

EFFECT OF AMOUNT OF DEFLOCCULANT ON CHANGE IN PHYSICOMECHANICAL PROPERTIES OF MEDIUM-CEMENT HEAT-RESISTANT CONCRETES DURING DRYING AND HEAT TREATMENT

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Results are provided for a study of the properties of a cement matrix of medium-cement heat-resistant concrete with deflocculant additions, i.e., sodium tripolyphosphate and polycarboxylate ester. The effect of amount of deflocculant and heat treatment temperature on the change in matrix hydration product composition after hardening at 20°C and drying at 60, 80, and 110°C is considered. Comparative analysis is performed for the change in concrete physicomaterial properties and their heat resistance is evaluated in relation to amount and type of deflocculant.

Keywords: medium-cement heat-resistant concrete, deflocculant, strätlingite C_2ASH_8 , hydration, drying, hardening, strength, concrete structure.

INTRODUCTION

Various deflocculants are used extensively in the production of heat-resistant concrete with addition of microsilica [1 – 4]. Their effect on concrete rheological properties, and reduction in water consumption in a concrete mix, have been studied quite well [5 – 9]. It has been established that particles of aluminat cement have a positive charge, but different additions (for example microsilicon) have a negative charge, and therefore a mixture without deflocculant on mixing with water particles attract each other and water consumption increases. The distance between particles in a cement mix without adding deflocculant is not less than 10 nm, but with addition of deflocculant it increases up to 200 nm [5].

Many publications [10 – 12] have been devoted to studying heat-resistant concrete using sodium tripolyphosphate and polycarboxylate ester deflocculants. It should be noted that best results of using deflocculants have been achieved in composition with high quality fillers, for example, mullite, corundum, silimanite, and andalusite. However, concrete producers often use cheaper filler, such as chamotte, in which the Al_2O_3 content varies in the range 30 – 35%, and

therefore a study of compositions with chamotte are prospective [13 – 15]. It is well known that concrete producers often experience difficulties in selecting the optimum type and amount of deflocculant. In a guide for use of deflocculant producers indicate quite broad ranges of the their possible application (0.05 – 0.40%), although in specific recommendations about how different amounts of deflocculant may affect concrete physicomaterial properties during hardening and after heat treatment are not supplied. Indications about possible consequences of overdosing deflocculant in a concrete mix are numerous [3, 6, 16 – 18]. It is well known that deflocculants may affect concrete porosity, density, and strength [6, 19]. In previous studies [6, 9, 11] it has been established that an increase in amount of sodium tripolyphosphate within the composition of alumina cement with addition of microsilica with a hardening temperature of 20°C accelerates hydration, although concrete shrinkage increases after firing and strength properties are worse. With an increase of amount of Castament FS 20 deflocculant in a concrete mix rheological properties are improved, but hydration slows down [9, 11, 20]. The effect of sodium tripolyphosphate on alumina cement hydration has been studied previously [21]. The mechanism for selecting specimen strength in relation to amount of deflocculant has shown that use of sodium tripolyphosphate does not worsen strength properties for composites of alumina cement with addition of zeolite

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(67% SiO₂) compared with composites without deflocculant, hardened at 38°C. However, presence of strätlingite, i.e., a product of reaction between microsilica and cement mix, in a composite with deflocculant has not been established. The authors of publication [3] also consider that hardening conditions are considerably important for forming strätlingite. With hardening in water at 20°C of an alumina cement paste (W/C = 0.3) with microsilica and addition of sodium tripolyphosphate, strätlingite is detected after seven days of hardening on a level with C₂AH₈. Use of microsilica combined with sodium tripolyphosphate in pastes based on alumina cement (W/C = 0.3) has shown [2] that addition of sodium tripolyphosphate improves slurry rheological properties and reduces the drop in strength of specimens during hardening at 38°C, although the mechanism of the effect of sodium tripolyphosphate in reducing strength of specimens remains unclear. It has been established [21–24] that alkali ions are considerably important in forming strätlingite.

