

# The effects of modifying additives and chemical admixtures on the properties of porous fresh and hardened cement paste



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## HIGHLIGHTS

- Additives and admixtures have an influence on the hydration process of fresh cement paste.
- This leads to the changed macro- and microstructure of hardened cement paste.
- Best properties were obtained when all additives and admixtures were used together.

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## ABSTRACT

Porous cement paste prepared using ordinary Portland cement (OPC), pozzolanic additives – metakaolin-based waste (MBW) or milled sand (MS), chemical admixtures – air entraining (AEA) and plasticizing (PA), and water from local supply system has been studied in this article. There has been studied the effects of the above mentioned additives and admixtures, and mixing time on the change of rheological properties and aeration degree of porous fresh cement paste. It was found that additives and admixtures prolong hydration of OPC paste from 400 to 1100 min, reaching the maximal temperature of exothermic effect from 90 to 60 °C.

Physical properties, such as dry density and compressive strength of hardened porous cement paste, have been identified. It has been found that the dry density of modified porous hardened cement paste, depending on mixing time, can be reduced about 3 times (from 2200 to 795 kg/m<sup>3</sup>). On the other hand, compressive strength of the porous hardened cement paste has been reduced from 100 to 2.5 MPa.

The effects of additives and admixtures on the macro- and microstructure of porous hardened cement pastes have been identified using X-ray diffraction (XRD), scanning electron microscopy (SEM) and optical microscopy methods. It was fixed, that MBW additive significantly reduces the amount of portlandite due to the active SiO<sub>2</sub> in it. Active SiO<sub>2</sub> is involved in reactions with the product of hydration of OPC minerals – portlandite and forms germs of calcium hydrosilicates. It leads to increased compressive strength of porous hardened cement paste. The influence of additives and admixtures on the aeration processes and formation of macro- and microstructure of porous hardened cement paste has been explained.

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## 1. Introduction

It is well known that AEA belongs to the class of chemical compounds which are called surfactants. One of the OPC paste density reduction (increase in porosity) methods is the use of surfactants [1–3]. Mixing of OPC with water and surfactant results in obtaining fresh cement paste of porous structure. The surface of particle, subject to the functional group of surfactants, is covered with absorbed anionic or cationic surfactant

molecules that significantly reduce a water surface tension [4]. Depending on the nature of mixing and the properties of the mixture, the mixing process, consisting of the whirling motion of mixer blades, will be turned into the free surface energy of air bubbles. A volume of the entrained air is subject to the amount of surfactant, chemical and granularity composition of OPC particles, flowability and temperature of the mixture, as well as mixing conditions [5].

Recently, for the preparation of OPC paste mixtures, the surfactants with anionic functional sulfo groups are widely used. Our previous tests of water solutions with 6 different surfactants have allowed choosing a much more efficient anionic surfactant – the AEA UFAPORE TCO [6].

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It is noteworthy that water plays an important role in the aeration process of fresh cement paste. Water content directly affects the flowability of mixture, increasing its porosity. However, if the flowability of mixture is higher than 150 mm, larger air bubbles become less stable due to the buoyancy forces, and the total content of air in the mixture decreases [7].

Strength of hardened cement paste is increased and its density is reduced by use of pozzolanic materials, containing active oxides, such as  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\text{Fe}_2\text{O}_3$ , which, at room temperature, react with the product of OPC mineral hydration – portlandite ( $\text{Ca}(\text{OH})_2$ ). These reactions generate calcium hydrosilicates, hydroaluminates, and other hydrates, which increase the strength of the hardened cement paste [4,8].

It should be noted that the main component of the fired clay – inert kaolinite – dehydrates and transforms into the active amorphized metakaolinite ( $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ) [9]. Its reaction with lime  $\text{Ca}(\text{OH})_2$  generates hydrated gehlenite ( $2\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{SiO}_2 \cdot 8\text{H}_2\text{O}$ ) and  $\text{C-S-H}_{(1)}$ , which leads to an initial hardening and strength of cement paste, as its hydration rate is higher than the one of amorphous  $\text{SiO}_2$  pozzolan [9,10]. There is much information about the positive impact of pozzolanic materials on the properties of the concrete. An effect of silica fume on hardened concrete properties has been examined in detail in work [11]. Authors [12] have studied a positive effect of amorphous silica on the hydration process of hardened cement mineral – alite. The properties of concrete are positively impacted by the fly ash admixture which enhances the density and strength of the hardened cement paste and facilitates its mixing process [13]. Mechanical properties and durability of concrete are also increased thanks to the complex paper – mill residuals and fly ash admixture [14]. The microsilica (MS) and fly ash admixture (5–15%) improves rheological properties of fresh, and increases the compressive strength of hardened cement paste [15].

A combined effect of various natural pozzolanic materials, by replacing the OPC by them, has been examined by authors [16]. It has been found that natural pozzolanic materials, containing active  $\text{SiO}_2$  – 46.48%,  $\text{Al}_2\text{O}_3$  – 14.74% and  $\text{Fe}_2\text{O}_3$  – 12.16% or high-performance fly ash with  $\text{SiO}_2$  – 62% and  $\text{Al}_2\text{O}_3$  – 18.9%, can be used effectively in producing sustainable concrete, improving the strength properties and flowability of concrete mixes. Author [17] has examined the effect of silica fume and fine aggregate particles on the rheology parameters of high performance cement-silica systems. He has found the resistance to flow behaviour of high performance concrete, as function of the particle size of aggregate. The use of silica dust in self-consolidating concretes increases the water requirement in mixtures or a number of agents that reduce water consumption [18]. It was established that the complex addition of silica fume and hydrated lime has lowered the total porosity of fly ash cement pastes [19]. There have been conducted studies of the complex pozzolanic fly ash – calcified metakaolin component, which was used as OPC replacement. It has been found that 25% of metakaolin can partially substitute the superplasticizer, as well as ensures finer pores and durability of concrete [20].

The pozzolanic behaviour of metakaolin and its effect on improvement of properties of concretes and mixtures has been presented by Rashad [9] in his review in detail. The effect of metakaolin in complex fly ash admixture on binding and hardening of mixtures was described in work [21]. Metakaolin was found to have a major effect on the consistency of mixtures.

A significant role in the process of hydration of OPC minerals belongs to polycarboxylate ether-based PA widely used in concrete production. Their main purpose is to restore colloidal particles in obtained dispersed systems: it was necessary to cover not each single particle of the suspension, but only those with a positive charge [22]. The effects of new generation polycarboxylate PA at different

dosages on the rheological properties, mechanical strength, final setting time and bleeding of cement grouts proportioned with a water to cement ratio (w/c) of 0.33, 0.40 or 0.50. The results showed that grouts with the new-generation polycarboxylate PA had highest viscosity and slightly bleeding. The polycarboxylate ether based PA improved final strength and decreased yield stress, rheological properties, mechanical strength. The higher dispersive action of polycarboxylate ether based PA released more entrapped water into the solution [23].

Influence of PA of polycarboxylic organic macromolecule results in formation of smaller megapore of calcium hydrosilicates [24].

Influence of different PA on pore structure of self-consolidating concretes is presented in work [25]. Research results have showed the mechanical properties of the studied concretes, including their compressive strength and the levels of cracking initiating stress.

PA of a new generation can significantly influence the air content in self-compacting concretes, regardless of the yield stress and viscosity of concrete [26]. The paper covers the results of the study of the efforts toward the development of a method based on surface tension measurements of plasticized suspensions.

While assessing the effect of pozzolanic additive and air entraining or plasticizing admixtures on properties of fresh and hardened cement paste and based on our previous studies, we select these test objects of research in order to obtain the lowest density porous cement paste:

- pozzolanic additive should increase the viscosity and stimulate the initial strength of fresh cement paste and reduce the density of hardened cement paste. For this purpose, we have chosen MBW from the expanded glass bead production;
- AEA should ensure the formation of very fine pores. For this purpose, we have chosen UFAPORE TCO;
- PA should increase the strength of hardened cement paste. For this purpose, we have chosen polycarboxylate ether based PA of a new generation CASTAMENT FS-40 [27].

This article presents the effects of the above mentioned additives and admixtures on the properties of porous fresh and hardened cement paste.

