

The Effect of Plasticizing Admixture on the Physical and Mechanical Properties of Concrete with Limestone Cement

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crossref <http://dx.doi.org/10.5755/j01.ms.19.3.2304>

Received 24 August 2012; accepted 06 January 2013

The article analyzes the effect of plasticizing admixture content on the physical and mechanical properties of concrete made of 50 % coarse aggregate. The plasticizer dosage levels proposed by the manufacturer (0.2 % – 1.2 %) were examined and the optimum plasticizer dose (0.8 % – 1.0 %) was established. The use of limestone Portland cement and the optimum content (0.8 % – 1.0 %) of plasticizing admixture in the production of concrete increases the compressive strength, density, frost resistance, whereas the water absorption is reduced. The test results have revealed that plasticizing admixture insufficient or overdosing can reduce the physical-mechanical properties of concrete. With the optimum content of plasticizing admixture durable and frost resistant concrete can be obtained.

Keywords: limestone cement, concrete, compressive strength, plasticizing admixture, physical and structural properties.

INTRODUCTION

Although concrete is a universal, reliable and one of the traditional materials used in construction, its production and properties change constantly as a result of new admixture development, growing energy costs and stricter environmental requirements. Limestone and slag are some of the most prospective admixtures both technologically and economically. It would be reasonable to develop the application of limestone Portland cement in Lithuania because limestone is a local raw material and the cheapest admixture. This type of Portland cement is also widely used in Europe. The attempts to reduce carbon footprint opens the future to composite cements. Limestone-blended cements have been widely studied [1]. The main effects of limestone filler are of physical nature. It improves the packing of cement granular skeleton and facilitates the dispersion of cement grains [2–3]. Furthermore, limestone filler acts as the nucleation seeds for the precipitation of CSH [4]. These simultaneous effects accelerate the hydration of cement grains. Due to the complex action – physical and chemical – the limestone filler induces changes of the hydration processes, physical-mechanical properties and durability of concretes. The presence of limestone in the binding system accelerates the initial hydration cement, especially the reaction tricalcium silicate [5]. Studies have shown that much finer limestone particles ‘lubricate’ the clinker particles and ensure a more uniform distribution of cementitious particles and a smoother and denser structure of hardening cement framework, especially in the cases of low W/C ratio [6–7]. Researchers have found [6] that limestone powder has dispersion properties and improves the distribution of fillers, such as SiO₂ dust in the cement matrix.

Yahia [8] has proved that limestone filler, having a lower density than clinker, improves the yield of the cement paste at the same W/C ratio and also improves the workability of concrete and filler distribution. Tsivilis et al [9] have found in their studies that limestone filler in cement clinker reduces water demand required to produce a neat cement paste of normal consistency. The substitution of 15 % cement clinker with limestone reduces water demand by 2 % compared with water demand without clinker substitution and with 35 % clinker substitution water demand reduces 3 %. Other researchers [10], however, found that fine limestone filler increases the viscosity and W/C ratio of the cement paste and that has an effect on concrete durability characteristics, such as frost resistance and resistance to chemical agents. Researchers have found [7, 11] that limestone filler either improve or has no effect on the initial strength, but it reduces the strength after 7 days or longer of curing. Tsivilis et al. [9] have also established that limestone affects concrete’s strength characteristics in two ways: high limestone content (35 %) reduces the compressive strength after 28 days of curing significantly almost 40 %, compared with ordinary cement clinker without fillers, whereas a small amount of limestone filler (5 %) improves the compressive strength slightly, about 5 %. It was established that the substitution of 5 % – 10 % of clinker by limestone results in higher compressive strength after 2 and 7 days of curing compared to the specimens without limestone filler.

Superplasticizers (SP) are widely used in concrete and have become common components of concrete, and the concrete properties are significantly influenced by such incorporation. The presence of mineral admixtures such as limestone in cement may affect the interaction between the SP and the cement [12]. The studies of limestone-blended cements and the SP have focused primarily on superplasticizers of polycarboxylate ether (PCE) and pointed the importance of the molecular weight and the