As is seen from this review, the mechanism of arranging strength of specimens of heat-resistant concrete with addition of microsilica in relation to type and amount of deflocculant used has not been studied sufficiently. The effects of different types of deflocculant on structure formation for hardening concrete during drying, when there is rapid conversion of crystal hydrates, has also hardly been studied. For this it is necessary to study the effect of each individual deflocculant on hydration of alumina cement, and also on cement operating properties. Therefore the aim of this work is to clarify how different amounts of deflocculant affect cement hydration during hardening and drying, and how this is reflected in physicomechanical properties of test medium-cement heat-resistant concrete based on a chamotte filler.

EXPERIMENTAL PART

The following materials were used for the study: microsilica (MS) grade RW-Fuller (SiO₂ 96.1%), whose average particle size is amount 150 nm; alumina cement Gorkal-70 (G70) (not less than 70% Al₂O₃); chamotte filler (fraction < 10 mm), obtained from chamotte brick ShA (Al₂O₃ ≥ 30%) by crushing and screening; deflocculant Castament FS 20 (FS), related to a group of polycarboxylate esters, and technical anhydrous sodium tripolyphosphate Na₅P₃O₁₀ (NT).

Seven compositions of cement matrix were prepared, consisting of G70 and MS with a ratio of 2.33:1.00, within which only the amount of deflocculant was varied. The ratio of distilled water and solid component was constant (0.27). This ratio was selected in order that experimental conditions approached to the maximum the actual ratio of water and cement matrix for concrete. For this purpose the dimensions of specimens for study was selected the same as for concrete specimens (70 × 70 × 70 mm). The first composition “0” was prepared without adding deflocculant. In the composition with addition NT and FS their amount in a matrix was 0.1,

0.2, and 0.3% of the amount of solid component; compositions were designated correspondingly NT 0.1, NT 0.2, NT 0.3 and FS 0.1 FS0.2, and FS 0.3.

X-ray phase analysis of concrete was performed in a DRON-7 x-ray diffractometer, copper anticathode, nickel filter, anode voltage 30 kV, and anode current 12 mA. An ICDD database was used in order to interpret peaks. Quantitative changes of minerals in specimens of the cement matrix after hardening for one day and drying for 1 h at 60, 80, and 100°C were evaluated from the peak height of the main test mineral diffraction maximum in an x-ray pattern.

In order to compare medium-cement properties six concrete compositions were prepared, differing only in type and amount of deflocculant. The composition of test concretes (wt.%): G70 10, MS 5, fine chamotte 20, chamotte filler 65, water 7.5 (above 100% dry weight of components). Within the compositions with addition of NT or FS their amount varied from 0.1 to 0.3% (above 100% dry components). Concrete compositions in relation to amount and type of deflocculant were designated: NT 0.1, NT 0.2, NT 0.3 and FS 0.1 FS0.2, and FS 0.3.

The structure of specimens was studied in a JSM 7600F scanning electron microscope (SEM) from JEOL, Japan. Concrete specimens were prepared and treated, and their main physicomechanical properties were determined according to GOST 20910 and LST EN 1402, dried and fired in accordance with instructions CH 156-79; ultrasonic pulse transmission rate (USP) in specimens was determined by means of a Pundit 7 instrument. Ultimate strength in compression σ_{co} of heat-resistant concrete after three days of hardening was studied in a ALPHA3-3000S hydraulic press in accordance with specifications of LST EN 12390-3:2009, thermal shock resistance was determined by a procedure described in [25]. Thermal cycling with heating to 800°C and cooling between two water-cooled metal plates was carried out on specimens 40X40X160 mm. USP was determined and thermal shock resistance criteria were calculated after the 1-st, 3-rd, and 7-th thermal cycles.

