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Lightweight concrete for 3D-printing with internal curing agent for Portland cement hydration

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Abstract. The development of 3D printing technology requires solving material science problems to ensure the rheology of concrete mixtures with controlled mobility and high retention of volume after extrusion and also the creation of favorable conditions for the hardening of the binder. This paper investigates lightweight concretes on hollow microspheres. The rheological properties of concrete mixtures and the peculiarities of the structure formation of cement stone in the presence of a superabsorbent polymer (SAP) have been studied. The paper used standardized test methods following EN 1015-3-2007, EN 12390-1-2009, and employing modern equipment and tools (K100 KRUSS processor tensiometer, comparator, calorimeter). It was found that concrete mixtures on hollow microspheres with an average density of 1400 kg/m^3 have a high ability to retain volume (buildability) of more than 90 % after extrusion with mobility of not more than 135 mm of the spread diameter. It has been shown that the use of superabsorbent polymer solutions with controlled polymerization is an effective solution to provide internal curing for the hydration of Portland cement. Calorimetric analysis of cement stone showed a positive effect of SAP on the processes of structure formation of cement stone. This is expressed by an increase in the amount of hydration products, in particular portlandite. The number of SAP solution of 0.50–1.0 % of the mass of Portland cement in lightweight concrete provides the least reduction in strength. It was found that the flexural strength varies in the range of 5.5–5.8 MPa and the compressive strength – 45.3–47.8 MPa. An increase in the SAP content of more than 1.5 % of the mass of Portland cement is characterized by a decrease in compressive strength by 8.5 %. The permissible amount of SAP in concrete is limited to 1.0–1.5 % of the mass of Portland cement. The possibility of providing internal curing for the hydration of Portland cement in lightweight concrete through the use of SAP solution has been substantiated. The obtained results of the study of lightweight concrete show high printability of concrete mixtures on hollow microspheres for 3D printing. Moreover, the implemented soluble SAP composition instead of the granular SAP is capable of providing the function of internal curing for the hydration of cement with no loss in strength.

1. Introduction

Interest in 3D printing technology in construction is constantly growing. It is evident from the many publications [1–5]. World experience in the development of equipment for construction 3D printing shows that the mobility of the concrete mix is selected in a wide range of rheological properties, depending on the features of the extrusion nozzle of the printer [6–10]. The principal technological feature of the extrusion process is the presence or absence of direct formation of the layer. In the first case, the concrete mixture is supplied according to the principle of form-less molding (Fig. 1,a [6]) using a rectangular extrusion nozzle.

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In the second case, the mixture just is supplied from a nozzle, usually of circular cross-section, without the formation of a layer (Fig. 1,b [6]). Requirements for the viscous-flow properties of the concrete mixture for each of the methods are significantly different.

a)  b) 

Figure 1. Extruded concrete layers produced using by forming (a) and non-forming (b) nozzle of printer.

As the main criteria for concrete mixtures in 3D printing technology, formability or extrudability, flowability, retention of volume (buildability), and retention of mobility (open time) are distinguished. The variability of these properties should ensure the continuity of the layer formation through the printer nozzle. And high mobility is necessary to ensure minimum deformation after extrusion of each layer and maximum storage time to improve interlayer adhesion [8].

The specificity of these properties and their interdependence complicates the universalization of their quantitative assessment using standard test methods. At the same time, various design features of 3D printers (including feeding and extrusion systems) require an individual approach to the selection of the "acceptable" suitability of the concrete mix for printing (printability).

Cement-based dispersion systems are usually considered to be thixotropic materials. That is, they can liquefy under mechanical stress and thicken when the load is removed. In this case, the description of the shear stress (τ) from the shear rate (γ) is carried out using the Herschel-Bulkley model:

$$\tau = \tau_0 + K \cdot \gamma^n, \quad (1)$$

where τ_0 is shear stress, K is consistency index, n is an indicator of non-Newtonian behavior.

In the process of 3D printing, the lower layers, in addition to their deformation, are subjected to additional influence from the upper layers. Due to the thixotropic dilution of the mixture, these influences can lead to plastic destruction of the structure (Fig. 2 [7, 11]). In this case, the limiting value depends on the density of the concrete (ρ) and the height of the structure (H) [12]:

$$A_{\text{tix}} \geq \frac{\rho g H}{\sqrt{3}}, \quad (2)$$

where g is gravitational constant.