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structure of the PCE on the rheological behaviour and the water-reduction of limestone blended cement [1]. Studies have shown [2, 13–14] that all kinds of SP work well for limestone cements in contrast to portland-pozzolana cements. It was found that in high-performance concretes with low W/C ratio (0.25–0.30), where SP supplementation is necessary, limestone filler (up to 15%–20%) does not have any significant effect on concrete strength properties compared to concrete without admixtures [13–14]. It should be noted that the strength of such concrete after longer curing periods may be slightly lower (7%–10%), but this concrete has smaller unconnected air voids and therefore lower permeability and better durability performance characteristics [13, 15]. Limestone filler, in contrast to other very fine constituents, improves the effectiveness of some SP [16]. For scientific and practical purposes, one of the key points in designing a composite cement concrete mix is to select the most suitable type and the optimum dose of SP. Despite the importance of admixture compatibility, the interactions between blended cements and SP have been scantily studied, and the interactions between limestone particles and SP have barely been researched at all. The issue of using Portland-limestone cement (CEM II/A-LL 42.5 R) made in Lithuania is important for concrete producers because this type cement has low plasticity, and inexperienced producers, unaware of the optimum SP dose, face certain rheology, structure formation, compressive strength and durability problems, arising from the interaction of SP and the main constituents of concrete. The producers are also interested in increasing the coarse aggregate content in concrete, but the interaction of SP with cement in such concrete needs further investigation. The goal of this study is to analyze the effect of the dosage of the mostly used SP on rheological, mechanical and structure formation properties of coarse-grain concrete made of Portland-limestone cement to establish the optimum dose of SP in concrete based on the test results and the effect of SP on freeze-thaw performance.

MATERIALS AND TEST METHODS

Limestone cement CEM II/A-LL 42.5 R produced by Akmenės cementas was used for the study. This cement is composed of 80%–94% clinker and 6%–20% limestone LL (CaCO₃) with a maximum total organic carbon ≤0.2% by mass and meets LST EN 197-1 requirements. The basic Portland cement characteristics are presented in Table 1. The coarse aggregates represent the most important component of concrete mixes; in our study it constituted 50% of dry concrete mix. The concrete was produced of natural raw materials selected in such proportions that would meet the requirements for the concrete (density, strength, durability, production process and workability). Concrete compositions differed by SP dose and water content (Table 2).

0/4 mm fraction sand meeting LST EN 12620 requirements was used as fine aggregate. 4/16 mm fraction gravel coarse aggregate meeting LST EN 12620:2003 requirements was used. Potable water of room temperature meeting LST EN 1008 requirements and dry aggregate were used to produce concrete mixes.

Superplasticizer (SP) based on synthetic polycarboxylate polymer was used.

Table 1. Characteristics of portland cement

Designation	Bulk density, kg/m ³	Specific density, kg/m ³	Specific surface area g/cm ²
CEM II/A-LL 42.5 R	1100	3100	4100

The cement and fillers were batched by mass and water was added by volume. SP was mixed into water used to produce cement pastes.

Concrete mixes were produced mechanically in the laboratory. Cube specimens of 10 cm in size were prepared and compacted on the vibration plate. After 24 hours the specimens were removed from the moulds and immersed into 20 °C temperature water before testing. Strength properties of concrete specimens were tested after 7 and 28 days of curing in water. The density of specimens was measured according to LST EN 12390-7:2009. The compressive strength was tested according to LST EN 12390-3:2009 procedures.

Table 2. The composition of concrete with SP

Mark	C, kg	S, kg	G, kg	W, kg	SP, kg	W/C	Slump class
0	455	700	1190	180	0	0.4	S3
0.2	455	700	1190	180	0.91	0.4	S3
0.4	455	700	1190	180	1.82	0.4	S3
0.6	455	700	1190	162	2.73	0.36	S3
0.8	455	700	1190	162	3.64	0.36	S3
1.0	455	700	1190	144	4.55	0.32	S3
1.2	455	700	1190	144	5.46	0.32	S3

Ultrasonic pulse velocity (UPV) in cement stone specimens was measured by PUNDIT tester. Ultrasonic pulse velocity was measured according to LST EN 12504-4:2004 requirements. Exothermic reaction (EXO) temperature tests were made according to the methodology developed by Alcoa. A cement paste specimen of 1.5 kg was placed into (10×10×10) cm Textolite mould. A T-type thermocouple with glass tubing was inserted into the specimen during curing. The mould filled with concrete specimen was immediately placed in a metal box insulated with 50 mm polystyrene foam. Temperature results were continuously recorded on the computer.

General characteristics of structural parameters by which the properties of concrete samples were analysed are presented in table 3 [17].