Results of X-radiographic study of specimens of cement matrix after hardening for one day at 20°C showed (Fig. 1a) that in a specimens without addition showed presence of CA (density 2.69 g/cm³), CAH₁₀ (density 1.6 g/cm³), and C₂AH₈ (density 1.95 g/cm³). In specimens with addition of NT, apart from CA, CAH₁₀, a very insignificant amount of C₂AH₈ was present. An increase in amount of NT in specimens leads to an increase in the CAH₁₀ content and a reduction in the amount of CA. This is possibly connected both with properties of the deflocculant itself (and alkali reaction in an aqueous medium), and with the fact that with a considerable amount of deflocculant the process of water molecule re-grouping of MS between grains of cement proceeds more intensely and from thicker water interlayers [26–28]; hydration occurred more intensely. In specimens with FS CA, CAH₁₀ and C₂AH₈, and also an amorphous phase are present. With an increase in the amount of FS in specimens

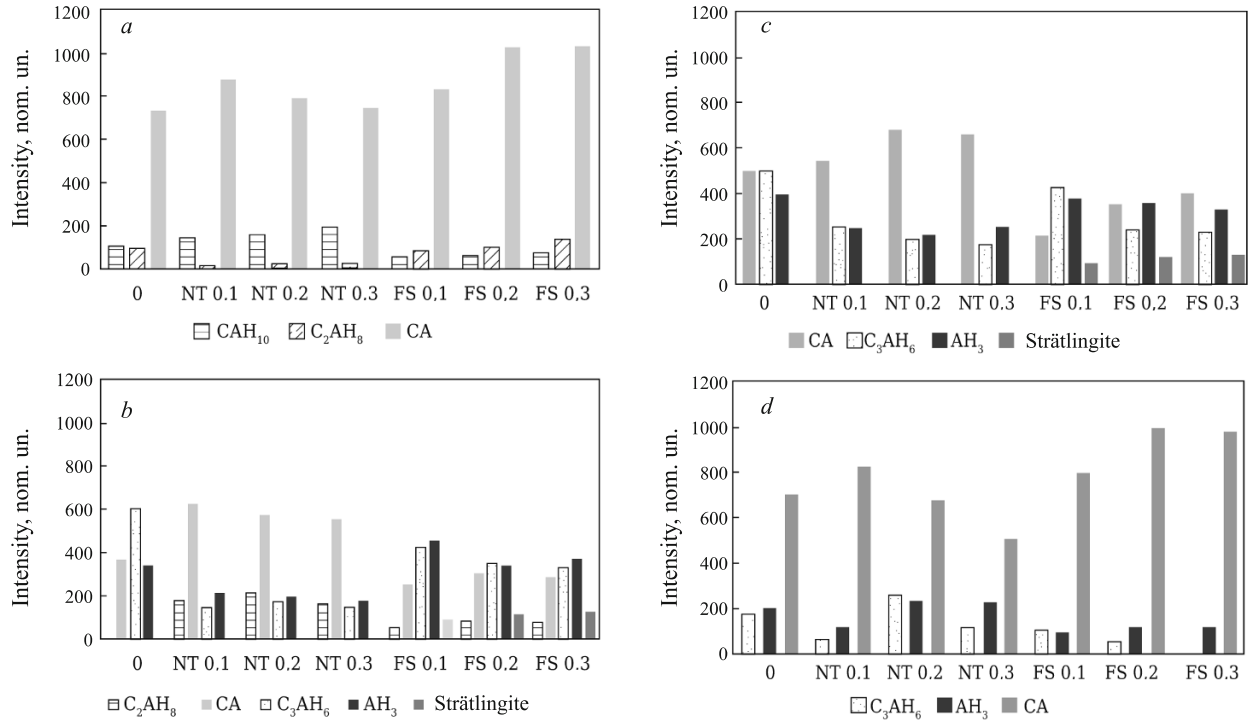


Fig. 1. Dependence of test mineral main diffraction maximum peak height in a cement matrix on amount of NT and FS after heat treatment at 20 (a), 60 (b), 80 (c), and 110°C (d).

unreacted CA remains greater, which is possibly explained by retarding hydration action of deflocculant, having an acid reaction in an aqueous medium. However, C₂AH₈ in specimens forms more than CAH₁₀. With an increase in amount of FS this tendency is even clearer.