## 2. Materials and the methods of testing

### 2.1. Materials

In this scientific work, the following materials were used: CEM I 42.5 R class OPC complied with requirements of the standard [28]. The specific surface of the OPC – 4200  $\text{cm}^2/\text{g}$ , bulk density – 1.15  $\text{g}/\text{cm}^3$ , the initial setting time – 140 min, the final setting time – 190 min, alkali content – maximum 0.8%. Mineral composition in %:  $\text{C}_3\text{S}$  – 56.64,  $\text{C}_2\text{S}$  – 16.72,  $\text{C}_3\text{A}$  – 8.96, and  $\text{C}_4\text{AF}$  – 10.59. OPC particle size distribution is presented in Fig. 1a.

Milled sand (MS) in line with the requirements of the standard [29] was used as one of a pozzolanic additives in the mixtures. The specific surface of MS – 4700  $\text{cm}^2/\text{g}$ , bulk density – 1.60  $\text{g}/\text{cm}^3$ . MS particle size distribution is presented in Fig. 1b.

MBW was used as another pozzolanic additive in the mixtures. It is process waste of the local company, generated during the blown glass beads manufacturing process. The specific surface of MBW – 8650  $\text{cm}^2/\text{g}$ , bulk density – 0.48  $\text{g}/\text{cm}^3$ . MBW particle size distribution is presented in Fig. 1c. SEM and XRD of the MBW showed, that it is slightly polluted with very fine fragments of foamed glass particles (Fig. 2, a) and most intensive patterns belong to kaolinite and quartz (Fig. 2, b).

AEA UFAPORE TCO (white powder, pH ~ 8 of 5% water solution, the active substance content of 94%) was used.

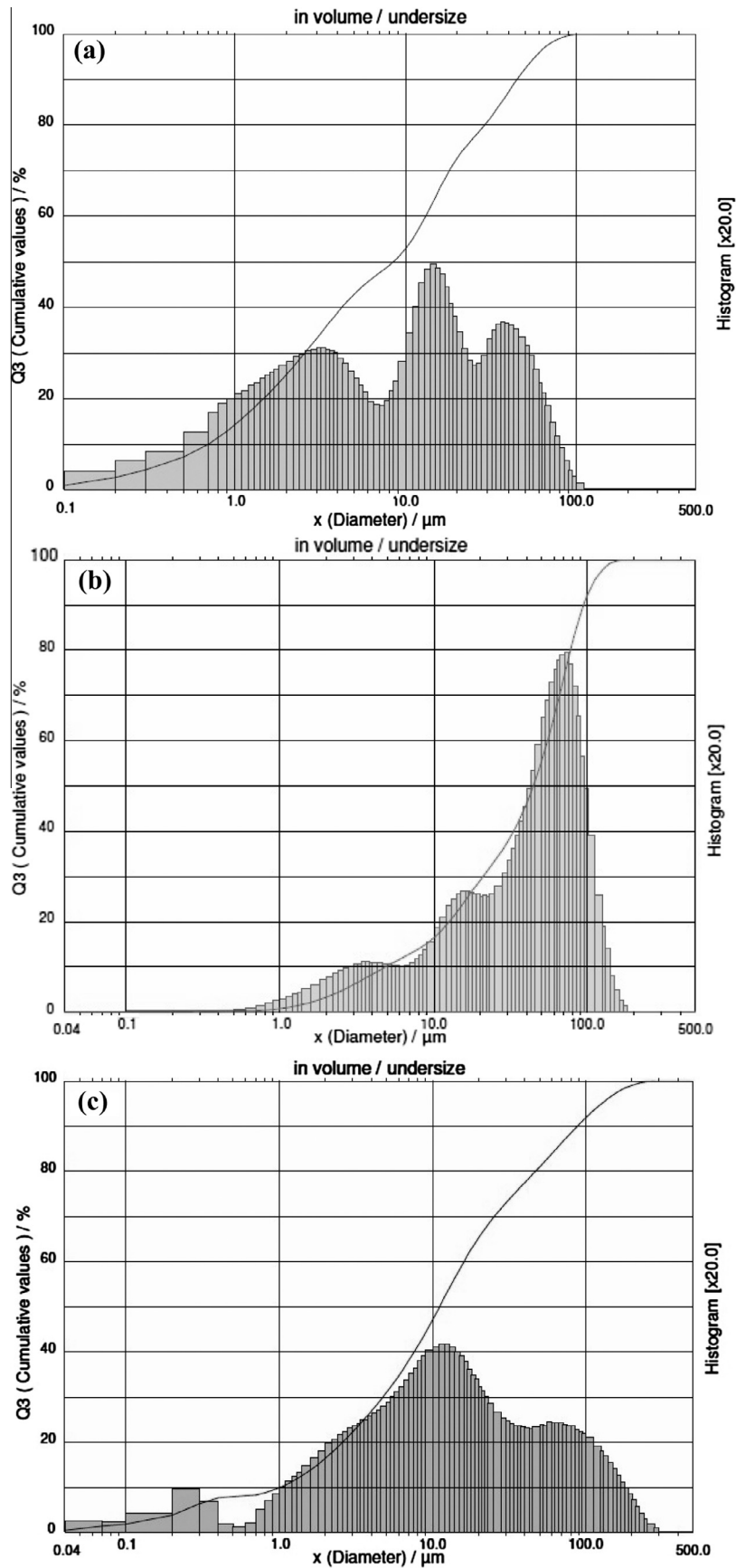


Fig. 1. Particle size distribution of: a – OPC, b – MS, c – MBW.

**Table 1**  
Chemical composition of the raw materials.

Used raw materials	Composition (in mass%)								
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O + Na <sub>2</sub> O	Ti O <sub>2</sub>	SO <sub>3</sub>	Other
OPC	20.8	6.08	3.37	63.5	2.5	1.03	–	0.80	1.92
MBW	46.1	37.2	1.10	0.20	0.20	0.70	0.70	–	13.8
MS	90.4	4.02	0.65	2.05	0.49	1.34	–	–	1.05

PA CASTAMENT FS-40 – a free flowing, spray dried powder of a polymerisation product based on polyethylene glycol (pH – 6.5 of 8.5%) was used. The chemical composition of used materials is provided in Table 1.

## 2.2. Preparation of mixtures and samples

The compositions of mixes are provided in Table 2.

All dry components, i.e. OPC, MS, MBW and PA, have been mixed for 3 min in the VARIMIXER at the rotational speed of 30 rpm. The water was added into the dry mixture and it was mixed for 5 min, then AEA was added and mixture was mixed for 2, 5 or 10 min, at the same speed. In each case, the water amount was taken considering the flowability of fresh cement paste determined by diameter of the spreaded sample (150 ± 5 mm) [30].

For determination of the hardened paste properties, there were formed samples of prism shape (160 × 40 × 40) mm. The mixture was poured into steel moulds and compacted for 1 min on the vibrating table. The moulds were kept in hermetic plastic bags for two days, then, the samples were remoulded and kept for next 26 days (50 ± 5% RH at 20 ± 2 °C) in a laboratory conditions.

## 2.3. Methods of testing

The physical and mechanical properties of samples were determined at 50 ± 5% RH and 20 ± 2 °C. All weights were measured by electronic balances 4200–2 NM with capacity of 4.2 kg and precision of 0.01 g.

The temperature of exothermic effect in modified fresh cement paste was established by methodology, developed by company ALCOA using a laboratory-designed calorimeter. Prepared fresh cement paste was poured into the laminate mould (100 × 100 × 100 mm) then a thermocouple of type T was placed in the centre of the mould. The mould was compacted for 1 min on the vibrating table and placed in a steel box which is coated by a layer of expanded polystyrene (50 mm) from inside. The change in temperature was recorded every minute by a computer.

Porous fresh cement paste density was determined after the appropriate time of mixing with AEA. A round metal container with capacity of 1.0 L was used for this purpose. Cement paste density ( $\rho_f$ ) was determined by weighing the empty container and later – the filled with cement paste container and calculated using Eq. (1):

$$\rho_f = \frac{m_f - m_e}{V} \quad (1)$$

where  $m_f$  is a weight of the container filled with fresh cement paste,  $m_e$  is a weight of the empty container,  $V$  is a volume of the container.