TEST RESULTS AND DISCUSSION

Exothermic reactions (EXO) occur during cement hardening and the heat released during the reaction increases the specimen temperature. EXO tests enable to establish the effect of different admixtures on the hydration process, i.e. EXO temperature and time to reach the maximum EXO temperature. EXO temperature tests (Figure 1) show that at the constant W/C ratio (composition with (0–0.4) % SP) the increase in SP content significantly

Table 3. Characteristics of structural parameters

Name of parameter and dimension	Calculation formulas	Description of components of the main parameter and dimensions
Effective porosity, %	$W_e = \rho \frac{m_1 - m_0}{m_0} \cdot 100$	ρ – sample density, g/cm ³ , m_1 – mass of sample saturated in normal conditions, g, m_0 – mass of sample dried to the constant mass, g.
Total open porosity, %	$W_r = \rho \frac{m_2 - m_0}{m_0} \cdot 100$	ρ – sample density, g/cm ³ , m_2 – mass of sample saturated in vacuum conditions, g, m_0 – mass of sample dried to the constant mass, g.
Reserve of pore volume, %	$R = \left(1 - \frac{W_e}{W_r}\right) \cdot 100$	W_e – effective porosity, %, W_r – total open porosity, %

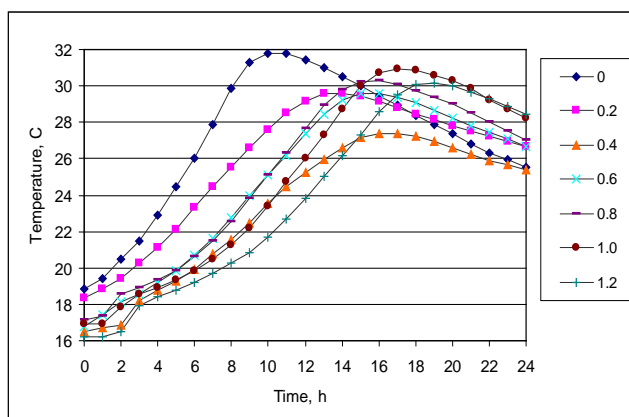


Fig. 1. Dependence of EXO temperature and duration on SP content in concrete

reduces the initial temperature of the paste and extends the time of reaching the maximum EXO temperature from 9.5 h to 16 h. With the increase of SP content in concrete (0.6 %–1.2 %) and decrease of W/C ratio, the maximum EXO temperature is achieved after 15–18 hours. It should be noted that in all concrete with SP the maximum EXO temperature is 1 °C–5 °C lower than in the reference specimen. It has been observed that the change in specimen temperature during hardening and especially the maximum EXO temperature was significantly influenced by the changing W/C ratio. Irrespective of higher SP content (0.8 %–1.2 %) in specimens the maximum EXO temperature was in the range of 30 °C–30.8 °C at the constant W/C ratio; higher SP content may retard the hydration process and the maximum EXO temperatures reduce as much as 5 °C. According to Winnefeld and Friedemann et al [18–19] all SP retarded the hydration of portland cements. Possible hydration mechanisms discussed in literature, e.g. [20], are that (i) adsorbed polymer molecules hinder the diffusion of water and calcium ions on the surface of the cement particles, (ii) complexes form between calcium ions and polymers in the pore solution, and (iii) the dispersive action of SP changes growth kinetics and morphology of hydrate phases. The retardation of cement hydration by PCEs is mainly due to adsorption of the polymers on the surface of the cement particles and due to growth kinetics and morphology of early hydrates, which is supported by the investigations. Tests have shown that with higher SP content and lower W/C ratio the maximum temperature of hydration is similar to the temperature in the reference specimen although it is

reached later. It means that under such conditions SP cannot cover the surface of cement particles in full and significantly retard the hydration process.

The tests of density in concrete specimens cured in water and dried until constant weight (Figure 2) have shown that at constant W/C ratio specimens with higher SP content had higher density compared to reference specimens. That is stated by Carazeanu et al [21], claiming that SP role is the intensifying action due to the stronger dispersion of the particles in aqueous medium and direct incorporation into the bulk of amorphous gels and by surface sorption onto the more crystalline phases. When the maximum dose of SP is added and W/C is reduced, the density of hardened cement increases ~3 % up to 2395 kg/m³ compared to the reference specimen having the density of 2331 kg/m³.

