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# Complex multifunctional additive for anchoring grout based on alkali-activated portland cement

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**Abstract.** Complex multifunctional additive (further, CA) which consists of aluminum powder, surfactant, salt-electrolyte ( $\text{NaNO}_3$ ) and calcium sulfate hemihydrate ( $\text{CaSO}_4 \cdot 0.5\text{H}_2\text{O}$ ) is proposed for ensuring necessary properties of anchoring grouts based on alkali-activated portland cement (further, AAPC) presented by the system «ordinary portland cement clinker - sodium metasilicate». Specified consistency of fresh AAPC mortar along with its strength after hardening are provided by water reducing due to application of modified polyethylene glycol characterized by stability of molecular structure in AAPC hydration medium. CA influence on shrinkage mitigation in AAPC mortar is explained both water-reducing effect and gas release with volume increasing during formation of dispersive-coagulation structure. Expansion of crystallization-condensation structure is ensured due to stress of calcium hydrosulfoaluminate  $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$  and calcium hydronitroaluminate  $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{Ca}(\text{NO}_3)_2 \cdot 10\text{H}_2\text{O}$  which form due to presence of  $\text{CaSO}_4 \cdot 0.5\text{H}_2\text{O}$  and  $\text{NaNO}_3$  in CA. Time matching of gas release with structure formation insures dense microstructure due to filling of pores by hydrated phases that causes lower excess stress during crystallization. CA ensures necessary performances of AAPC anchoring grout: consistency 190 mm; workability retention time 15 min; tensile strength in bending / compressive strength 6.1 / 25.7 MPa and 12.9 / 68.5 MPa in 1 d and 28 d agreeably; adhesion 0.9 MPa; linear extension up to +0.37 mm/m.

## 1. Introduction

According to requirements [1] and experience [2] dry mixes for anchoring application should meet requirements which are difficult to combine: consistency of fresh mortar, workability retention time, strength, adhesion and expansion. Alkali-activated cements (further, AAC's) are perspective also for such materials in view of modern tendencies concerning efficient consumption of raw materials and energy. At that, AAC mortars and concretes are characterized by increased strength [4], sulphate-resistance [5], freeze-thaw resistance [6] and weather resistance [7, 8] in comparison with analogues based on ordinary portland cement clinker (further, OPC clinker) cements.

AAPC [9] differs among the known types of AAC's [10]. The feasibility of an alkaline component in AAPC is based primarily on the idea of accelerated structure formation, especially at the early stages of hardening, and thus ensuring high early strength of artificial stone. Application of soluble sodium silicates provides the highest activity of AAPC due to formed low-calcium hydrosilicates and alkaline as well as alkaline earth hydroaluminosilicates during hydration [11]. Hence, AAPC is relevant for anchoring grouts.



Nowadays performances of fresh and hardened mortars can be provided primarily by modifiers. It was shown the effectiveness of their different nature (organic and inorganic) as well as of their complexes concerning enhancement of strength [12, 13], water impermeability [14], corrosion resistance [15], freeze-thaw resistance [16, 17], crack resistance [18] and water resistance [19].

Control of mortar properties, including proper deformations, is possible due to surfactants as organic admixtures of plasticizer group. Positive effect of surfactants on mortar is caused firstly by reduction of water content (water-reducing effect) [20]. Most of surfactants are ineffective for AAC's and therefore the principles for their choice were proposed [21, 22]. Thus, the highest effect in this case can be provided by sodium lignosulfonate [23], polyethers [21], polyols and as well as by other acyclic low and high molecular compounds [24]. Additional effects of such compounds are known. For example, decreasing of AAC's shrinkage in the presence of polypropylene glycol (polyether) is caused by redistribution of porous space and higher volume of pores with diameter of 0.1–1  $\mu\text{m}$ , the capillary pressure of which is much smaller than that of capillaries with smaller size [25].

