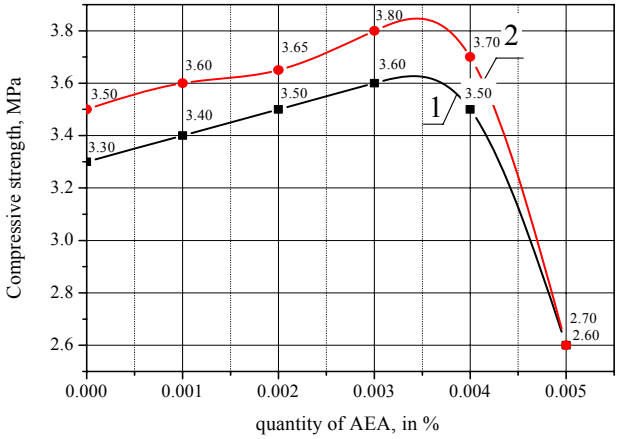

Fig. 3. AEA impact on compressive strength of samples in the presence of the following plasticizers: 1 – CEM I 42.5 R; 2 – CEM I 52.5 N at equal content of super plasticizer FS-40, 0.2 % of cement mass (test variation coefficient was less than 6.3 %)

The data from Figures 2 and 3 show that the strength of samples out of composite material is most effectively

influenced by AEA of 0.003 %÷0.004 %. In all cases the increase in strength, in particular of bending strength, versus the samples without AEA is obvious: about 50.0 % for bending strength and about 9.0 % for compressive strength. This phenomenon may be explained by the fact that small quantities of AEA (0.003 %÷0.004 %) in the mix remove aerated concrete dust present on surface of AAC chips (modify the surface), as well as open larger pores of AAC particles clearing "plugs", which were generated during crushing and sieving of AAC waste, and simultaneously improve the cohesion of crushed AAC granules with matrix of Portland cement. Greater quantities of AEA (more than 0.004 %) start making the pores in matrix itself, therefore, the strength of samples decreases.

The investigations were also led as to impact of SiO_2 micro dust on mechanical strength of the composite. The values of compressive strength of molded samples with additive of micro dust of 2.0 % and 4.0 % are provided in Fig. 4.

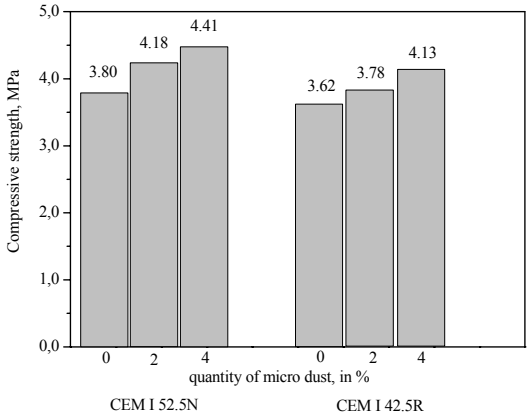

Fig. 4. Impact of SiO_2 micro dust additive on compressive strength of samples at content of super plasticizer FS-40 in all samples, 0.2 %, and that of AEA, 0.004 % (test variation coefficient was less than 3.3 %)

As it is seen from the columns provided in Fig. 4, the quantity of SiO_2 micro dust as low as 2.0 % increases the compressive strength of concrete, subject to used mark of cement, averagely by 7.5 % and in case of greater quantities (4.0 %) the increase in strength is more considerable, up to 15.0 %. The increase in concrete strength may be accounted by the reaction of portlandite, which emerges during the cement minerals hydration, with SiO_2 micro dust, and this reaction results in formation of gel calcium hydrosilicate (CSH (I) [12].

After determination of optimal content values for all additives (composition of complex additive), the investigations were carried out to the aim of establishing the impact of individual additives and of whole complex additive on matrix of the composite, i. e. on exothermal reaction of Portland cement hydration (Fig. 5).