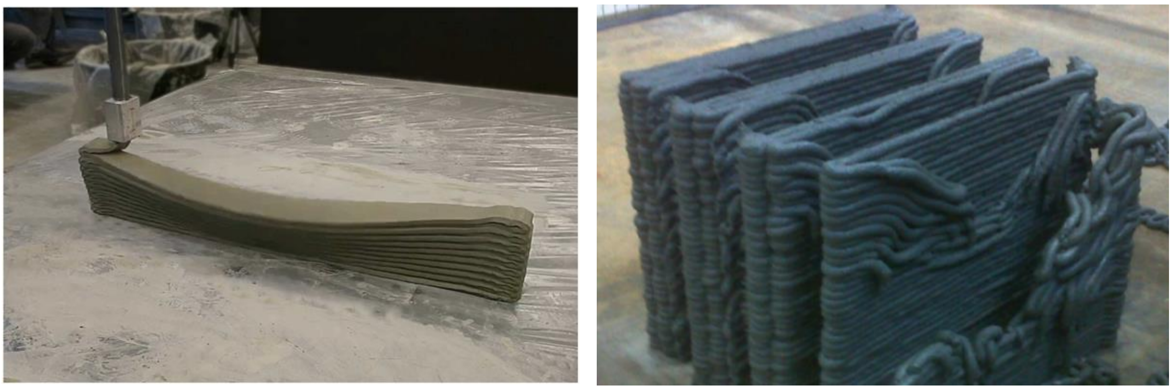

Figure 2. Plastic failures of 3D printed constructions.

Thus, it is natural to conclude that the use of lightweight concrete mixes is promising. Following (2) the lower average density of such compositions in comparison with heavy concrete will allow to increase the height of the extrusion layer with equal liquefaction or to increase this limit of liquefaction. Taking into account the positive experience of obtaining lightweight concrete with high strength [13, 14] one of the tasks can be formulated as the need to ensure the possibility of controlling the rheology of mixtures saturated by the gas phase. The object of research is lightweight concrete on hollow microspheres.

At the same time, the construction of concrete layers by extrusion at the construction site is associated with the emergence of a large open surface area of the finished product. The lack of external care in such conditions creates unfavorable preconditions for the hydration of Portland cement. Intense water loss due to evaporation leads to a lack of water for structure formation and a subsequent decrease in concrete density, shrinkage, cracking, and loss of strength.

The indicated disadvantages are less pronounced when providing a reserve of water in the particles of the porous aggregate, the so-called internal curing. This approach is known in the technology of lightweight concrete [15, 16] when the preparation of lightweight concrete is preliminarily performed by water saturation of the aggregate. A similar function can be performed by special polymer additives, the so-called superabsorbent polymers (SAP) [18–20]. As a rule, these are micrometer-sized granules or fibers capable of absorbing water up to 50 times the original volume. As a rule, these are micrometer-sized granules or fibers capable of absorbing water in an amount more than 50 times the original volume.

Currently, experience in the use of SAP in cement systems has been accumulated. There is a positive effect of SAP on reducing shrinkage and as a result, reducing the risk of cracking. However, there are several disadvantages. One of them, SAP polymer granules, require adjusting the water consumption in the mixtures to maintain their mobility [21, 22].

That is, the use of superabsorbent polymers in cement systems is characterized by both positive and negative effects [18, 23, 24].

On the one hand, SAP in the cement composite is the carrier of the water reserve to ensure the hydration of the binder. This has the positive effect of reducing shrinkage. On the other hand, the granular polymer component not only requires preliminary water saturation (up to 30 minutes) to ensure sufficient mobility of the mixture but also is a source of additional pores by reducing its volume, contributing to a decrease in mechanical properties.

Previously, the authors proposed a solution [25] which consists of using solutions of acrylate compositions with delayed polymerization for the internal curing of cement. Such solutions allow to form the polymer films at the right time, starting the process of retaining water in the volume (absorption) after extrusion of the mixtures. It is shown that the use of such a solution reduces the loss of strength of the cement stone during hardening in unfavorable conditions.