Dry density of the hardened cement paste samples was determined in accordance with requirements of a standard [31]. The samples were dried to constant mass at 105 ± 5 °C temperature. Dry density of hardened cement paste samples ( $\rho_h$ ) was calculated using Eq. (2):

$$\rho_h = \frac{m}{V} \cdot 10^6 \quad (2)$$

where  $m$  is a weight of dry hardened cement paste sample,  $V$  is a volume of hardened cement paste sample.

Compressive strength of the hardened cement paste samples was determined after 28 days according to the requirements of a standard [32]. The test was conducted in the powerful electromechanical testing machine H200kU of capacity 200 kN and load measurement accuracy ±0.5% of applied load from 0.2 to 100% capacity. The speed of load increment was 60.0 N/s until destruction of the hardened cement paste samples. The final test result was taken as the average value calculated out of at least 3 successful measurements. The compressive strength ( $f_c$ ) of the hardened cement paste samples was found from Eq. (3):

$$f_c = \frac{F}{A_c} \quad (3)$$

where  $F$  is the maximal failure load,  $A_c$  is cross-section area of the hardened cement paste sample.

**Table 2**  
Compositions of the mixtures.

Composition No.	Composition (in mass%)				Chemical admixtures (from the mass of OPC %)	
	PC	MW	MS		SP	AEA
1	100	–	–	–	–	–
2	100	–	–	–	–	0.05
3	100	–	–	–	0.50	–
4	100	–	–	–	0.50	0.05
5	90	10	–	–	–	–
6	90	10	–	–	–	0.05
7	90	10	–	–	0.50	–
8	90	10	–	–	0.50	0.05
9	90	–	10	–	–	–
10	90	–	10	–	–	0.05
11	90	–	10	–	0.50	–
12	90	–	10	–	0.50	0.05

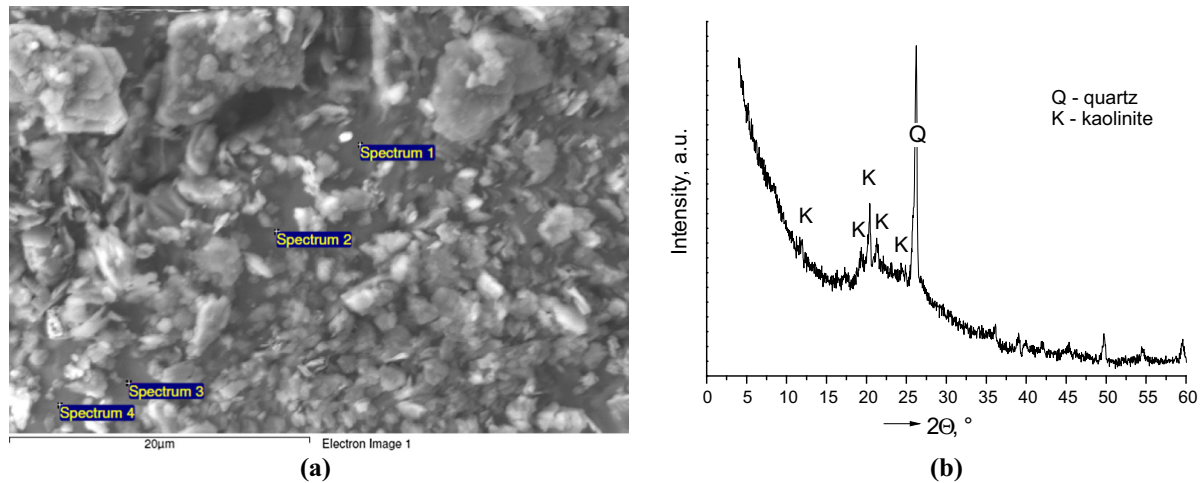


Fig. 2. Characterization of MBW: a – SEM image, b – XRD patterns.

The measurement errors were calculated by the following procedure: firstly, the average value of measurements ( $\bar{x}$ ) was calculated from Eq. (4):

$$\bar{x} = \frac{1}{n} \sum_{i=1}^n x_i \quad (4)$$

where  $x_i$  is the result of successive measurements,  $n$  is number of measurements.

Secondly, the average square deviation was calculated using Eq. (5):

$$S_x = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n - 1}} \quad (5)$$

For the examination of macrostructure, there were areas of interest (contact areas between fillers and the matrix) chosen and observed by optical microscope MOTIC with a digital camera PVC 100C (magnification is up to 100 times) which is connected to a computer.

The distribution of OPC, MBW and MS particles was determined by a laser particle analyser ANALYSETE 22.

The XRD analysis was performed by diffractometer DRON-7. Anode – Cu, filter – Ni, anode operating voltage – 30 kV, anode emission current – 8 mA, goniometer apertures (0.5; 1.0; 1.5) mm. The phase composition was determined by employing ICDD database.

The microstructure of MBW particles and hardened cement paste samples were studied by a field emission scanning electron microscope JSM 6490 LV. Accelerating voltage 20.0 kV. The surface of samples were coated by conductive carbon layer.

### 3. Research results

#### 3.1. The effects of additives and admixtures on the properties of fresh cement paste

##### 3.1.1. Water content

At the beginning of the experiment, there was initially determined a water content in the mixtures of fresh cement paste ensuring diameter of the spreaded sample ( $150 \pm 5$  mm).

We can see that water content also varies when the OPC is partly replaced by MBW or MS (see Fig. 3). When replacing 10% of OPC mass by MBW, the required water content for mixture of fresh cement paste increases by 3.6%, meanwhile, when replacing

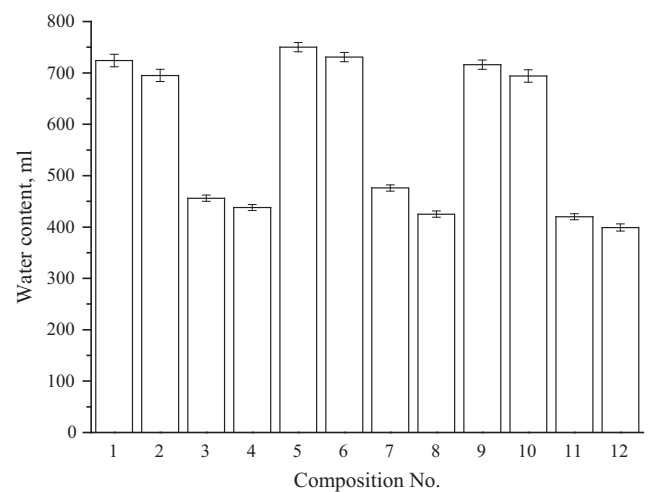


Fig. 3. The effects of additives and admixtures on the water content in fresh cement paste mixtures: 1 – OPC; 2 – OPC + AEA; 3 – OPC + PA; 4 – OPC + PA + AEA; 5 – OPC + MBW; 6 – OPC + MBW + AEA; 7 – OPC + MBW + PA; 8 – OPC + MBW + PA + AEA; 9 – OPC + MS; 10 – OPC + MS + AEA; 11 – OPC + MS + PA; 12 – OPC + MS + PA + AEA.

10% MS, the water content for mixture of fresh cement paste slightly decreases about 1% (compared Fig. 3, columns 1, 5 and 9). This can be explained by the fact that the use of MBW, compared to MS, results in the increased volume of mixture of fresh cement paste due to different bulk density of these materials, particle fineness, their sorption properties, and the increased required water content for wetting of the particles.

PA reduces the required water content in mixture of fresh cement paste by 37%, when only OPC is used (compared Fig. 3, columns 1 and 3). We can see that using PA and replacing 10% of OPC mass by the MBW required water content for mixture of fresh cement paste decreases up to 37%, while PA and replacing MS – as much as up to 41% (compare Fig. 3, columns 5 and 7, also 9 and 11). This could be explained by the specific effect of the PA on particles of mixture of fresh cement paste components. It disperses them, facilitates the free flow of water among them. Its mechanism is presented in the discussion section.

When the AEA is used in the mentioned mixtures of fresh cement paste, the required water content slightly decreases by 5% (compared Fig. 3, columns 1, 3, 5, 7, 9 and 11, with 2, 4, 6, 8, 10 and 12).