Hydrosulfoaluminate expansion is traditional mean to regulate proper deformations while using of inorganic additives. Increasing volume of hardened mortar is ensured due to formation of hydrosulfoaluminate, i.e. ettringite ( $3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot3\text{CaSO}_4\cdot32\text{H}_2\text{O}$ ) [2, 26], while sulfate-containing substances are applied. For example, the effectiveness of calcium sulfate hemihydrate ( $\text{CaSO}_4\cdot0.5\text{H}_2\text{O}$ ) was shown for AAC as well unlike to calcium sulfate dehydrate ( $\text{CaSO}_4\cdot2\text{H}_2\text{O}$ ) [27, 28].

Regulation of mortars proper deformations can be also realized due to fiber reinforcement [29, 30].

Effective mean for mitigation of proper deformations in AAC mortars is complex multifunctional additives (CA's) which contain both organic and inorganic substances. The actuality of specified method when using salts-electrolytes, i.e.  $\text{Na}_2\text{SO}_4$  and  $\text{NaNO}_3$ , was shown [31, 32, 33]. The effectiveness of these CA's was explained by their co-acting for reduction of water content, acceleration of crystallization, alteration of porous structure as well as morphology of hydrated phases.

Well-known mean for mitigation of proper deformations in mortars is expansion due to release of gas (hydrogen) when using aluminum powder [26]. Volume increasing in such way is widely used in technology of aerated concretes and mortars based both on traditional cements [34, 35] and AAC's [36, 37].

The above results allow to predict higher effectiveness of CA's in the system «surfactant - salt-electrolyte», which was proposed for anchor AAPC grouts [32], due to involving of aluminum powder and calcium sulfate hemihydrate ( $\text{CaSO}_4\cdot0.5\text{H}_2\text{O}$ ). Supposed in this case CA provides effect on proper deformations in AAPC mortar during different stages structure formation.

Aluminum powder should ensure expansion of AAPC mortar during formation of dispersive-coagulation structure. Unlike to aerated material technology gas release should happen in dispersive-coagulation structure of AAPC mortar which is characterized by a certain plastic strength [38]. At that, gas release will be accompanied by expansion with stressing of initial structure.

Interaction of  $\text{CaSO}_4\cdot0.5\text{H}_2\text{O}$  with aluminate phases ensures expansion of AAPC mortar during crystallization-condensation structure formation as a result of ettringite formation [26]. However, calcium sulfate hemihydrate decelerates plastic strength due to formation of ettringite cover on surface of aluminate phases during dispersive-coagulation stage [27, 28].

It is well-known, surfactants also decelerate plastic strength of AAC pastes [21]. Moreover, depending on molecular structure and content, surfactants can ensure both acceleration and deceleration of gas release [39, 40].

It can be predicted that the retarding effect of  $\text{CaSO}_4\cdot0.5\text{H}_2\text{O}$  and surfactant on plastic strength of AAPC mortar can be minimized by presence of salt-electrolyte in CA. It was shown, that sodium nitrate ( $\text{NaNO}_3$ ) is more efficient in comparison with other salts-electrolytes [31, 32].

It was assumed that combination of aluminum powder, surfactant, salt-electrolyte ( $\text{NaNO}_3$ ) and  $\text{CaSO}_4\cdot0.5\text{H}_2\text{O}$  in CA composition can ensure time matching of gas release with structure formation of AAPC mortar. Such mean allows control of fresh and hardened mortars performances due to reduction of water content, formation of effective porous structure and morphology of hydrated phases.

So the aim of this research was to investigate the co-application of aluminium powder,  $\text{CaSO}_4\cdot0.5\text{H}_2\text{O}$ , surfactant and  $\text{NaNO}_3$  in CA for ensuring necessary properties of AAPC anchoring grout.