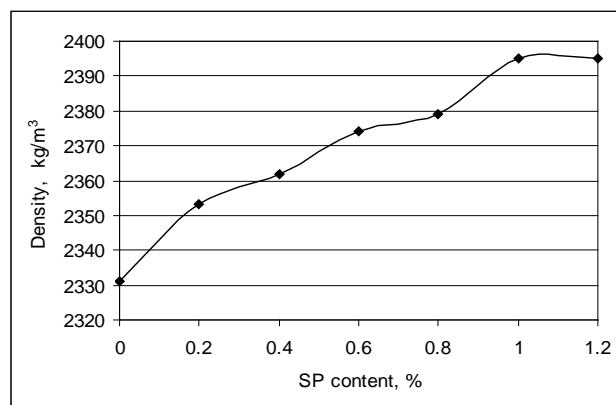


Fig. 2. Dependence of density on SP content in concrete

After 7 days of hardening in water (Figure 3) the highest strength of 55 MPa–66 MPa is achieved when SP content in the cement mix is 0.8 %–1.2 %. The lowest compressive strength was recorded in the reference specimen without SP (~40.9 MPa). The compressive strength in specimens with the minimum SP content of 0.2 %–0.6 % changed from 43 MPa to 52 MPa. We can see that even a small dose of SP slightly improved the strength properties of concrete. Apparently, better strength properties are partly due to higher density of these specimens (Figure 2). Similar trends were also observed after 28 days of hardening (Figure 3): the compressive strength of reference specimens did not change and the strength of specimens with the minimum SP content increased in proportion to SP content. The most significant strength changes were observed in concrete containing

0.4 %–0.8 % SP. In concrete specimens with 0.8 %–1.0 % SP the compressive strength increased to 68 MPa–75 MPa, and in specimens with the maximum SP content of 1.2 % the compressive strength reduced to 66 MPa. It is known [5] that the additions of the limestone filler in cement cause significant decreases of the compressive strengths, in comparison with the reference, especially after 28 days of hardening. In the initial stages of the process, the increase in the hydration degree in compositions the dilution effect of the binding material by the limestone filler, as a consequence of the formation of a higher volume of hydrates. Therefore, pastes, mortars and concretes can develop higher initial strengths, but diminished after a long time, compared to ordinary Portland cement, because the binding dilution effect becomes important. Our tests have shown that SP interacts with limestone cement and after 28 days of curing the compressive strength does not decrease; on the contrary, the strength increases, except for specimens with the maximum SP content. According to the test results, the optimum SP content in concrete is between 0.8 and 1.0 %. In summary we may claim that high strength properties in concrete made of limestone cement can be achieved only when SP is added at maximum levels.

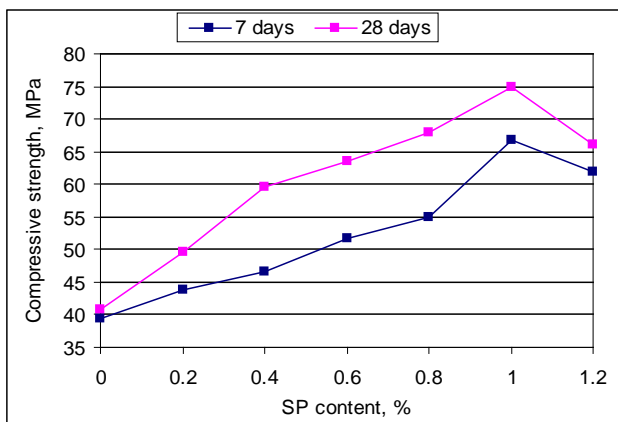


Fig. 3. Dependence of compression strength on SP content in concrete after 7 and 28 days of hardening

UPV test method enables to observe the process of structure development in hardening binders [3–4, 21]. UPV tests in concrete specimens after 7 and 28 days of hardening reveal a certain relation between the compressive strength and development of concrete structure (Figure 4). In spite of constant W/C ratio in specimens with 0.2 %–0.4 % SP, after 7 days of hardening UPV values are 150 m/s–200 m/s higher compared to the reference specimen. That shows that SP participates in the process of concrete structure development. When SP content is increased from 0.6 % to 1.2 %, UPV increases much slower. After 28 days of hardening a small drop in UPV was observed in all specimens thig SP concrete. These findings contradict the data given in referenced literature [22–23] claiming that UPV in specimens increases with time. However, such UPV changes may be related to the aforementioned changes in the volume of hydration products. Irrespective of such UPV changes the previously observed dependence on SP content remains.