Thus, research aimed at solving the cumulative problems of ensuring the required rheology of concrete mixtures and proper care for the hydration of cement after molding is an actual direction for the development of effective compositions for 3D printing technology in construction. To achieve this goal, it is necessary to determine the mobility and buildability of the extruded volume of the concrete mixture of lightweight concrete, to establish the effect of the superabsorbent polymer solution on the mobility, strength, and shrinkage of the concretes, and to substantiate the effectiveness of such a solution to ensure the internal curing of cement hydration.

2. Methods

The object of study is lightweight concrete on hollow microspheres with an average density of 1400 kg/m^3. Concrete is made according to [26] using Portland cement CEM I 42.5 N Lipetskcement, microsilica MK-85 (NLMK) with a density of 2200–2350 kg/m^3, a particle size of 1...100 microns, and a SiO_2 content of more than 97.8 %, quartz sand 0.16–0.63 mm fraction, quartz sand flour with a specific surface of 700...800 m^2/kg, water and plasticizer Melflux 1641F. Hollow glass microspheres "Foresphere 3000" (average particle size 30 microns) were used as a lightweight aggregate to reduce the average concrete density. The subject of research is the rheological and physical-mechanical properties of concrete compositions on hollow microspheres with SAP solution. Acrylate composition "Renovir-hydrogel" [21] was used as a SAP solution. The SAP solution is obtained by mixing water (W) with the three components of the polymer part ($\Sigma A = A_1 + A_2 + A_3$) and catalyst (B). The component "A_1" is acrylic acid (propenoic acid – $CH_2 = CH - COOH$) or salt (sodium polyacrylate $[–CH_2 – CH(COONa)–]_n$). The component "A_2" is a crosslinking agent in which poly-saturated compounds are widely used. The component "A_3" is an initiator from peroxides, hydroperoxides, hydrogen peroxide, persulfates, azo compounds, or redox systems. Varying the concentrations of each component allows to control the polymerization process and form a different degree of crosslinking and polymerization speed (Table 1).

Table 1. Compositions of the studied concrete.

Variation of the plasticizer			Variation of the SAP			
No	Name	C_{Plast}, % by weight of cement	No	Name	C_{SAP}, % by weight of cement	Ratio W/ΣA
1	P/C-1.0	1.0	6	A/C-0	–	–
2	P/C-1.1	1.1	7	A/C-0.4	0.4	95.0
3	P/C-1.2	1.2	8	A/C-0.8	0.8	47.0
4	P/C-1.3	1.3	9	A/C-1.5	1.5	23.0
5	P/C-1.4	1.4	10	A/C-2.3	2.3	15.0
			11	A/C-3.9	3.9	8.5

Notes: C_{Plast} is concentration of plasticizer; C_{SAP} is the amount of acrylate part of SAP (ΣA); W is the amount of water.

The mobility of the lightweight concrete mix with varying concentration of a plasticizer (1.0...1.4 % of the mass of Portland cement (Table 1)) was determined by the diameter of the cone spread according to EN 1015-3-2007. Retention of volume (buildability) was additionally evaluated. It was evaluated by the change in the volume of the mixture extruded from a metal cylinder with a ratio of diameter to height of 1: 2.

The evaluation of the compatibility of the superabsorbent polymer with the polycarboxylate plasticizer was carried out by changing the surface tension of aqueous solutions using a K100 KRUSS processor tensiometer.

The assessment of the degree of hydration was carried out using the calorimetric method by the total thermal energy [27]. Calorimetric analysis of cement mixtures was carried out for compositions with W/C-ratio 0.5 within 72 hours using an isothermal calorimeter. Cement compositions with SAP were prepared with a polymer content of 0.5...1.5 % of the mass of Portland cement at a constant catalyst B/A_1 = 0.003.

The compressive strength tests were carried using static loading by the servo-hydraulic press “Advantest 9” according to EN 12390-1-2009. The shrinkage deformations were determined by the change in the distance between the reference points during hardening in unfavorable conditions (temperature 25... 27 °C and humidity 50…60 %) using a comparator.