## 2. Raw materials and testing techniques

The known AAPC composition was used as base of mortar [41], %: OPC clinker – 97.6, sodium metasilicate – 2.4 (0.70 % by Na<sub>2</sub>O). OPC clinker (content, %: CaO – 66,15; SiO<sub>2</sub> – 22,61; Al<sub>2</sub>O<sub>3</sub> – 5,29; Fe<sub>2</sub>O<sub>3</sub> – 3,93; MgO – 0,84; Na<sub>2</sub>O – 0,15; K<sub>2</sub>O – 0,98; SO<sub>3</sub> – 0,50), specific surface= 450 m<sup>2</sup>/kg (by Blaine), was used as AAPC aluminosilicate component. Five-water sodium metasilicate Na<sub>2</sub>SiO<sub>3</sub>·5H<sub>2</sub>O (dry state) was used as alkaline component.

The reference composition of AAPC mortar (AAPC – 40 %, mix of aggregates and filler – 60 %) was chosen according to the proper functionality [42].

The modifiers for AAPC mortar were presented by:

- surfactants of different types having water-reducing effect: 1 - sodium lignosulphonate (further, LST) according to CAS № 8061-51-6 (pH ≥ 8.5); 2 - polycarboxylate («JK-04PP», Jiankai); 3 - polyacrylate («Vinavil fluxe», Mapei); 4 - admixture based on modified polyether polyethylene glycol («Melflux PP100F», Mapei); 5 - sodium gluconate according to CAS № 527-07-1; 6 - sorbitol (C<sub>6</sub>H<sub>14</sub>O<sub>6</sub>) according to CAS № 50-70-4; 7 - admixture based on hydroxyethylated fatty alcohol («OC-25», JSC «Barva»);

- aluminum powder according to CAS № 7429-90-5 (content, %: active aluminum – 85.00 - 93.00, Fe – 0.50, Si – 0.40, Cu – 0.05, Mn – 0.05, moisture – 0.20, fatty admixtures – 3.80), water covering ≥ 7000 cm<sup>2</sup>/g;

- calcium sulfate hemihydrate (CaSO<sub>4</sub>·0.5H<sub>2</sub>O) according to the national standard of Ukraine DSTU B V.2.7-104: 2000;

- sodium nitrate (NaNO<sub>3</sub>) according to CAS № 7631-99-4.

The aggregates were presented by: quartz sands fr.< 0.63 mm and fr.= 0.63...1.25 mm; washed granite screening fr.= 1.25...2.5 mm.

Milled sand fr.< 0.16 mm (sieve residue, %: < 0.05 mm – 80.0, 0.05 mm - 9.5, 0.063 mm - 7.0, 0.10 mm - 2.5, 0.16 mm - 1.0) was used as filler.

Optimal ratio between aggregates and filler was calculated by approaching of total grain-size curve to ideal one for enhancement of AAPC mortar (strength, proper deformations). Optimal proportion between aggregates and filler guarantees the lowest interparticle porosity along with minimal summary surface of grains [43, 44], %: milled sand – 25, sand fr.< 0.63 mm – 20, sand fr.= 0.63 - 1.25 mm – 30, granite screening – 25.

Water-reducing effect of surfactants was evaluated by W/C ratio and compressive strength of AAPC mortar characterized by constant consistency measured according to the national standard of Ukraine DSTU B V.2.7-126:2011.

Proper deformations in AAPC mortar were fixed from start of mixing with water were determined due to dial test indicator in rectangular profile assembled form with dimensions of 40×40×170 mm. Form filled by fresh mortar was closed by cover, which could to move along inner surface, and was placed between tips of dial test indicator. The step in measuring was 1 h.

The AAPC mortar strength and proper deformations in it from initial technological moisture down to equilibrium moisture were determined on specimens 40x40x160 mm. After 1 d hardening in forms with an insulated surface, the samples were removed from forms and stored for 7 d under the normal conditions (t= 20 ± 2 °C, R.H. = 95 ± 5 %). Then the samples were stored over saturated solution of potassium carbonate (NH<sub>4</sub>NO<sub>3</sub>) at t= 20 ± 2 °C till the control age. The length of samples after 1 d was taken as the initial one (zero).

The microstructure of AAPC after hardening during 14 d was carried out by electronic microscope with microanalyzer.