Water absorption is one of the most important parameters influencing the durability of concrete and, ultimately, its performance. Limestone cement concrete can give lower water absorption rate compared with pure cement concrete and the higher is the value of the effective W/C ratio, the higher is absorption [24]. The test results have revealed that higher SP content reduces water absorption and this that improving the properties of hardened concrete (Figure 5). The water absorption in reference specimens was 4.39 %, whereas by adding SP at optimum levels between 0.8 %–1.0 % the water absorption was reduced to 3.2 %–2.88 %. The water absorption stopped changing (2.9 %) when the maximum SP content of 1.2% was used Therefore we may state that water absorption decreases (~30 %) with the increase of SP content up to 0.8 %–1.0 %. These results were confirmed by [25] claiming that the influence of SP is expressed by an increase in the compressive strength after 28 days, lowering of water absorption, and improvement in frost resistance compared to concrete without admixture.

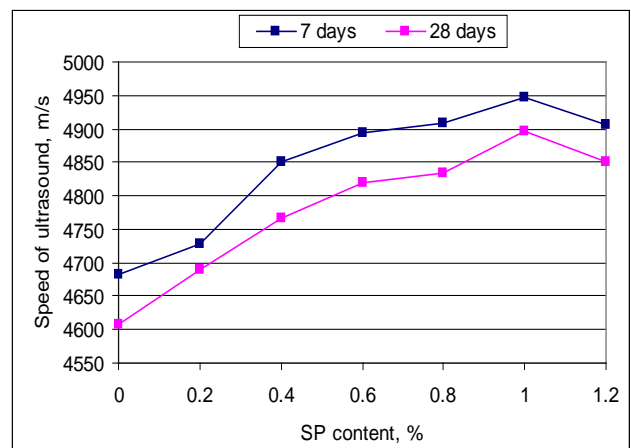


Fig. 4. Dependence of speed ultrasound on SP content in concrete after 7 and 28 days of hardening

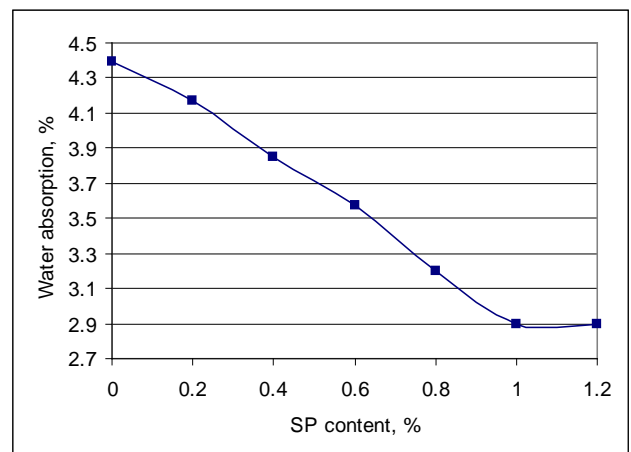


Fig. 5. Dependence of water absorption on SP content in concrete

Porosity is fundamental importance for practically all properties of cement-bonded building materials that are relevant to durability. The analysis of the dependence of effective porosity on the content of SP in concrete (Fig. 6) has shown that effective porosity decreases in proportion with higher SP content. The effective porosity in

specimens without SP is 10.75 % and reduces to 7.09 % with the maximum SP content of 1.0 % – 1.2 %.

The dependence of total porosity on SP content (Figure 6) shows that lower SP content increases the total porosity, which is 13.89 % in the reference specimen. When SP is added at the optimum level of 0.8 % – 1.0 %, the porosity goes down to 10.5 % – 9.43 %. Higher SP content of 1.2 % does not reduce the porosity.

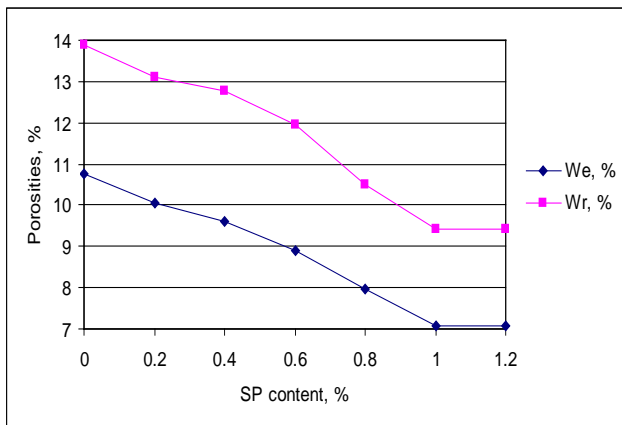


Fig. 6. Dependence of effective (We) and total porosity (Wr) on SP content in concrete