3.1.2. Setting time

Once the required water content in mixtures was determined, there were carried out calorimetric measurements of all compositions of fresh cement paste mixtures, which enabled to identify the effect of different admixtures on the hardening process of OPC (see Fig. 4). We can see that the highest temperature of the fresh cement paste (Fig. 4, curve 1) obtained when OPC without additives and admixtures was used. Its hydration exothermic effect ( $T_{egz} = 93\text{ }^{\circ}\text{C}$ ) is reached within 400 min. Hydration of pure OPC minerals is fastest at high temperature. When OPC partially replaced by MBW hydration takes slightly longer due to the decreased maximal temperature of the exothermic effect (Fig. 4, curve 5). MS admixture in fresh cement paste mixtures prolongs hydration of OPC minerals due to the decreased temperature, which is subject to the heat susceptibility of mixture components (Fig. 4, curve 9).

AEA decreased (about  $20\text{ }^{\circ}\text{C}$ ) the exothermic effect of the 2 fresh cement paste mixtures where only OPC or OPC is partly replaced by MBW is used and slightly prolonged hydration process of OPC minerals (Fig. 4, curves 2 and 6).

PA significantly slowed down the hydration process of OPC minerals. In fresh cement paste where only OPC – to 960 min, when OPC is partly replaced by MBW – to 1050 min, MS – to 1450 min. This process is influenced by a thick water layer, accumulated on the surface of OPC particles, covered with a plasticizer monomolecular-thickness film, which prevents mineral hydration (Fig. 4, curves 3, 7 and 11). Hydration process is slowed down even more in fresh cement paste mixtures, where OPC is partly replaced by MS, as PA monomolecular-thickness film is strengthened and compacted due to the chemical reaction between the lime, separated during the hydration of initial OPC minerals, a functional polycarboxylate carbon group, and the sand  $\text{SiO}_2$ .

When AEA and PA are used together, the exothermic effect of all 3 fresh cement paste mixtures decreased (about  $10\text{ }^{\circ}\text{C}$ ) and prolonged hydration process of OPC minerals comparing with fresh cement paste mixtures where only PA was used (compared Fig. 4, curves 3, 7 and 11 with 4, 8 and 12).

3.1.3. Density

Variation of fresh cement paste density on mixing time is presented in Fig. 5. The mixing time of fresh cement paste was till 10 min. It should be noted that a longer mixture mixing is useless technologically and economically.

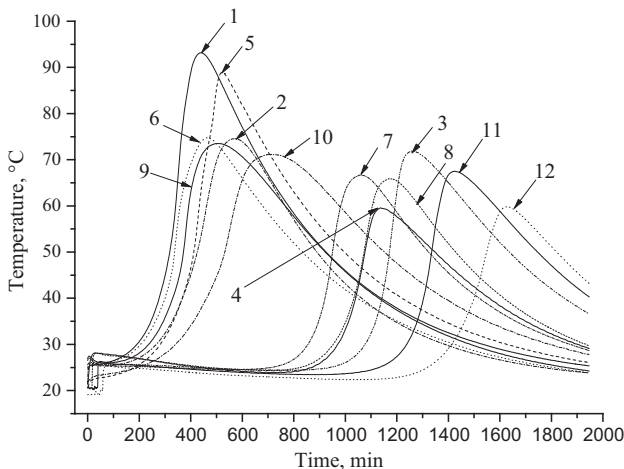


Fig. 4. The effects of additives and admixtures on the hydration of fresh cement paste mixtures: 1 – OPC; 2 – OPC + AEA; 3 – OPC + PA; 4 – OPC + PA + AEA; 5 – OPC + MW; 6 – OPC + MBW + AEA; 7 – OPC + MBW + PA; 8 – OPC + MBW + PA + AEA; 9 – OPC + MS; 10 – OPC + MS + AEA; 11 – OPC + MS + PA; 12 – OPC + MS + PA + AEA.

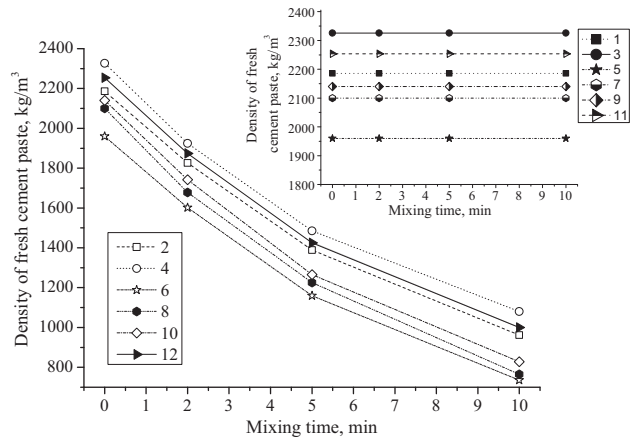


Fig. 5. The effects of a mixing time on the density of fresh cement paste mixtures: 1 – OPC; 2 – OPC + AEA; 3 – OPC + PA; 4 – OPC + PA + AEA; 5 – OPC + MBW; 6 – OPC + MBW + AEA; 7 – OPC + MBW + PA; 8 – OPC + MBW + PA + AEA; 9 – OPC + MS; 10 – OPC + MS + AEA; 11 – OPC + MS + PA; 12 – OPC + MS + PA + AEA.

We can see that the density of fresh cement paste without AEA does not depend on mixing time (see Fig. 5, curves, 1, 3, 5, 7, 9 and 11).

The density of fresh cement paste with AEA always is decreased with an increase in mixing time. The lowest density of fresh cement paste is obtained when OPC is partly replaced by MBW with AEA, comparing with fresh cement paste with pure OPC or OPC partly replaced by MS (compare Fig. 5. curves 2 and 10 with 6). A crucial role in this process goes to MBW additive that allows the formation of stable air bubbles in the condensed fresh cement paste mixture. The fresh cement paste density is reduced by 2.6 times – from 1960 to 735  $\text{kg/m}^3$  (see Fig. 5, curve 6).

MBW together with PA and AEA reduce the fresh cement pastes density by 2.9 times – from 2100 to 765  $\text{kg/m}^3$ , after 10 min of mixture mixing (see Fig. 5, curve 8). It is 29% lower than the density of fresh cement paste mixture with MS together with PA and AEA, and 32% lower than the density of the fresh cement paste with pure OPC together with PA and AEA (see Fig. 5, curves 4 and 12).

3.2. The effects of additives and admixtures on the properties of hardened cement paste

3.2.1. Dry density

The effect of mixing time on the dry density of hardened cement paste is presented in Fig. 6. We can see that the dry density

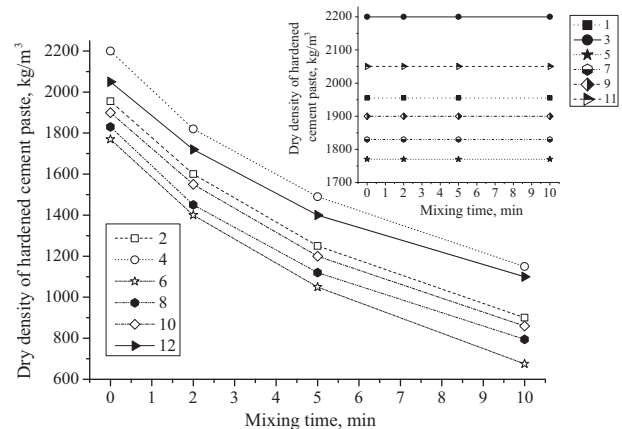


Fig. 6. The effects of additives and admixtures on the dry density of hardened cement paste: 1 – OPC; 2 – OPC + AEA; 3 – OPC + PA; 4 – OPC + PA + AEA; 5 – OPC + MBW; 6 – OPC + MBW + AEA; 7 – OPC + MBW + PA; 8 – OPC + MBW + PA + AEA; 9 – OPC + MS; 10 – OPC + MS + AEA; 11 – OPC + MS + PA; 12 – OPC + MS + PA + AEA.

of hardened cement paste without AEA does not depend on mixing time (see Fig. 6, curves, 1, 3, 5, 7, 9 and 11). When replacing 10% of OPC mass by the MBW, the dry density of hardened cement paste reduces from 1955 to 1770 kg/m<sup>3</sup>, while replacing 10% of OPC mass by MS – up to 1900 kg/m<sup>3</sup> (compare Fig. 6, curve 1 with 5 and 9).