## 3. Results and discussions

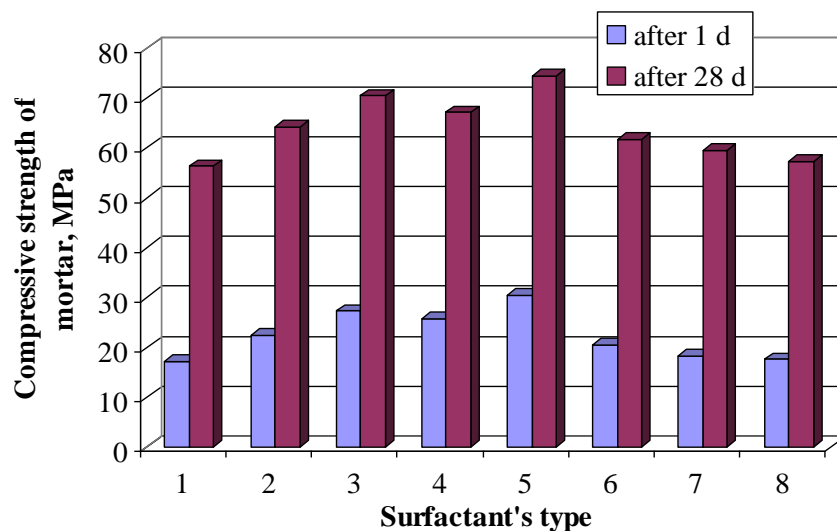
**The effectiveness of surfactants in AAPC mortar was researched. The content of surfactants was 0.5 % by mass of OPC clinker (further, %). The compressive strength of the reference AAPC mortar (W/C = 0.47) corresponds to values of 17.1 and 56.4 MPa after 1 d and 28 d agreeably while ensuring fresh mortar consistency of (190±10) mm. Admixture based on modified polyether is characterized by the highest water-reducing effect. Compressive strength of**

**modified AAPC mortar increases from 17.1 up to 30.5 MPa after 1 d and from 56.4 up to 74.3 MPa after 28 d regarding to the reference AAPC mortar while decreasing of W/C ratio from 0.47 down to 0.38 (figure 1).**

Surfactant based on polycarboxylate is characterized by lesser water-reducing effect than the modified polyether. Thus, compressive strength of AAPC mortar, modified by the this polyester, increases up to 27.3 and 70.5 MPa after 1 d and 28 d agreeably while decreasing of W/C ratio down to 0.38. Compressive strength of AAPC mortar, modified by polyacrylate, corresponds to values of 25.7 and 67.1 MPa agreeably with W/C ratio of 0.4.

LST ensures increasing of AAPC mortar compressive strength up to 22.4 MPa and 64.3 MPa after 1 d and 28 d agreeably while decreasing of W/C ratio from 0.47 down to 0.43. Compressive strength of AAPC mortar, modified by sodium gluconate, corresponds to values of 20.5 and 61.7 MPa agreeably with W/C ratio of 0.44.

**Polyol and hydroxyethylated fatty alcohol are characterized by the lowest effectiveness. Thus, sorbitol ensures increasing of AAPC mortar compressive strength up to 18.3 MPa 59.5 MPa after 1 and 28 d agreeably while decreasing W/C ratio down to 0.46. Compressive strength of AAPC mortar, modified by hydroxyethylated fatty alcohol, corresponds to values of 17.5 MPa after 1 d and 57.3 MPa after 28 d along with similar decreasing of W/C ratio.**



**Figure 1.** Effect of surfactants on AAPC mortar strength: 1- without surfactant; 2 - LST; 3 - polycarboxylate; 4 - polyacrylate; 5 - modified polyether; 6 - sodium gluconate; 7 - polyol; 8 - hydroxyethylated fatty alcohol

Hence water-reducing effect of surfactants depending on type reduces in the order: modified polyether > polyester > LST > sodium gluconate > polyol > hydroxyethylated fatty alcohol. This data corresponds to previous results [28, 29].