The obtained results show that the exothermal effect of hydration of pure Portland cement ($t_{exo} = 93.0$ °C) is reached as early as in 6.5 h (Fig. 5, curve 1). A small content (0.004 %) of AEA has no influence on this process (Fig. 5, curve 2), since both curves coincide, meanwhile the added 0.2 % of plasticizer FS-40 retards greatly the hydration of Portland cement and the exothermal effect is reached only after 16.0 h. Furthermore, its temperature is lower by 6 °C ($t_{exo} = 87.0$ °C) (Fig. 5, curve 3). However, when both additives are added to the mix, the course of hydration is slowed down yet more: the exothermal effect is reached in 16.6 h and its temperature is as low as 72.0 °C (Fig. 5, curve 4). The positive impact of SiO_2 micro dust on this process is evident: the hydration reaction of cement minerals accelerates up to 6.3 h, though the temperature of exothermal effect differs but little from the hydration of pure (without additives) Portland cement (Fig. 5, curves 5 and 1). The same tendency may be observed with the complex additive: with AEA and plasticizer in cement paste and added SiO_2 micro dust, the exothermal effect is reached after 9.2 h ($t_{exo} = 93.0$ °C) (Fig. 5, curve 7), i. e. by 6.8 h more rapidly than in the same mix without SiO_2 micro dust additive (Fig. 5, curve 3). In the presence of all 4 additives (complex additive), the exothermal reaction passes after 9.5 h (versus 16.6 h without SiO_2 micro dust additive) at $t_{exo} = 78.0$ °C (versus that without SiO_2 additive, 72.0 °C) (Fig. 5, curves 4 and 8).

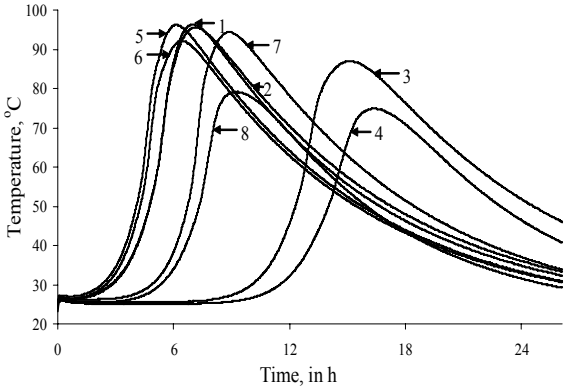

Fig. 5. Impact of additives on exothermal reaction of Portland cement minerals hydration: 1 – reference sample (without additives), 2 – sample with 0.004 % of AEA, 3 – sample with 0.2 % of super plasticizer FS-40, 4 – sample with 0.2 % of super plasticizer FS-40 and 0.004 % of AEA, 5 – sample with 4 % of SiO_2 micro dust additive, 6 – sample with 4.0 % of SiO_2 micro dust and 0.004 % of AEA additive, 7 – sample with 4.0 % of SiO_2 micro dust and 0.2 % of super plasticizer FS-40, 8 – sample with 4.0 % of SiO_2 micro dust, 0.2 % of super plasticizer and 0.004 % of AEA, W/C of all samples being 0.27

At formation of lightweight composite, the impact of complex additive on hardening of concrete decreases, since porous AAC granules absorb a great quantity of water from the mix (up to 80.0 % of their mass), therefore, with the complex additive containing plasticizer FS-40 in a not greater quantity than 0.2 % of Portland cement mass, it is possible to demold samples as early as after (9÷11) h.

The investigated operational properties of the new composite (dry density, compressive and bending strengths, thermal conductivity, water vapour transmission, sorption moisture, drying shrinkage) should be close to those of autoclaved aerated concrete, since ~90.0 % volume of the composite consists of AAC granules. The comparative values of determined properties for the composite and AAC are provided in Table 2.