After heat treatment at 60°C mineral CAH₁₀ was not identified within specimens of all compositions (Figs. 1b, 2); a reduction in CA and formation of minerals C₃AH₆ (density 2.53 g/cm³) and AH₃ (density 2.34 – 2.39 g/cm³) are observed. In specimens with NT, apart from the minerals mentioned above, there is rapid crystallization of C₂AH₈. In specimens with FS C₃AH₆ and AH₃ form more intensely than in specimens with NT; a product of cement mineral reaction and MS also appears, i.e., strätlingite C₂ASH₈, whose presence with hardening above 40°C has been confirmed previously by researchers [22, 29, 30]. It should be noted that in specimens with FS a reduction in the amount of C₂AH₈ alongside crystallization of strätlingite is not observed by chance; this is indicated by data [29]. The fact that during hardening of specimens with FS C₂AH₈ predominates, is a basic prerequisite for forming strätlingite. The overall amount of hydration products in specimens with FS is markedly higher than in specimens with NT. This may be explained by the fact that the amount of unreacted CA in specimens with FS is markedly less than in specimens with NT. It may be concluded that at the given temperature FS markedly accelerates both hydration and conversion.

After heat treatment at 80°C in specimens of all compositions mineral C₂AH₈ is not identified (Fig. 1c); a change is observed in the quantitative ratio of CA, C₃AH₆, and AH₃, caused by dehydration and crystal hydrate conversion. These processes are reflected in the quantitative ratios between CA and crystal hydrates. In specimens with FS this ratio is approximately 1:2, in specimens with NT it is 3:2, i.e., they are denser. Strätlingite is present in specimens with FS, and the amount of unreacted of CA in specimens with FS is even less marked than in specimens with NT. After heat treatment at 110°C (Fig. 1d) dehydration facilitates a reduction in the amount of hydration product and an increase in the amount of CA, particularly in specimens with FS. The main hydration product is AH₃, whose density is less than for C₃AH₆. Strätlingite is not identified in specimens after treatment at this temperature, which is confirmed by previous studies [31]. In specimens with an increase in the amount of NT the ratio between C₃AH₆ and AH₃ is approximately the same as with heat treatment at 80°C, and this means more dense crystal hydrates predominate.

It may be said that use of deflocculants of different type may change the ratio and composition of cement matrix hydration products both during hardening, and during heat treatment. It has been established that during heat treatment in the range 60 – 80°C within a matrix with addition of FS deflocculant hydration proceeds more rapidly, there are more products, and strätlingite forms.

Studies in an SEM showed that the microstructure of cement matrix specimens with the maximum amount of NT

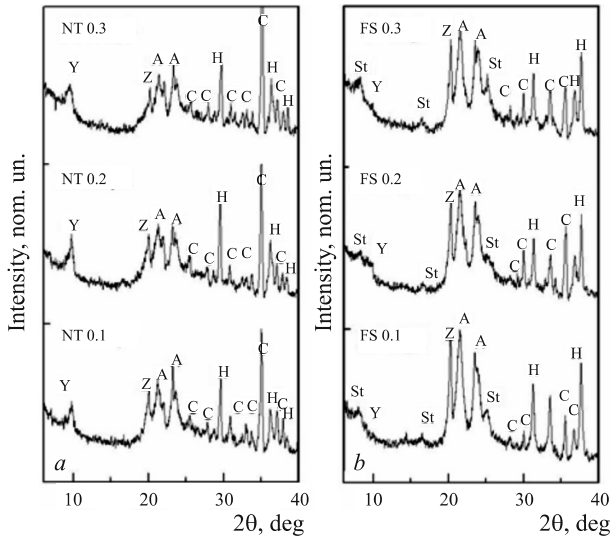


Fig. 2. Matrix specimen x-ray patterns with different amount of deflocculants NT (a) and FS (b) after heat treatment at 60°C: C) CA; H) CA₂; Y) C₂AH₆; A) AH₃; Z) C₃AH₆; St is strätlingite C₂ASH₈.

and FS deflocculants (0.3%) after heat treatment at 60°C is different. In specimens with NT (Fig. 3a) coarse aggregates of hydrated CA mineral predominate. A contact zone between MS microspheres and matrix is not observed. In specimens with FS (Fig. 3b) there are numerous contacts between matrix and MS microspheres; various new formations are seen at the surface of microspheres.