The effect of CA composition «surfactant - aluminum powder -  $\text{CaSO}_4 \cdot 0,5\text{H}_2\text{O}$  -  $\text{NaNO}_3$ » on proper deformations in AAPC mortar was researched. The content of aluminum powder was 0.01 % to ensure expansion while minimal negative effect on AAPC hardened mortar [38]. Contents of  $\text{CaSO}_4 \cdot 0,5\text{H}_2\text{O}$  (9.0 %) [41] and  $\text{NaNO}_3$  (2.5 %) [31, 32] were accepted basing on previous investigations. It must be noted that CA ensures insignificant decreasing of AAPC mortar compressive strength regarding to one modified only by «Melflux PP100F», i.e. from 30.5 down to 27.6 MPa after 1 d and from 74.3 down to 71.5 MPa after 28 d while W/C= 0.38.

The reference AAPC mortar is characterized by early shrinkage of dispersive-coagulation structure (-1.73 mm/m after 7 h) due to water evaporation (figure 2). At that, CA ensures expansion of AAPC

mortar which begins after 1 h since mixing with water and continues during next hour mainly. As result the expansion of AAPC mortar corresponds to value of 0.435 mm/m after 7 h.

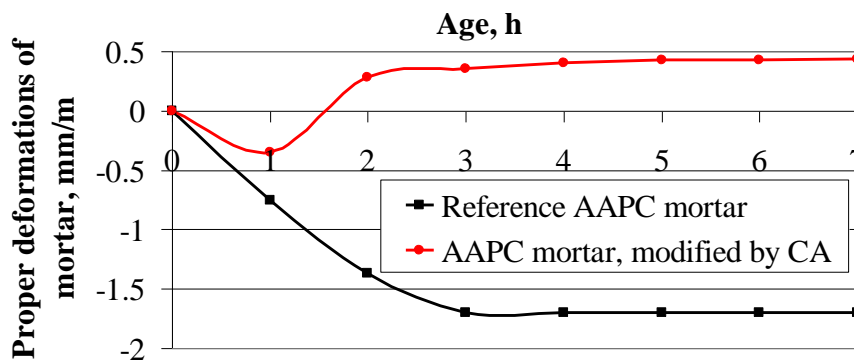


Figure 2. Early age proper deformations of AAPC mortar

The further investigations of the reference AAPC mortar showed increasing shrinkage up to value of -2.075 mm/m after 80 d (figure 3 a). Instead of that, the proper deformations of AAPC mortar, modified by CA, despite insignificant initial decreasing from +0.430 down to +0.381 mm/m after 6 d, increases up to +0.454 mm/m after 14 d (figure 3 b). Thus, the modified AAPC mortar characterizes by insignificant decreasing of expansion deformation down to +0.385 mm/m after 80 d.