Table 2. Comparative data for the developed composite and AAC [29 – 30]

Material	Dry density, kg/m^3	Compressive strength, MPa	Ben-ding strength, MPa	Ther-mal onductivity λ_{10}, W/m·K	Relative vapour resistance μ, [-]	Sorption moisture %, when φ = 94.0 %*	Drying shrinkage, mm/m
Composite material	780 ÷ 820	3.2 ÷ 4.0	0.6 ÷ 0.8	0.185 ÷ 0.195	4.5	3.0	0.25
AAC	400 ÷ 650	1.5 ÷ 3.5	0.5 ÷ 1.1	0.110 ÷ 0.160	4.2 ÷ 6.1	4.2 ÷ 5.6	0.30

* φ – relative air moisture.

In Table 2 the comparative values show that certain properties of the developed composite (e. g. dry density, sorption moisture and thermal conductivity) differ from those of AAC. The difference between properties of these two materials is predetermined by the influence of thin-layer cement matrix on surface of AAC chips.

Table 2 provides the moisture content due to hygroscopic adsorption of the composite with AAC chips at relative air moisture of 94.0 %. The kinetics of variation in moisture content due to hygroscopic adsorption at various relative air moistures is provided in Fig. 6.

Such operational properties (data of Table 2 and Fig. 6) are predetermined by the structure of the developed composite, as this structure is composed of a thin layer of cement stone coating and joining porous AAC granules only in their contact places. Therefore, in the new material, the voids among granules are generating what accounts for the better water vapor permeability than that of AAC, regardless of AAC density, lower by 1.3 ÷ 2.0 times.

As we can see, the sorption moisture of the composite undergoes greater changes mostly in the course of first 4 weeks. The balanced value is reached after 5 ÷ 7 weeks. E. g. at relative air moisture of 58.0 %, the hygrosorption moisture after exposure of 4 weeks (Fig. 6, curve 2) equaled to 1.5 % and further changed insignificantly (1.65 % after 7 weeks).

The photos of structure in the samples of composite are provided in Fig. 7. In Fig. 7, a, we can see the picture of the reference sample (formed out of cement, AAC waste and super plasticizer additive) in fracture, which shows that the surface of granules is not fully coated by binding material. On uncoated surface, free fine AAC particles and dust grains are visible. At a small content of AEA (up to 0.003 %) (Fig. 7, b), the surface of granules acquires a different picture: even and full coating, no free fine AAC particles, a full layer of densified hardened binder, about 50.0 μm thick, around granules what ensures good cohesion of binder with surface of granules and interconnection of chips. In the presence of AEA, the adsorption of AAC granules by cement paste improves; therefore, the particles of binder easier penetrate into voids and pockets of chips.

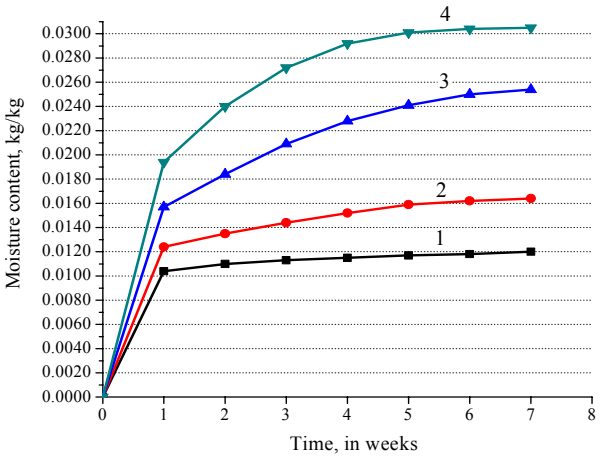

Fig. 6. Moisture content in composite material with AAC chips due to hygroscopic adsorption at relative air moisture of %: 1 – 43.0; 2 – 58.0; 3 – 81.0; 4 – 94.0

The AEA micro additive modifies the surface of AAC granules, cleans them of dust and opens the plugged pores of AAC granules. Therefore, the mechanical strength of this sample (Figs. 3 and 7, b) is higher than that of reference sample (Figs. 3 and 7, a). Along with increase in AEA content in the sample (0.005 %) (Figs. 3 and 7, c), the process of pore making begins in the matrix and the density and strength of cement stone start decreasing.