The microstructure of specimens after heat treatment at 80°C is also different. In specimens with NT (Fig. 3c) apart from MS microspheres, there are both fine flaky platelets and also radially radiating aggregates of AH₃ and coarse cubic crystals of C₃AH₆. Presence of these crystal hydrates is confirmed by previous research [32]. In analyzing the microstructure of a specimen with FS apart from aggregates of AH₃ and crystals of C₃AH₆ formation of hexagonal strätlingite platelets is noted (Fig. 3d).

In order to study the effect of NT and FS additions on physicommechanical properties of specimens of medium-cement heat-resistant concrete after hardening for 3 h, drying at 60, 80, and 110°C and firing at 1000 and 1200°C, their ultimate strength in compression σ_{co} (see Fig. 4) was studied. After hardening specimens with NT they differ insignificantly, whereas an increase in amount of FS significantly reduces σ_{co} (from 45 to 28 MPa), and this is confirmed by X-radiographic studies (CA is hydrated more slowly. These tendencies have been observed previously [6]. After drying at 60°C σ_{co} of specimens with FS increases more, the greater the amount of deflocculant within the concrete composition, i.e., correspondingly by 11, 32, and 67% compared with σ_{co} after hardening; σ_{co} of specimens with NT only increases by 5–6%. After drying at 80°C σ_{co} of specimens with NT increases by 15–28%, but specimens with FS by 6–8% compared with σ_{co} after drying at 60°C. This increase in speci-

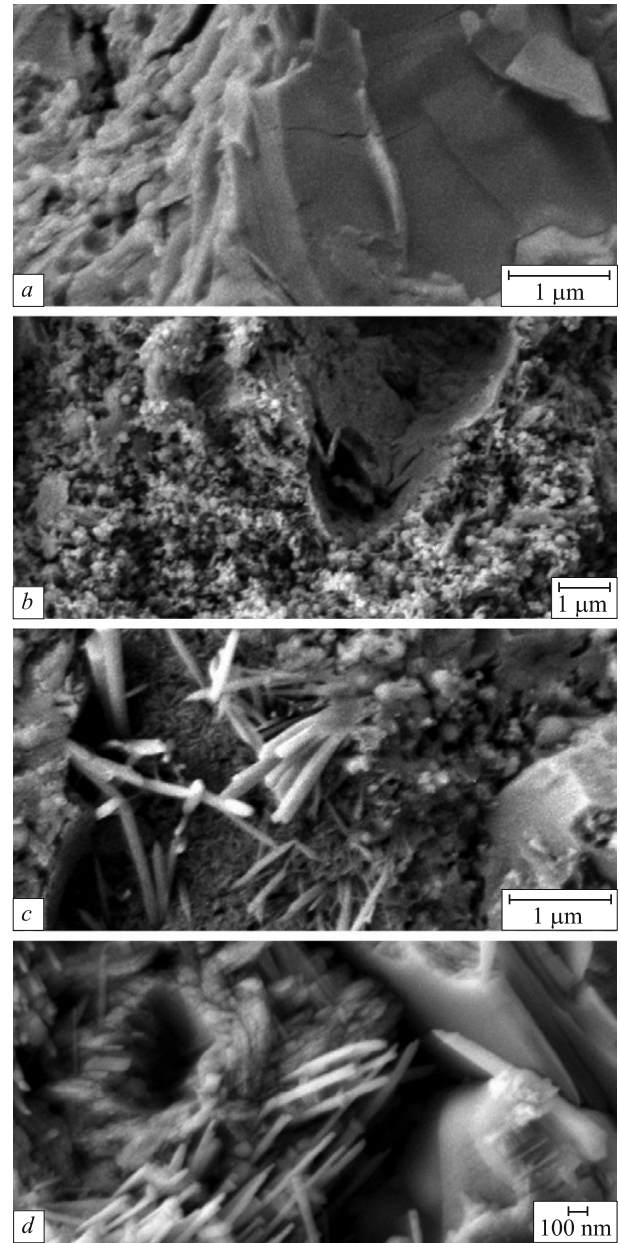


Fig. 3. Matrix specimen microstructure with addition of NT (a, c) and FS (b, d) after heat treatment at 60°C (a, b) and 80°C (c, d).