The dry density of hardened cement paste with AEA always is decreased with an increase in mixing time. The lowest dry density of hardened cement paste is also obtained when OPC is partly replaced by MBW with AEA, comparing with hardened cement paste with pure OPC or OPC partly replaced by MS. After 2 min of mixing, dry density of hardened cement paste with pure OPC decreased from 1955 to 1600 kg/m<sup>3</sup>, when OPC is partly replaced by MBW dry density of hardened cement paste decreased from 1770 to 1400 kg/m<sup>3</sup>, and when OPC is partly replaced by MS – from 1900 to 1550 kg/m<sup>3</sup>. After 5 and 10 min of mixing, the dry density of hardened cement paste with pure OPC decreased accordingly 1250 and 900 kg/m<sup>3</sup>, with OPC partly replaced by MBW – 1050 and 675 kg/m<sup>3</sup> and with OPC partly replaced by MS – 1200 and 860 kg/m<sup>3</sup> (Fig. 6, curves 2, 6 and 10).

When PA is used together with AEA, the dry density decrease of hardened cement paste with pure OPC after 2, 5, 10 min of mixing was accordingly 1820, 1490 and 1150 kg/m<sup>3</sup>, with OPC partly replaced by MBW – 1450, 1120 and 795 kg/m<sup>3</sup> and with OPC partly replaced by MS – 1720, 1400 and 110 kg/m<sup>3</sup> (Fig. 6, curves 4, 8 and 12).

3.2.2. Compressive strength

Variation of compressive strength of hardened cement paste is mainly determined by its dry density, which depends on the specification of used additives and admixtures and is presented in Fig. 7.

We can see that the compressive strength as well as the density of fresh and hardened cement paste without AEA does not depend on mixing time (see Fig. 7, curves, 1, 3, 5, 7, 9 and 11). When replacing 10% of OPC mass by the MBW, the hardened cement paste compressive strength reduces from 95 to 73 MPa, while replacing 10% of OPC mass by MS – up to 85 MPa (compare Fig. 7, curve 1 with 5 and 9). MBW reduces the dry density of the hardened cement paste and, thus, enhances new formations in it. Due to MBW, the lowest dry density and 23% lower strength were obtained, compared to the hardened cement paste with pure OPC (see Fig. 7, curves 1 and 5).

Compressive strength of hardened cement paste, as well as the density, decreases depending on the amount of entrained air. Most air bubbles are generated during the mixing of the lightest fresh cement paste mixtures, therefore, strength of such hardened

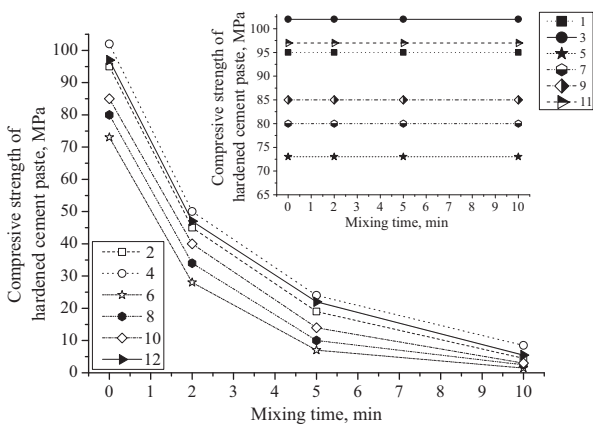


Fig. 7. The effects of additives and admixtures on the compressive strength of hardened cement paste: 1 – OPC; 2 – OPC + AEA; 3 – OPC + PA; 4 – OPC + PA + AEA; 5 – OPC + MBW; 6 – OPC + MBW + AEA; 7 – OPC + MBW + PA; 8 – OPC + MBW + PA + AEA; 9 – OPC + MS; 10 – OPC + MS + AEA; 11 – OPC + MS + PA; 12 – OPC + MS + PA + AEA.

cement pastes is lower. AEA helps to significantly reduce the dry density of hardened cement paste that is why its compressive strength with AEA always is decreased with an increase in mixing time. The lowest compressive strength of hardened cement paste is obtained when OPC is partly replaced by MBW with AEA, comparing with hardened cement paste with pure OPC or OPC partly replaced by MS. After 10 min of mixing the compressive strength

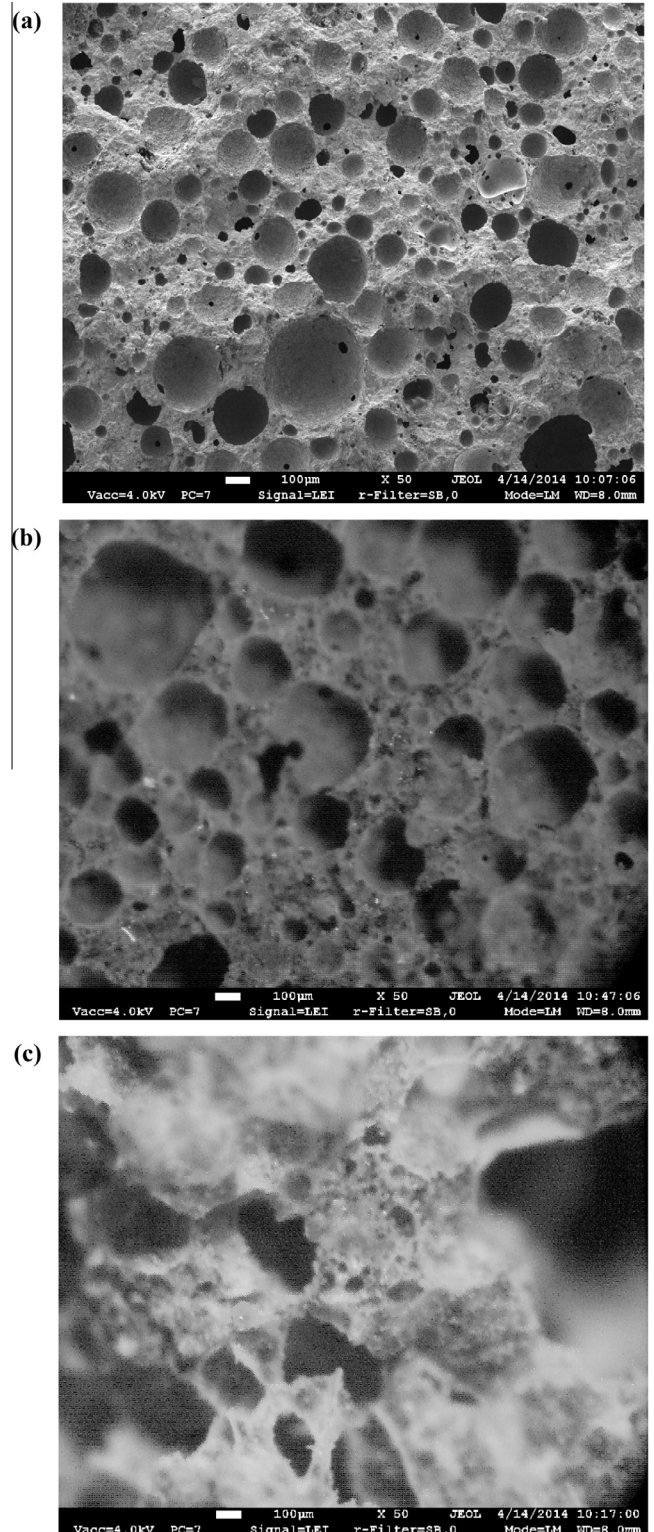


Fig. 8. Macrostructure of hardened cement paste, consisting of OPC, MBW, AEA and PA after different mixing time (magnification 50×): a – 2 min; b – 5 min; c – 10 min.

of hardened cement paste with pure OPC decreased from 95 to 4.5 MPa, with OPC partly replaced by MBW – from 73 to 1.5 MPa and with OPC partly replaced by MS – from 85 to 3.0 MPa (compares Fig. 6, curves 2 and 10 with 6). Such compressive strength of hardened cement paste is too small to insure a wide area of its application in building structures. For this reason, PA was used to increase the compressive strength of hardened cement paste, due to increase in its dry density. After 10 min of mixing, the compressive strength of the lightest hardened cement paste has been increased by 60% (see Fig. 7, curve 8).