A similar picture we can see in the photo of sample with SiO_2 micro dust additive (Fig. 7, d). Here, the matrix of composite coats evenly the porous surface of AAC granules.

The micro structure of the composite with AAC chips was tested by X-ray diffraction (XRD). The XRD patterns of the samples are provided in Fig. 8 (curves 2 – 6).

Besides, the XRD patterns of AAC chips are provided (curve 1) to identify diffraction peaks of the compounds, which appeared during hydration of Portland cement minerals in the samples of the composite. In the XRD of AAC, the diffraction patterns of quartz, calcite, feldspar and tobermorite are shown (Fig. 8, curve 1). In the samples of the composite (Fig. 8, curves 2 – 6) we can also see the peaks of dolomite, newly formed portlandite and tobermorite, the latter being slightly more intense than in the XRD of AAC waste. This may be explained by the fact that portlandite, which emerges during hydration of Portland cement minerals, reacts with milled sand present in AAC waste (Fig. 8, curves 2 – 6) and with SiO_2 micro dust additive (Fig. 8, curve 6). So, we can maintain that AAC chips in this composite are not an inert aggregate, but react chemically with the products of Portland cement hydration. Thereby, the bond of the composite's matrix (cement stone) with inclusions (AAC chips) carries not only a mechanical, but also chemical character.

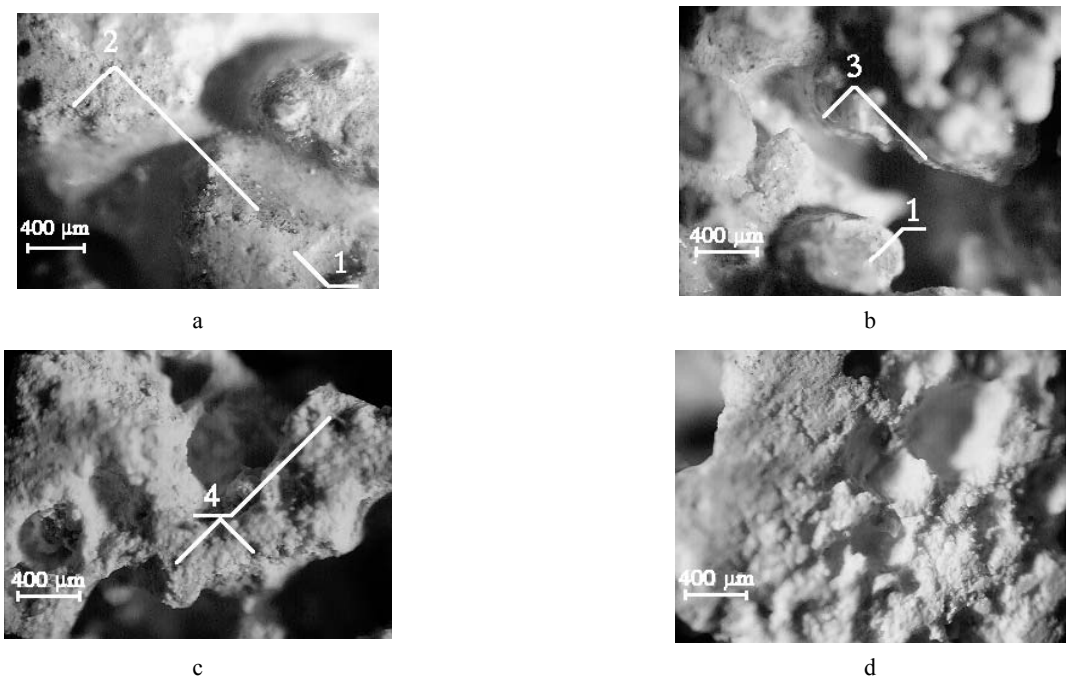

Fig. 7. Micro structure of composite material with AAC granules in fracture: a – reference sample; b – sample with 0.003 % AEA; c – sample with 0.005 % AEA; d) – sample with 4.0 % of SiO_2 micro dust additive. 1 – AAC granule; 2 – surface of a granule not coated by binder; 3 – densified layer of hardened binder; 4 – pores generated in the matrix