mens strength with FS may be explained by the effect of new formations [33]. It also well known that with formation of strätlingite there is less formation of C₃AH₆ and this leads to a reduction in porosity and simultaneously to an increase in σ_{co} [34]. Drying of specimens with the maximum FS addition at 110°C makes it possible to increase σ_{co} by approximately a factor two compared with σ_{co} after hardening; in specimens with NT σ_{co} increases by 35–45%. After firing specimens with FS there is a previously noted feature: with an increase in amount of FS σ_{co} of specimens increases. In addition, it is possible to note a tendency of a reduction in σ_{co}

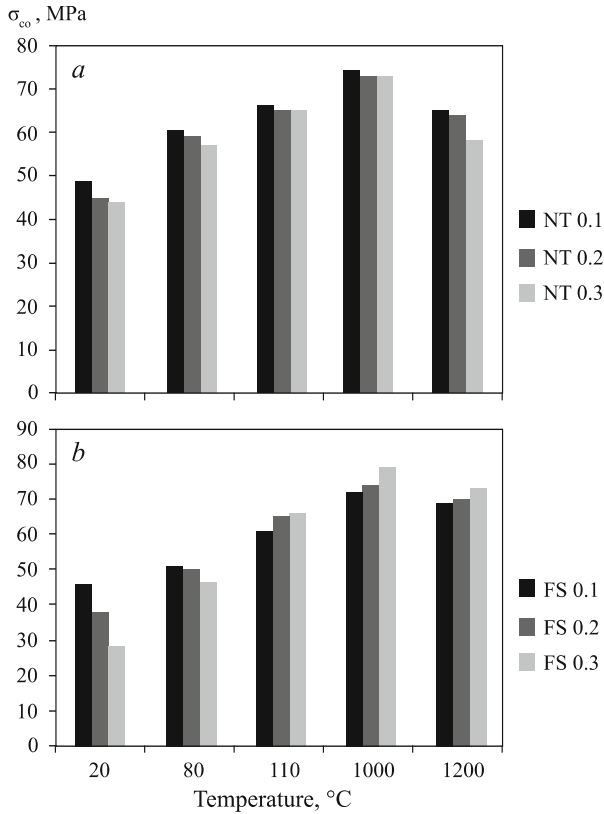


Fig. 4. Concrete specimen σ_{co} values after heat treatment at different temperatures in relation to amount of deflocculant NT (a) and FS (b).

after firing and an increase in the amount of NT in specimens.

New structures of strätlingite are possible during dehydration forming spatial contact structures (zones) within a concrete matrix and facilitate development of strength properties with a reduction internal stresses within concrete during firing. Independent of the type of deflocculant the greatest σ_{co} for specimens is achieved after firing at 1000°C. Maximum values of σ_{co} after firing at 1000 and 1200°C for specimens with FS are a little higher for specimens with NT, and this is possibly explained by less intense shrinkage [6, 11].

The effect of amount and type of deflocculant on concrete structure formation during hardening and heat treatment at 20, 60, 80, 110, 800, 1000, and 1200°C (Fig. 5) has been evaluated from the change in USP velocity in concrete specimens. It has been established that an increase in the amount of FS deflocculant in contrast slows down structure compaction during hardening, and this particularly concerns formation of coarse crystal hydrates and possible occurrence of hydration reactions within an amorphous phase.