### 3.2.3. Macrostructure

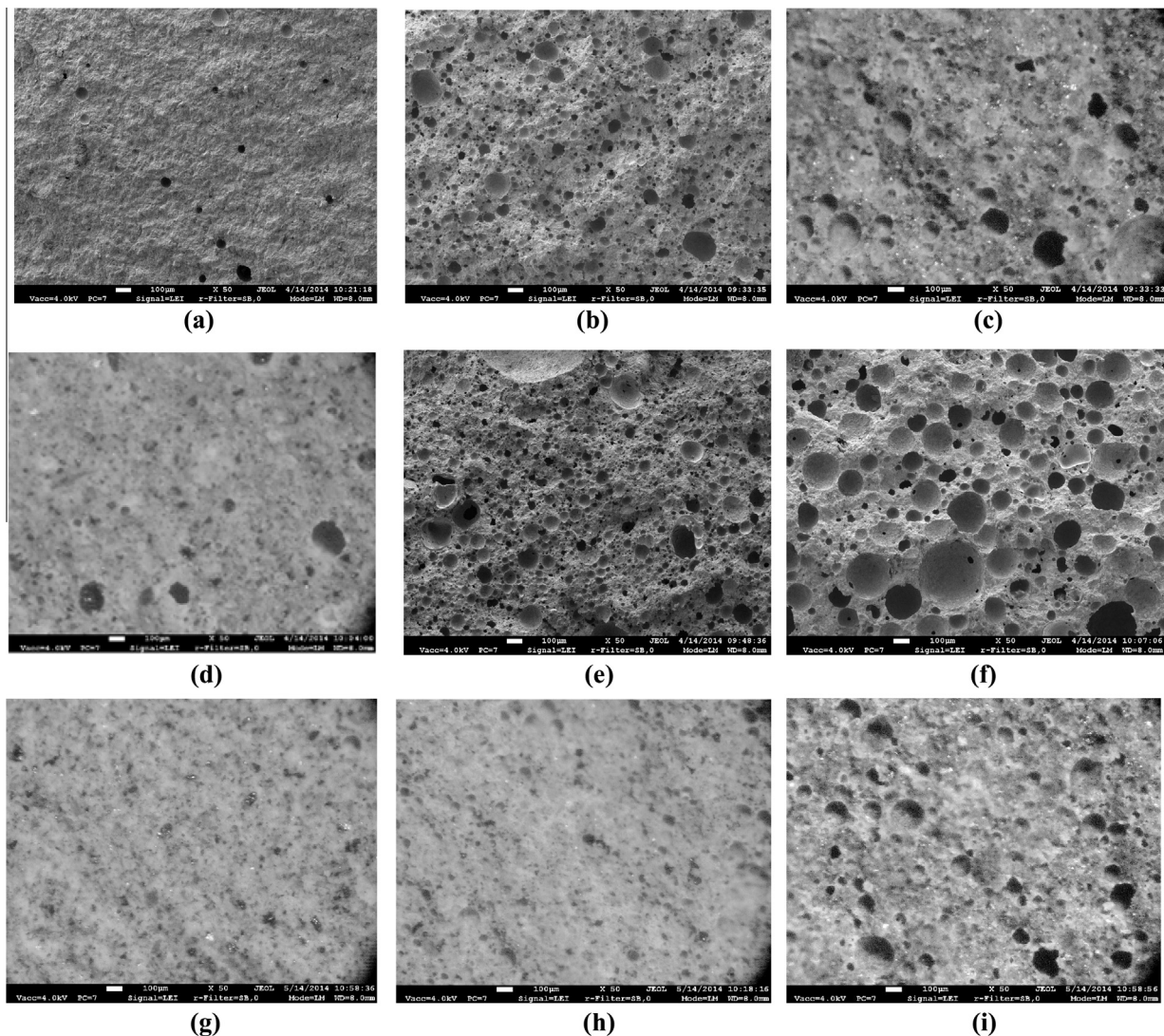
Macrostructure of hardened cement paste, when OPC partly replaced by MBW, after 2, 5 and 10 min of mixing with AEA is shown in Fig. 8. Macrostructure of hardened cement paste after 10 min of mixing with AEA has shown that the pores are of irregular shape and interconnected between each other. We also can see that after 2 min of mixing there are more less air voids, and their diameter is smaller comparing with the macrostructure of hardened cement paste after 5 min of mixing with AEA. The macrostructure of hardened cement paste after 2, 5 and 10 min of mixing with AEA using pure OPC or when OPC partly replaced by MS has the same tendency and was not showed in Fig. 8.

Macrostructure of hardened cement paste using additives and admixtures after 5 min of mixing is presented in Fig. 9. When OPC partly replaced by MBW or MS, the macrostructure is changing insignificantly (compare Fig. 9, a with d and g). AEA entrains the air and forms closed air voids in the fresh cement paste during its mixing. We can see that air voids have different sizes, and their maximum content is in hardened cement paste with OPC partly replaced by MBW (see Fig. 9, e). We can see that when OPC partly replaced by MS, pores are mostly the smallest (see Fig. 9, h).

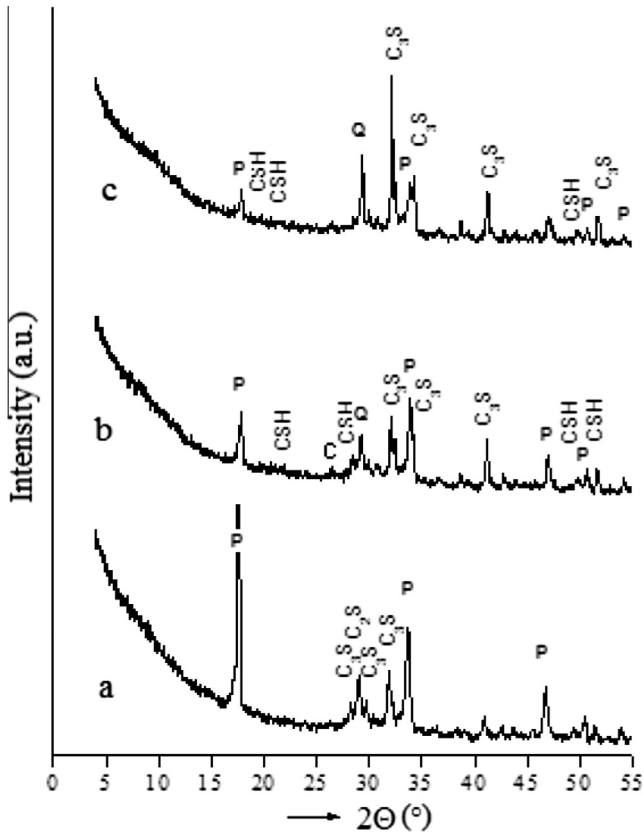
Complex usage of AEA and PA changes the macrostructure of hardened cement paste. When OPC partly replaced by MS acquire air voids of irregular shape (see Fig. 9, i), and when OPC partly replaced by MBW – closed voids of regular shape (see Fig. 9, f). Pores of pure OPC of hardened cement paste macrostructure take an intermediate position between OPC partly replaced by MBW and MS (compare Fig. 9, c with f and i).

### 3.2.4. Microstructure

A microstructure of hardened cement paste is examined applying two methods – XRD and SEM. The first method analyses the degree of crystallization. The second method analyses the effects of the used additives and admixtures on development of a new formations.



**Fig. 9.** Macrostructure of hardened cement paste (magnification 50 $\times$ ): a – OPC; b – OPC + AEA; c – OPC + AEA + PA; d – OPC + MBW; e – OPC + MBW + AEA; f – OPC + MBW + AEA + PA; g – OPC + MS; h – OPC + MS + AEA; i – OPC + MS + AEA + PA.



**Fig. 10.** The XRD patterns of hardened cement paste: a – OPC + AEA; b – OPC + MBW + AEA; c – OPC + MBW + AEA + PA. P – portlandite; C – calcite; Q – quartz; C<sub>2</sub>S – belite; C<sub>3</sub>S – alite; CSH – calcium hydrosilicates.

**3.2.4.1. XRD.** The previous research results have showed that the best properties of fresh and hardened cement paste are achieved when OPC is partly replaced by MBW, comparing with pure OPC or when OPC partly replaced by MS. The effect of AEA, PA and MBW on the hydration process of cement minerals in pure OPC is studied in greater depth in this paragraph.