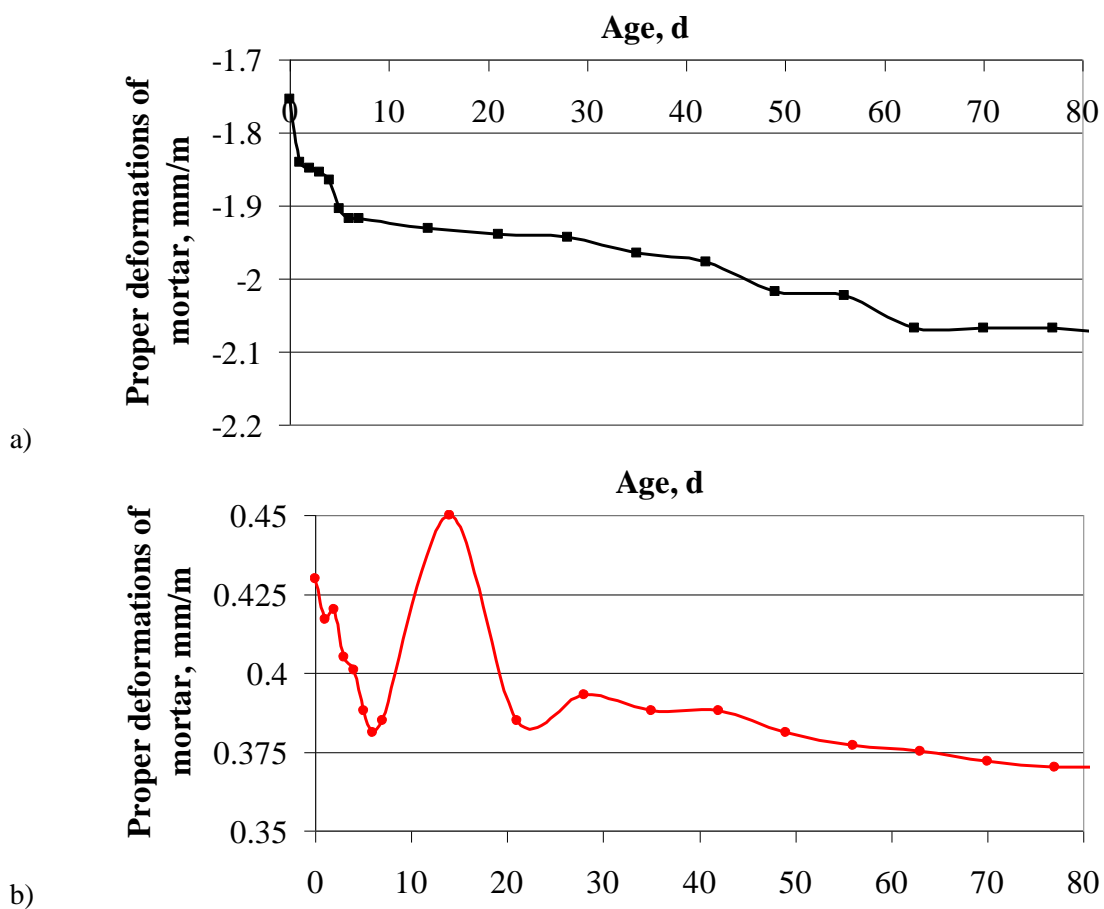


Figure 3. Proper deformations of the reference (a) and modified AAPC mortar (b)

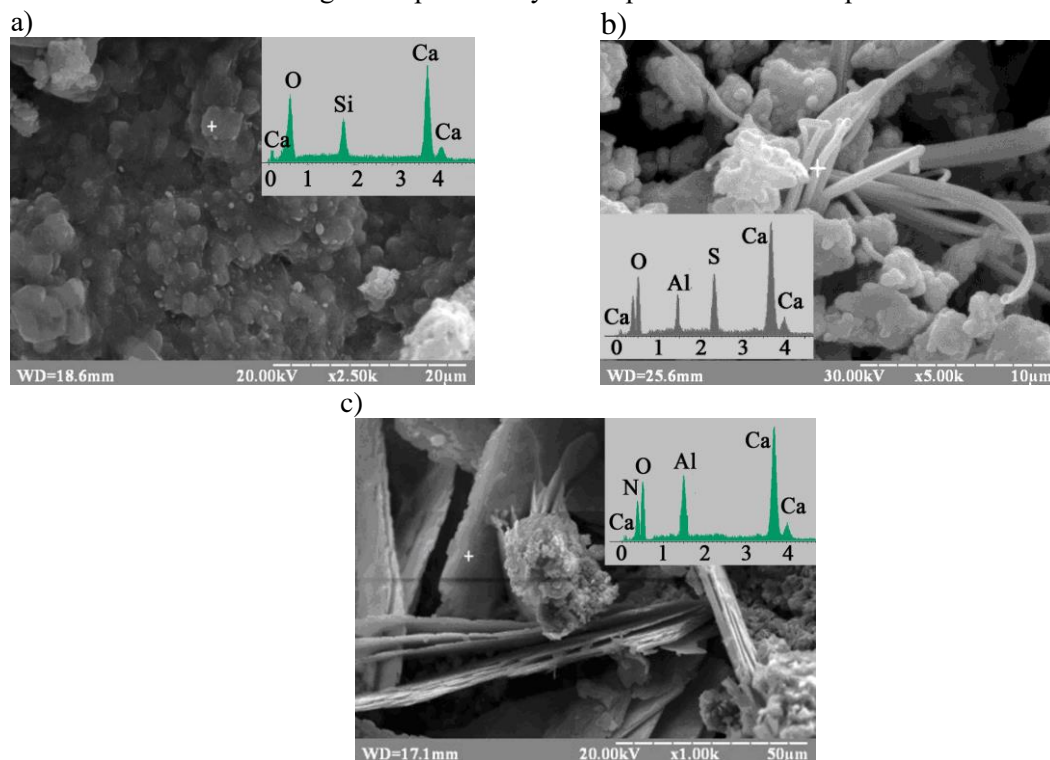
So it can be established that the proper deformations of AAPC mortar can be regulated by mean of CA due to less content of water as well as effect on structure formation. Expansion of AAPC mortar