During drying at 60°C the rate of structure compaction in specimens with addition of FS is higher, the greater the addition to the composition. However, in specimens with NT after drying at 110°C the value of USP reaches 4600 –

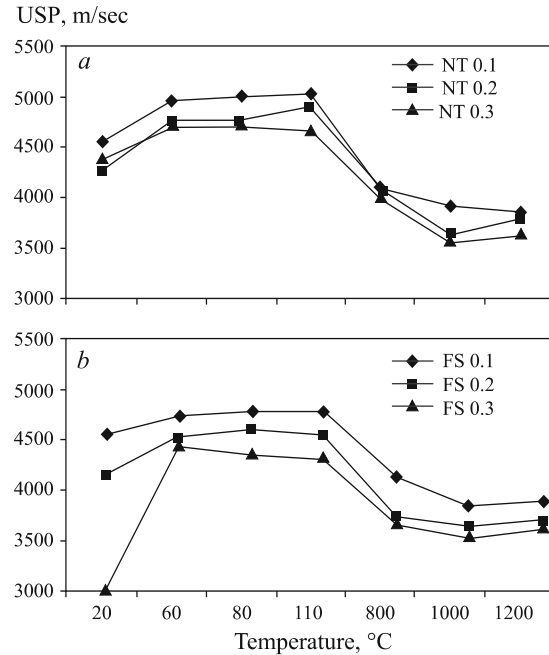


Fig. 5. Change in USP in concrete specimens in relation to heat treatment, type, and amount of deflocculant NT (a) and FS (b).

5000 m/sec, but in specimens with FS after drying it is 4250 – 4700 m/sec. This indicates that addition of FS deflocculant facilitates formation of a more porous structure.

After firing at 800 and 1000°C independent of type and amount of deflocculant USP values decrease in all specimens. However, whereas in specimens with NT this reduction is 20 – 25% of the value after drying, in specimens with FS it is 12% in total. Thus, the type and amount of deflocculant considerably affects concrete structure during firing. Concrete specimens with addition of NT, within which there is reaction between MS and cement minerals, is confirmed by destruction to a greater extent in specimens with added FS, within which these reactions proceed. An increase in amount of NT and FS deflocculant does not lead to a marked change in USP velocity in specimens after firing at 1200°C.

Thermal shock resistance of concretes (Figs. 6 and 7) was evaluated at 900°C by cyclic heating and cooling of concrete specimens fired at 800°C. The greatest change in USP occurs after the first cycle, i.e., for concrete with addition of NT the USP velocity decreased from 12 to 21%, and for concrete with FS from 13 to 15%. The higher the amount of NT in a specimen, the more specimens are subject to destruction during the first thermal cycle. For specimens with FS this difference is 2%. Then after seven thermal cycles in specimens with NT the value of USP decreases by 22 – 37% of the USP value of specimens after firing at 800°C, and for specimens with FS this difference is 24 – 27%. It may be concluded that with a high amount of deflocculant in concrete of this type there is a greater effect on concrete thermal

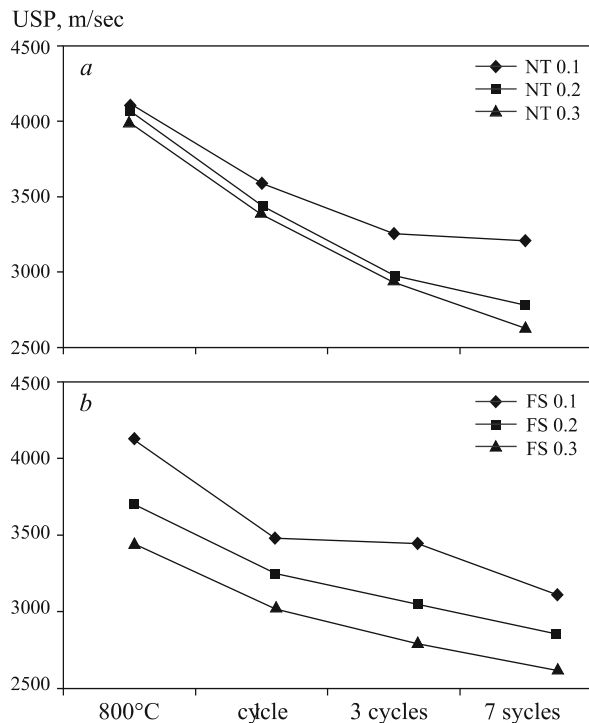


Fig. 6. Change in USP in concrete specimens with addition of NT (a) and FS (b) after firing at 800°C and after thermal cycles.

shock resistance, and specimens with NT are subject more destruction than specimens with FS.