3. Results and Discussion

As shown earlier [28], the mobility of lightweight concrete on hollow microspheres substantially depends on the polycarboxylate plasticizer. Varying its amount is one of the most effective prescription methods for establishing a parity combination of mobility and buildability suitable for 3D printing.

After extrusion from a 3D printer nozzle, the concrete mix changes its original shape under its weight. Ensuring minimal layer deformation is important for such mixtures. In this work, a study was carried out on the preservation of the volume of the mixture after extrusion from the cylinder. Such deformations can be quantified by the value of the vertical settlement of the layer. However, vertical deformation does not fully reflect the volume change, since the layer also changes in the horizontal plane. It is also important for 3D-printing.

Since both the height and the diameter of the cylinder are subject to change than a parameter that takes into account both these changes is taken as the criteria for the preservation of the volume of the concrete mixture. In the initial state, the volume of the mixture takes the shape of a cylinder. In this case, the largest and smallest diameters are $D_1 = D_2$. Therefore, the volume for each of them (at $H = H_0$) is equal to $V_1 = V_2$ (Fig. 3). Then the maximum preservation of the form expressed through the ratio V_1/V_2 will be equal to 1. After deformation, the volumes of the concrete mix specimens calculated with D_1 and D_2 will differ. The V_1/V_2 ratio will also bi changed. Thus, the ratio of the volumes V_1/V_2 and heights $H / H_0 \rightarrow 1$ characterize the best preservation of the concrete mix shape (Fig. 4).

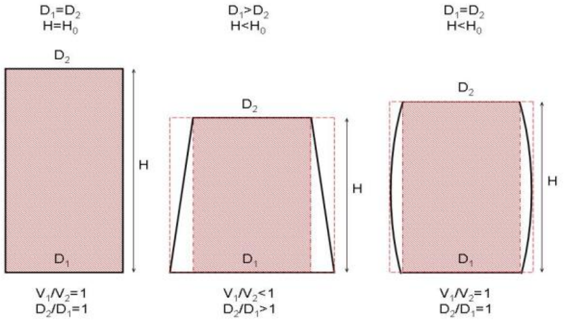

Figure 3. Change in cylinder volume.

Figure 4. The nature of the slump of the concrete mixture after extrusion from the cylinder.

The study of the change in the mobility and buildability of concrete mixtures of lightweight concrete on hollow microspheres on the amount of plasticizer was carried out in this work (Table 2).

Table 2. Rheotechnological properties of concrete mixes for 3D printing.

No	Name	Spread diameter, D_{sp}, mm	Buildability, V_1/V_2	Relative deformation, H/H_0
1	P/C-1.0	129 ± 2	0.97 ± 0.02	0.95 ± 0.02
2	P/C-1.1	135 ± 3	0.94 ± 0.02	0.93 ± 0.02
3	P/C-1.2	160 ± 4	0.88 ± 0.03	0.90 ± 0.03
4	P/C-1.3	165 ± 3	0.83 ± 0.03	0.84 ± 0.01
5	P/C-1.4	182 ± 5	0.62 ± 0.02	0.78 ± 0.02

Table 2 shows that an increase in the spread diameter of the concrete mixture due to the increase in the plasticizer leads to a decrease in buildability. At the same time, more than 90 % of the cylinder shape is retained with a plasticizer content of not more than 1.1 % of the mass of Portland cement. This corresponds to the mobility of the mixture according to the standardized method of 135 mm.

Fig. 5 shows the effect of the rheological properties of the mixture on the geometry of the extruded rectangular layers. It is seen that an increase in the amount of plasticizer, leading to an increase in the mobility of the mixture, contributes to the deterioration of the retention of the shape of the layers.

Figure 5. An example of deformations of extruded lightweight concrete layers in the horizontal (left) and vertical (right) planes.

Thus, it has been established that concrete mixtures on hollow microspheres with an average density of 1400 kg/m^3 can provide a high buildability of more than 90 % with mobility of up to 135 mm along the spread diameter at the varying content of the plasticizing additive. That is, the cross-section of the layer after extrusion will change within 10 %. In practice, it will ensure the stability of the technological process of manufacturing a structure using the 3D printing method (from adjusting the stroke of the printer nozzle to the consumption of materials).