We can see the new formations in hardened cement paste with pure OPC which have formed during hydration process of OPC minerals. The main new formation in hardened cement paste with pure OPC and AEA is portlandite. Also there are observed partially hydrated minerals of OPC – C<sub>3</sub>S (alite) and C<sub>2</sub>S (belite) (see Fig. 10, curve a).

MBW significantly reduces the amount of portlandite due to the active SiO<sub>2</sub> oxide in it. SiO<sub>2</sub> oxide is involved in reactions with the product of hydration process of OPC mineral – portlandite, and forms calcium hydrosilicates. Peaks of these new formations are observed in Fig. 10, curve b.

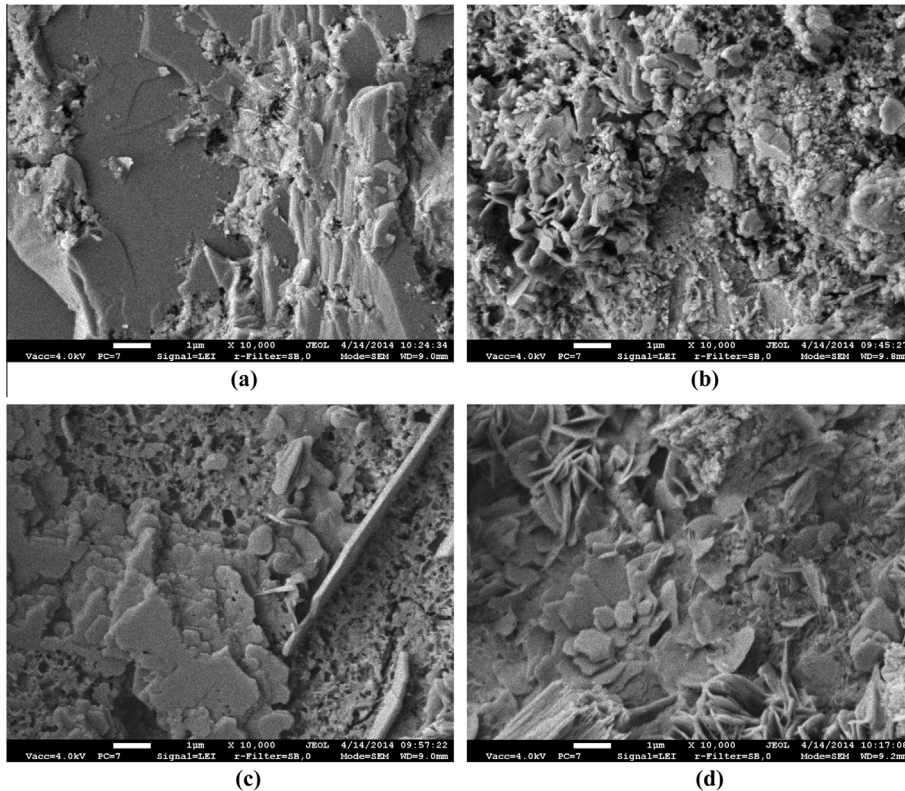
When OPC is partly replaced by MBW with AEA and PA, the amount of portlandite is also reduced and calcium hydrosilicates are formed too. Peaks of other crystals show about the presence of non-hydrated OPC minerals, as well as quartz and kaolinite contained in MBW (see Fig. 10, curve c).

**3.2.4.2. SEM.** A microstructure of the same hardened cement paste studied by XRD is also examined using SEM in this paragraph.

The new formations formed in hardened cement paste of pure OPC are provided in Fig. 11, a. Mainly, there are geoxal plates of portlandite and various-size rectangular shapes of alite.

The microstructure of hardened cement paste of pure OPC with AEA reflects porous sub-microcrystalline with calcium hydrosilicates (see Fig. 11, b).

The microstructure of hardened cement paste where OPC partly replaced by MBW with AEA reflects calcium hydrosilicates and sharp hexagonal plates of non-reacted kaolinite (see Fig. 11, c).



**Fig. 11.** SEM images of hardened cement paste (magnification 1000×): a – OPC; b – OPC + AEA; c – OPC + AEA + PA.

The microstructure of hardened cement paste where OPC partly replaced by MBW with AEA and PA reflects the crystals identified by the X-ray patterns (see Fig. 10, c): non-hydrated OPC minerals, portlandite, kaolinite, quartz and new formations – calcium hydrosilicates (see in Fig. 11, d).

#### 4. General discussion

The introduction states that one of methods to produce the porous cement paste is the use of AEA. After adding AEA to the fresh cement paste mixture, molecules are adsorbed onto the surface of OPC particles and early precipitation hydration products. There are also particles interactions taking place at the solid – liquid air interfaces [33]. The density of fresh cement paste can be reduced by 2.3 times in this way. This effect can be enhanced even more when preparing fresh cement paste mixture where OPC is partly replaced by MBW (lower bulk density comparing to OPC) [33,34].

MBW is a product of blown glass bead manufacturing process obtained during the bead treatment by heat. Kaolin flour loses two molecules of water at 650–850 °C temperature, and transforms into the active pozzolanic material – MBW ( $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O} \rightarrow \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 + 2 \text{H}_2\text{O}$ ) [9,10,35].

This MBW is contaminated with a small amount of fine blown glass fragment particles, not involved in chemical reactions and performing the role of fine filler in the cement paste mixtures (see Fig. 2, a).

In an aqueous solution, MBW particles adsorb water, expand and increase the viscosity of the mixture – it thickens and becomes difficult-to-mix. In this case, it is necessary either to increase the water amount in mixtures, thus reducing the strength of the hardened cement paste, or to use PA which disperses particles of the mixture components, thereby, facilitating the free flow of water

between particles of the mixture components. PA reduces the surface tension of water. In the aqueous solution, water accumulates on external chains of polycarboxylate PA polymer, while polycarboxylate, with its main chain, including distributed functional carbo-groups, surrounds OPC particles [36]. This effect makes the water layer, adsorbed on the OPC particle, thicker, and the space between the OPC particles is smaller. At the next phase, the OPC particles, along with the thickened water layer and polycarboxylate monomolecular-thickness film, are compressed against each other, the space between the particles narrows to form closed, static water-filled cavities [26]. MBW particles, involved in this process, expand by adsorbing water, and thicken the solution. In the medium between the cement particles and the static water-swollen MBW particles, with volume growing and pressing the static water, there generates a pressure directed to the outside of the system, thus, restricting the free flow of unbound  $\text{H}_2\text{O}$  between the particles in the mixture. There is formed a stable condensed cement paste, modified with active pozzolanic material – MBW. Mixing the cement with water, results in the hydration of its minerals. The major part of OPC consists of alite  $\text{C}_3\text{S}$  mineral (about 60%), which decomposes during hydration to form calcium hydrosilicates and releasing lime – portlandite:



Active MBW minerals react with lime and water to form additional amount of calcium hydrosilicates, calcium aluminium hydrates, and calcium aluminium hydrosilicates:  $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 + \text{Ca}(\text{OH})_2 + \text{H}_2\text{O} \rightarrow \text{C-S-H}, \text{C}_4\text{AH}_{13}, \text{C}_2\text{ASH}_8$  [10].

These changes are obvious on XRD (Fig. 11, patterns b and c), where the intensity of portlandite peaks decreases and the calcium hydrosilicate-identifying peaks emerge. Other hydrates are not