Having the values of effective and total porosity we can calculate the reserve pore volume – an indicator showing the part of the specimen’s pore space, which initially is not filled with water, but fills up later in cyclic tests. The results of reserve pore volume are presented in Figure 7. These tests show that the reserve pore volume increases with higher SP content in concrete. The lowest reserve of 22.61 % is observed when SP content is 0 %, and with SP content being 1.0 % the reserve goes up to 29.86 %.

Theoretical exploitation frost resistance to cold cycles are given in Figure 7.

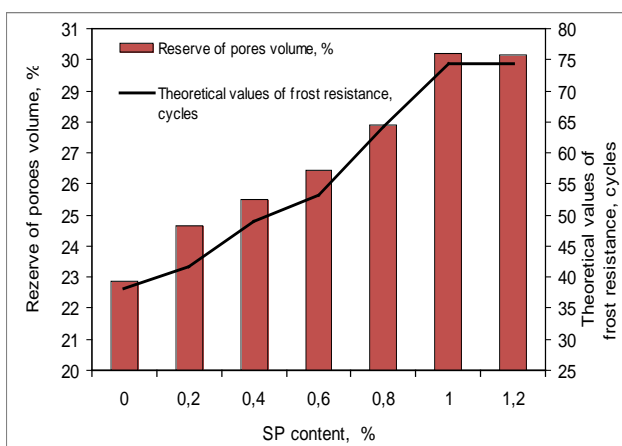


Fig. 7. Dependence of reserve of pore volume, theoretical values of frost resistance on SP content in concrete

The calculations show that the theoretical frost resistance in conditional cycles increases in proportion with SP content. When no SP is added to concrete, the number of conditional cycles is 39.24. When SP is added at the optimum level (1.0 %), the number of conditional cycles increases almost two-fold and reaches 75.9.

CONCLUSIONS

1. The tests have shown that SP content must be carefully selected in concretes made of limestone cement because SP content has an effect on the physical, mechanical and structural properties of concrete. The optimum SP content in concrete is 0.8 % – 1.0 % by weight of the limestone cement.
2. The process of hydration in concrete specimens depends on SP content and W/C ratio. The increase of SP content at constant W/C reduces the maximum EXO temperature and extends the time of reaching it. The increase of SP content in concrete extends the time of reaching the maximum EXO temperature from 9.5 h to 18 h and reduces the temperature from 32 °C to 27 °C compared to the reference specimen. Lower W/C ratio and higher SP content (0.6 % – 1.2 %) reduce the maximum EXO temperature insignificantly.
3. The compressive strength of the reference specimen is 40.9 MPa after 7 days of hardening in water, in specimens with the minimum SP content (0.2 % – 0.6%) it is 43 MPa–52 MPa, and in specimens with 0.8 % – 1.2 % SP content it is 55–66 MPa. After 28 days of hardening the compressive strength of concrete specimens increases (41 MPa–75 MPa) with the increase of SP content, except for the specimens with the maximum SP content of 1.2 %, where the compressive strength dropped from 66 MPa to 62 MPa.
4. Ultrasonic pulse velocity increased from 4664 m/s to 4896 m/s after 7 days of hardening when SP content was increased from 0 % to 1.0 %. In specimens with the maximum SP content of 1.2 % UPV reduced to 4830 m/s. In specimens with SP content of 0.2 % – 0.4 % SP at constant W/C ratio UPV values are 150 m/s – 200 m/s higher compared to the reference specimen. This is because SP is involved in the process of concrete structure formation. This statement was proved by density and strength tests. After 28 days of hardening UPV slightly reduced in all specimens due to the change in the volumes of hydration products.
5. Higher SP content reduces water absorption from 4.39 % in the reference specimen to 3.2 % – 2.88 % in specimens with the optimum SP content of 0.8 % – 1.0 %. The increase of SP content gradually reduces the effective and total porosity: the effective porosity reduces from 10.75 % to 7.09 % and the total porosity decreases from 13.89 % to 9.43 %. The maximum SP content of 1.2 % has no effect on either the effective or total porosity.

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