For the studied compositions, the content of the plasticizer in an amount of no more than 1.1 % of the cement mass makes it possible to provide variability in the intensity of liquefaction at the high buildability. Thus, we can conclude that concrete mixes on hollow microspheres are highly suitable for 3D printing in construction.

Also, it can be concluded that it is necessary to develop a universal method for assessing the suitability of concrete mixes for 3D printing by rheological criteria. And the existing standardized test methods require the establishment of boundary ranges of properties for specific types of 3D printers.

Both the mobility of the mixture and its strength after extrusion are important for the development of materials for 3D printing. As is known, an important condition of a controlled increase of design strength values of concrete is the W/C-ratio. However, the large exposed surface of the extruded layers has a significant impact on 3D printing technology. The evaporation of moisture leads to a decrease in the actual value of the W/C-ratio and a deficiency of water for hydration of the binder and also shrinkage. The use of a superabsorbent polymer in the solution form with a controlled polymerization process, in contrast to granular analogs, should allow preserving the mechanical properties of concrete in unfavorable conditions. The results of studying the effect of SAP solution on the properties of compositions for 3D printing are presented in Table 3.

Table 3. Rheological properties of mixtures and physical and mechanical properties of lightweight concrete with SAP solution.

No	Name	D_{sp}, mm	ρ, kg/m^3	R_{fl}, MPa	R_{com}, MPa	ε, mm/m
1	A/C-0	225 ± 2	1370 ± 30	5.52 ± 0.12	45.3 ± 1,1*	1.22 ± 0.05
2	A/C-0.4	212 ± 3	1360 ± 20	5.59 ± 0.09	45.2 ± 0,9	1.16 ± 0.03
3	A/C-0.8	199 ± 2	1360 ± 35	5.81 ± 0.14	47.8 ± 1,0	1.02 ± 0.05
4	A/C-1.5	181 ± 2	1355 ± 30	6.83 ± 0.15	47.6 ± 1,2	1.00 ± 0.04
5	A/C-2.3	180 ± 1	1355 ± 30	6.33 ± 0.14	44.2 ± 1,0	0.95 ± 0.04
6	A/C-3.9	170 ± 1	1325 ± 25	6.02 ± 0.10	37.1 ± 0,8	0.89 ± 0.03

Notes: D_{sp} is spread diameter, ρ is average density, R_{fl} is flexural strength, R_{com} is compressive strength, ε is relative deformation, * is the compressive strength of lightweight concrete hardened under normal conditions is 57.0 ± 1.4 MPa.

It was found that the mobility of lightweight concrete mixes on hollow microspheres decreases after the introduction of SAP in contrast to cement pastes [27]. An increase in the content of polyacrylates to 2.3 % by weight of Portland cement leads to a decrease in the spread diameter of the mixture by 20 %. This pattern can be associated with a significant contribution of the plasticizer to the provision of the rheological properties of such compositions.

One of the possible reasons for the decrease in the mobility of the concrete mix may be the unsatisfactory compatibility of the plasticizer solution and SAP components. However, an assessment of the compatibility of the plasticizer solution and the components of the superabsorbent polymer indicates that this assumption is not valid. The results of the study of changes in the surface tension of the solution of the used plasticizer (0.04 %) from the amount of the acrylate part of SAP are presented in Table 4.

Table 4. Dependence between the surface tension of the plasticizer solution and the content of the acrylate part of SAP.

The physical state	SAP solution concentration, W/ΣA					
	0	95.0	47.0	23.0	15.0	8.5
Before polymerization	47.5 ± 1.4	44.5 ± 1.4	44.4 ± 1.6	43.7 ± 1.3	43.4 ± 1.4	42.5 ± 1.3
After polymerization		45.2 ± 1.3	45.0 ± 1.3	44.6 ± 1.5	44.5 ± 1.2	43.0 ± 1.3

The obtained results show that each of the components of the studied solution (polycarboxylate plasticizer and acrylate SAP) reduces the surface tension regardless of the polymerization degree of SAP. This indicates the compatibility of the plasticizer and SAP. That is, it has been found that the used polycarboxylate plasticizer and polyacrylate SAP can be combined to prepare a solution for producing concrete mixtures and lightweight concrete products.