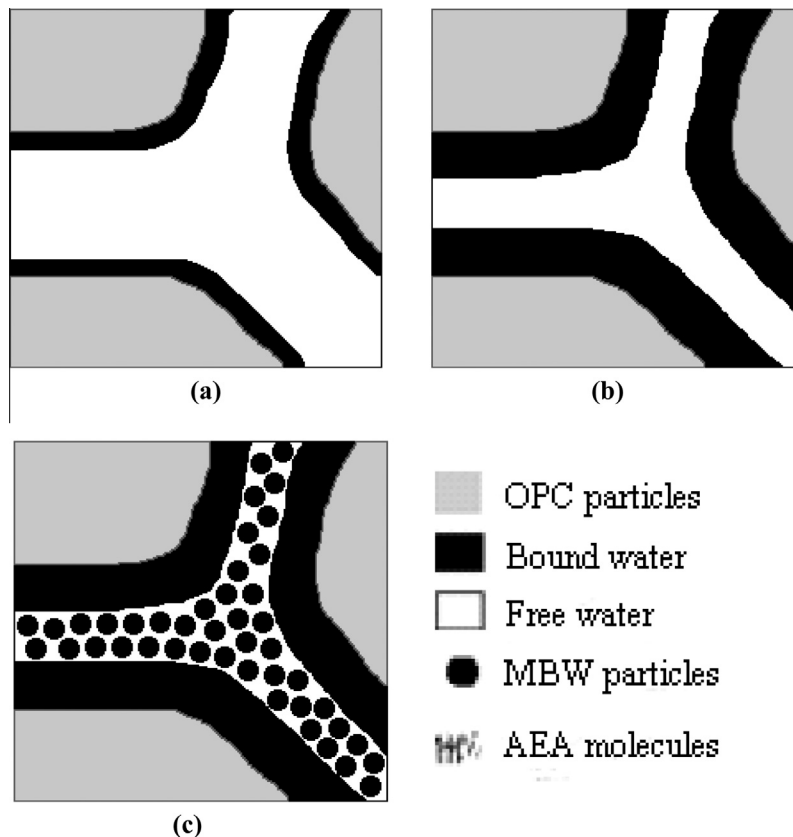
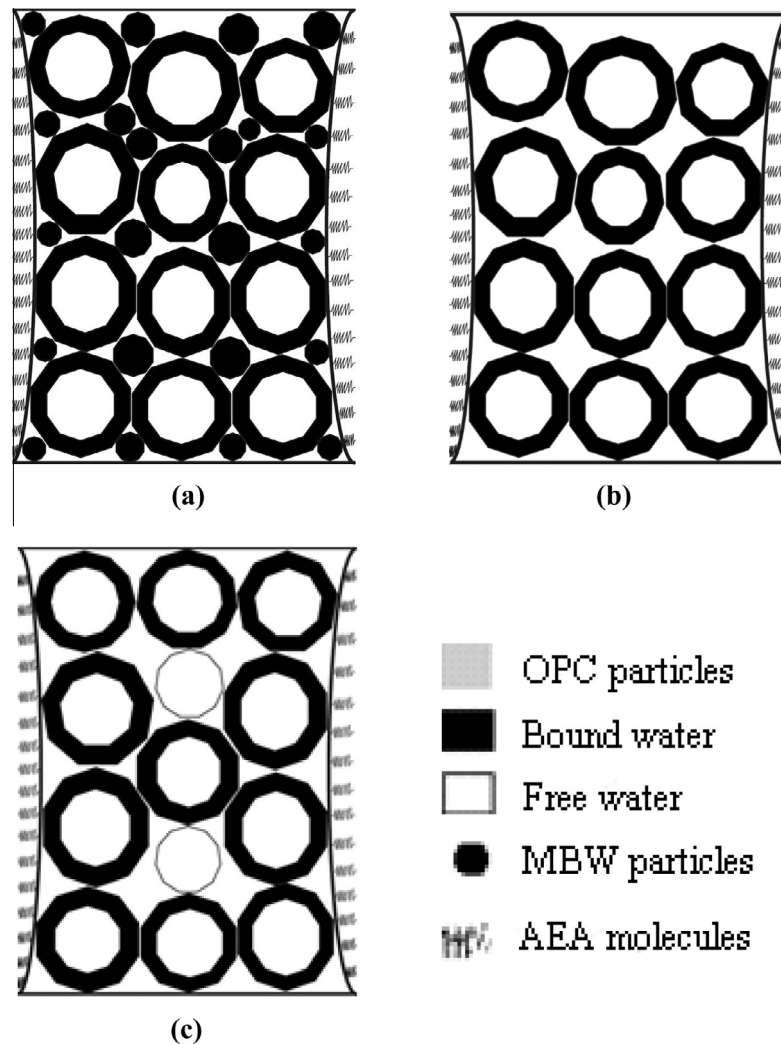


Fig. 12. Principal schemes of fresh cement paste, modified with different additives and admixtures: a – OPC; b – OPC + PA; c – OPC + PA + MBW.



**Fig. 13.** Principal schemes of particle interaction in fresh cement paste, modified with different additives and admixtures: a – OPC + MBW + PA + AEA; b – OPC + PA + AEA, c – OPC + MS + PA + AEA.

visible on XRD because of the small amount of new formations and a small degree of crystal formation.

After adding an AEA to the compacted fresh cement paste mixture, there begins aeration during the mixing. AEA molecules adsorb on the surface of OPC particles and create a connection with the newly formed air bubbles, interact among solid, liquid and air phases, which will need to be overcome a pressure force resulting in inner cavities, so that air bubbles cover as large surface of fresh cement paste mixture particles (OPC and MBW) as possible [5]. This process mechanically breaks down the connections, generated by the OPC particles, and increases the content of air bubbles in contact zones of AEA molecule phases. This process is illustrated schematically in Figs. 12 and 13.

In assessing the amount of components in the fresh cement paste mixture, we can see that in case of pure OPC, the mixture's density is the highest one and should be aerated to a certain size only. Density of the fresh cement paste when OPC partly replaced by MBW is smallest and, depending on the mixing time, can be reduced from 2186 to 735 kg/m<sup>3</sup>, while other compositions of mixtures do not allow to achieve it (see Fig. 5). Porous interpose wall mass in the mixture exceeds tension forces on the air bubble surface, so, the mixture aeration level declines.

It should be noted that the density of mixtures with any compositions, mixed from 2 to 10 min, decreases; however, the

density of porous cement paste, with the mixtures mixed for 2 min, decreased, and, after mixing the mixture for 5 and 10 min, the density of porous cement paste varied insignificantly. This means that the air content in the mixture after 2 min of mixing is stable. Tension forces on the air film surface ensure their stability, and density of cement paste decreases most due to evaporated water weight loss. Meanwhile, the density of porous hardened cement paste, with the mixtures mixed for 5 and 10 min, exceeded the density of the cement paste, with the mixtures mixed for 2 min, which means that the degree of aeration decreased. Mixtures with OPC partly replaced by MBW were most stable, while the pure OPC or when OPC partly replaced by MS were less stable.

## 5. Conclusions

1. It was found that when OPC is partly replaced by MBW with AEA and PA, hydration of fresh cement paste is prolonged from 400 to 1100 min, reaching the maximal temperature of exothermic effect of 90–60 °C. Hydration process when OPC partly replaced by MBW becomes slightly longer, though the highest temperature of exothermic effect is 8 °C higher due to additional pozzolanic reaction of the product of hydration of OPC mineral – portlandite.

2. It has been found that when OPC is partly replaced by MBW together with PA and AEA reduces the density of fresh cement paste by 2.9 times – from 2100 to 765 kg/m<sup>3</sup>, after 10 min of mixture mixing. It is 29% lower than the density of fresh cement paste with MS together with PA and AEA, and 32% lower than the density of the fresh cement paste with pure OPC together with PA and AEA. The density when OPC is partly replaced by MBW with AEA amounts to about 735 kg/m<sup>3</sup>, while the pure OPC – 962 kg/m<sup>3</sup> and with the MS – 828 kg/m<sup>3</sup>. It should be noted that a longer mixture mixing is useless technologically and economically
3. When PA is used together with AEA, the dry density decrease is most obvious of hardened cement paste with OPC partly replaced by MBW – 1450, 1120 and 795 kg/m<sup>3</sup> after 2, 5, 10 min of mixing, with pure OPC was accordingly 1820, 1490 and 1150 kg/m<sup>3</sup>, and with OPC partly replaced by MS – 1720, 1400 and 1100 kg/m<sup>3</sup>.
4. Compressive strength of hardened cement paste, as well as the dry density, decreases depending on the amount of entrained air. The lowest compressive strength of hardened cement paste is obtained when OPC is partly replaced by MBW with AEA, comparing with hardened cement paste with pure OPC or OPC partly replaced by MS. After 10 min of mixing, the compressive strength of hardened cement paste with pure OPC decreased from 95 to 4.5 MPa, with OPC partly replaced by MBW – from 73 to 1.5 MPa and with OPC partly replaced by MS – from 85 to 3.0 MPa.
5. It has been found that when OPC is partly replaced by MBW with AEA and PA, it influences the changes in sub-microcrystalline structure of hardened cement paste. That allows the hardened cement paste to have low density – 795 kg/m<sup>3</sup> and sufficient compressive strength – 2.5 MPa (after 10 min of mixing).

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