After firing at 1000 and 1200°C, when there is specimen sintering, their shrinkage was measured. Studies showed a significant difference between the effect of both deflocculants on concrete shrinkage after firing. Shrinkage of concrete specimens with 0.1 and 0.2% NT after firing at 1000°C exceeds by a factor of two to three shrinkage of specimens with addition of FS. However, when the amount of NT and FS reaches 0.3% the amount of shrinkage approximately coincides. Shrinkage of concrete specimens with addition of NT after firing at 1200°C increases, but for specimens with addition of 0.1 and 0.2% FS it is unchanged. In a concrete specimen with addition 0.3% FS shrinkage decreases. It may be concluded that shrinkage of concrete specimens with FS in an amount of 0.1 to 0.3% is less than for concrete specimens with the same amount of NT.

CONCLUSION

It has been established that type of deflocculant and amount of it affect cement matrix structure formation, consisting of alumina cement with microsilica in a ratio (2.33:1.00), and as a consequence properties of medium-cement heat-resistant concrete. It has been established that during hardening at 20°C an increase in amount of NT in matrix leads to formation of CAH_{10} , and an increase in amount of

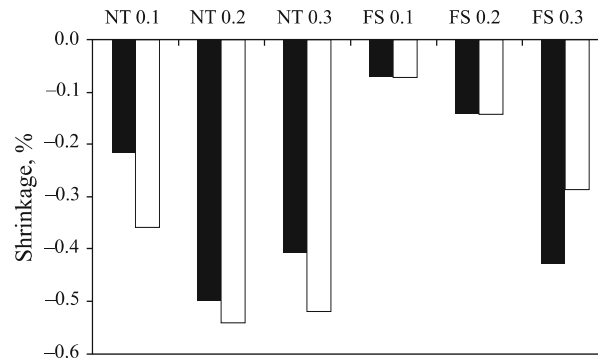


Fig. 7. Concrete specimen shrinkage after firing at 1000 (□) and 1200°C (■) in relation to type and amount of deflocculant.

FS promotes formation of C_2AH_8 . During drying at 60 and 80°C hydration reaction proceeds more intensely within specimens with FS, within which as a result of reaction of C_2AH_8 and microsilica there is additional formation of strätlingite, and this is confirmed by SEM studies. After drying at 110°C AH_3 predominates in specimens with addition of FS, and in specimens with addition of NT it is more dense C_3AH_6 .

It has been established that an increase in amount of NT additive has an insignificant effect on strength of concrete specimens after hardening, whereas an increase in amount of FS additive reduces strength. After drying at 60°C as a result of new formations the strength of specimens with FS increases more rapidly than for specimens with NT. The greater the amount of FS in specimens, the greater (by up to a factor of two) is the increase in strength after drying at 110°C. In specimens with the same amount of NT strength increases by 35 – 45%.

After firing specimens with addition of NT a tendency is followed of an increase in strength with an increase in amount of additive, and in specimens with addition of FS conversely there is some increase in strength with an increase in amount of additive. Measurement of USP velocity confirms that an increase in amount of FS additive in contrast to NT slow down structure compaction during hardening and drying, and promotes formation of a more porous structure. Therefore after firing the structure of specimens with NT is more prone to destruction than the structure of specimens with FS. The USP velocity in specimens with addition of NT decreases by 20 – 25%, but in specimens with FS by 10 – 12% of values after drying.

After firing concrete specimens shrinkage with addition of FS is significantly less than for specimens with addition of NT. Study of concrete thermal shock resistance showed that denser structure specimens with addition of NT are more subject to destruction during the first and third cycles than for specimens with addition of FS.

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