Thus, it has been shown that each of the concrete compositions under study has sufficient mobility of the mixture to perform technological operations. The mobility of the mixtures D_{sp} > 150 mm ensures sufficient formability during extrusion and buildability after extrusion of the next layer (Fig. 6). The rational concentration of SAP in the developed concrete receipts should not exceed 1.5 % of the mass of Portland cement.

Figure 6. Example of extruded lightweight concrete layers with SAP.

As shown above, the use of granular SAP leads to a decrease in the strength properties of cement composites. This is due to the formation of additional porosity in the structure of the material after desorption of water from the polymer additive. Therefore, the use of SAP in the form of a solution to reduce shrinkage deformations can be justified by the preservation of mechanical characteristics. Table 3 shows the dependence of the flexural strength and compression of lightweight concrete on the number of SAP.

It has been established that the use of SAP solution in the amount of 0.50–1.0 % of the mass of Portland cement provides the smallest decrease in concrete strength. The flexural strength of such concretes varies in the range of 5.5–5.8 MPa, and the compressive strength – 45.3–47.8 MPa at the age of 28 days of hardening in unfavorable conditions. The amount of acrylate part of not more than 2.3 % of the mass of Portland cement in the composition of concrete provides an increase in flexural strength by 17 %. This may be due to the formation of polymer films or fibers that perform the function of a reinforcing additive after desorption. At the same time, an increase in the SAP content of more than 1.5 % of the mass of Portland cement is characterized by a decrease in compressive strength by 8.5 %. Then, the permissible amount of SAP in concrete is limited to 1.0–1.5 % of the mass of Portland cement.

The study of the shrinkage showed the possibility of reducing the deformations of lightweight concrete by 25 % due to the introduction of SAP. The use of SAP solution in small amounts (up to 0.25 % of the mass of Portland cement) leads to a decrease in shrinkage deformation by 26.9 %. The maximum reduction in shrinkage compared to the control composition is achieved with a SAP content of 2.3 %. Taking into account the change in rheological and physical-mechanical properties, varying the SAP content in the range of 0.8–1.5 % of the mass of Portland cement provides the best shrinkage value.

Since the use of SAP is justified by the possibility of providing internal care during the hydration of Portland cement, an important condition for such compositions is the establishment of parity concentrations of the polyacrylate solution, which do not lead to a decrease in the degree of hydration.

The integral thermograms of the total heat release of cement mixtures (Fig. 7) show that the range of SAP concentrations less than 1.5 % provides a heat release level similar to the control composition (without SAP). This indicates a similar degree of binder hydration. That is, the process of SAP polymerization in the indicated concentrations is carried out during the period when the competition between the sorption of polyacrylates and the hydration of Portland cement is the least. At the same time, the presented data of calorimetric studies indicate some inhibition of the process of hydration of Portland cement in the presence of a superabsorbent polymer. But, the effect of SAP is not limited to the first 72 hours of the hydration and hardening process. Assessment of the influence of SAP on the structure formation of cement stone at a later date was carried out by the magnitude of the heat flow in the temperature range of 470 ... 510 °C.

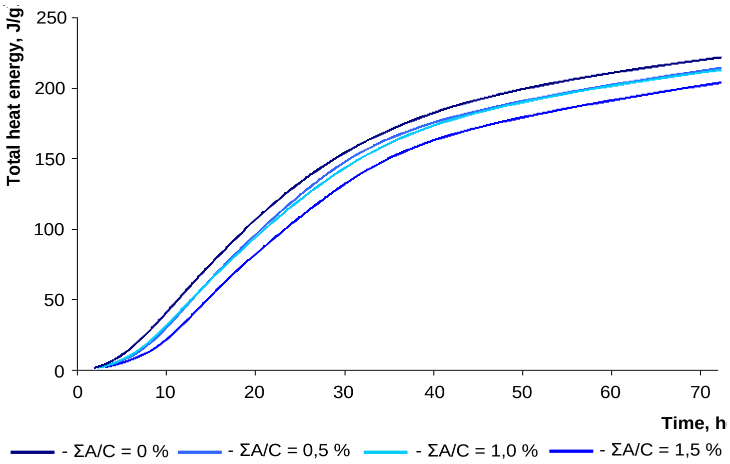

Figure 7. Kinetics of heat energy during hydration of Portland cement with SAP.