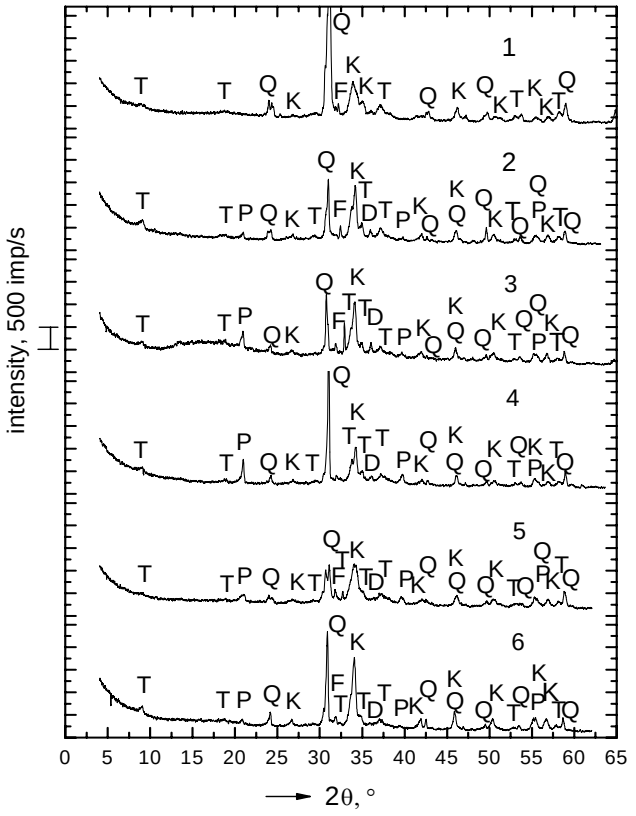

Fig. 8. X-ray diffraction patterns of samples of composite with AAC chips: 1 – AAC chips; 2 and 4 – references samples (without additives): 2 – cement CEM I 42.5 R, 4 – CEM I 52.5 N; 3 – sample with 0,004 % of AEA and CEM I 42.5 R; 5 – sample with 0,004 % of AEA and CEM I 52.5 N; 6 – sample with 4.0 % of SiO_2 micro dust and CEM I 42.5 R. Minerals: T – tobermorite, P – portlandite, Q – quartz, C – calcite, D – dolomite, F – feldspars

CONCLUSIONS

1. We have developed the composite of lightweight concrete with AAC chips sized 2.5 mm ÷ 10 mm and this material is characteristic of physical properties, which are close to AAC products (thermal conductivity, water vapor permeability, sorption moisture, drying shrinkage).

2. It was established that the compressive strength of the newly developed composite can be increased averagely by 2.3 times thank to the complex additive (CA) containing super plasticizer FS-40 of 0.2 % of Portland cement mass, air entraining additive "Ufapore" of 0,004 % of solids mass and SiO_2 micro dust of 4.0 % of Portland cement mass.

3. The impact of CA on hardening of matrix of the composite was investigated. It was established that CA extends the duration of exothermal reaction of cement minerals hydration from 6.5 h (without additive) to 9.0 h (with additive), mostly due to the retarding effect of the super plasticizer FS-40.

4. The investigation of the micro and macro structure of the composite showed that AAC granules are coated by a thin layer, ~50.0 μm thick, of cement stone, which tightly interconnects granules in the zones of their contact. The tightness of this bond is subject to impact of AEA micro quantities, which modify the surface of granules, as well as to chemical interaction of portlandite and milled sand present in granules.

Acknowledgments

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