dispersive-coagulation structure takes place due to gas release. But the proper deformations of AAPC mortar during further stages of structure formation is caused by effect of CA on crystallization processes in artificial stone.

The features of hydrated phases in AAPC, modified by CA, were investigated by electronic microscopy (figure 4).

Sub-microcrystal (gel-like) low-calcium hydrosilicates (content in probe, %: CaO – 41.19, SiO<sub>2</sub> – 53.42) are identified in the reference AAPC (figure 4 a), which cause increasing of AAPC mortar shrinkage.

Crystalline structures of ettringite (content in probe, %: CaO – 28.37, Al<sub>2</sub>O<sub>3</sub> – 9.18, SO<sub>3</sub> – 19.40) (figure 4 b) [19] and calcium hydronitroaluminate 3CaO·Al<sub>2</sub>O<sub>3</sub>·Ca(NO<sub>3</sub>)<sub>2</sub>·10H<sub>2</sub>O (content in probe, %: CaO – 38.26, Al<sub>2</sub>O<sub>3</sub> – 17.87, N<sub>2</sub>O<sub>5</sub> – 21.87) (figure 4 c) [45] were identified in pores of modified AAPC after 14 d of hardening. The specified hydrated products ensure expansion of AAPC mortar.



**Figure 4.** SEM images and probe analysis of AAPC: reference (without CA) (a); modified by CA with formation of ettringite  $3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot3\text{CaSO}_4\cdot32\text{H}_2\text{O}$  (b) and calcium hydronitroaluminate  $3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{Ca}(\text{NO}_3)_2\cdot10\text{H}_2\text{O}$  (c)

The properties of AAPC anchoring grout with specified CA, that is produced by dry mix technology, are in compliance with mandatory basic requirements [1]: consistency 190 mm; workability retention time 15 min; tensile strength in bending / compressive strength of mortar 6.1 / 25.7 MPa and 12.9 / 68.5 MPa after 1 d and 28 d agreeably; adhesion 0.9 MPa; linear extension up to +0.37 mm/m.

#### 4. Conclusions

1. The AAPC mortar with adjustable proper deformations can be obtained due to application of CA containing aluminum powder, calcium sulfate hemihydrate, surfactant and sodium nitrate as ingredients. Specified CA also provides regulation of strength of AAPC mortar while ensuring specified consistency.

2. AAPC mortar expansion while strength increasing is explained by reduction of water content, formation of effective porous structure, time matching of gas release with structure formation with

additional structure stressing due to formation of ettringite and calcium hydronitroaluminate in hydrated AAPC.

3. The characteristics of anchoring AAPC grout, containing specified CA, are in compliance with mandatory requirements including on the order greater expansion (+0.37 mm/m) regarding to analogue, modified by CA system «surfactant - salt-electrolyte». The obtained results indicate the need for optimization of CA composition and its content.

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