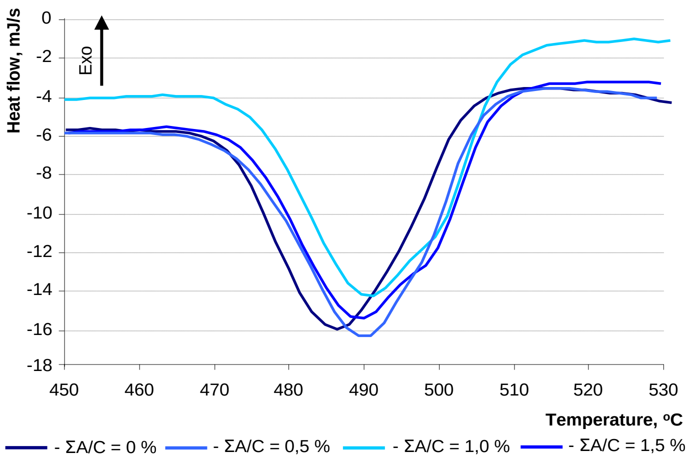

Figure 8. Thermogram of cement stone in the temperature range 470…510 °C.

The total area of the anomaly for the endothermic effect (S) (Fig. 8) of cement stone in the specified temperature range is proportional to the amount of portlandite. This allows to conclude about the number of hydration products or the degree of binder hydration. It is seen that an increase in the SAP content leads to an increase in the area of the anomaly for the endothermic effect. The maximum is achieved when the content of the superabsorbent polymer is 1.0 % by weight of Portland cement (Table 5). A further increase in SAP leads to a decrease in enthalpy. However, the area of the anomaly for the endothermic effect at 1.5 % by weight of Portland cement is more than the S value for the control composition.

Table 5. The total area of the anomaly of the cement stone in the temperature range 470…510 °C.

Index	Ratio A/C, %			
	0	0.5	1.0	1.5
S, mJ·ºC/s	223.0	228.9	252.7	239.7

The study of the structure formation of cement stone samples after hardening for 28 days was carried out. Differential thermal analysis shows an increase in the enthalpy of decomposition of portlandite in cement stone, hardening in the presence of SAP. That is, it can be concluded that the degree of hydration of Portland cement is increased. Thus, the hypothesis about the possibility of using SAP in the form of solutions with controlled polymerization instead of granular additives for the internal curing of Portland cement in the development of concretes for 3D printing technology was confirmed.

The obtained results of the study of lightweight concrete show the high suitability of concrete mixtures on hollow microspheres for extrusion using a forming nozzle of a 3D printer [6]. Also, the implemented solution of using SAP in the form of a solution instead of a granular form is capable of providing the function of internal curing for the cement [18, 21] without loss of concrete strength.

4. Conclusions

Based on this research the following conclusions were made:

1. Concrete mixtures on hollow microspheres with an average density of 1400 kg/m^3 provide buildability after extrusion of more than 90 % and mobility of up to 135 mm at varying content of the plasticizing additive. The content of the plasticizer is not more than 1.2 % by weight of the cement allows to vary the intensity of the dilution for the studied mixtures. The tested concrete mixtures are highly suitable for 3D printing in construction. The tested concrete mixtures have high buildability for 3D printing in construction.

2. The use of SAP solutions with controlled polymerization is an effective solution for providing internal curing for the Portland cement in the production of concrete for 3D printing. The calorimetric analysis shows the positive influence of SAP on the processes of structure formation of cement stone. The SAP in an amount of not more than 1.0 % of the mass of Portland cement corresponds to the maximum amount of Portlandite formed during the hydration of Portland cement.

3. The use of SAP solution in an amount of 0.50...1.0 % of the mass of Portland cement provides the least reduction in the strength of lightweight concrete. The flexural strength varies in the range of 5.5...5.8 MPa and the compressive strength – 45.3...47.8 MPa. An increase in SAP content of more than 1.5 % of the mass of Portland cement is characterized by a decrease in the compressive strength by 8.5 %. The optimal amount of SAP in the concrete composition is limited to the range of 1.0...1.5 % of the mass of Portland cement.

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