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The Effect of Deflocculants on the Structure and Physical-Mechanical Properties of Fireclay Refractory Castables

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For production of fireclay refractory castables, various deflocculants are used. In recent years new polycarboxylate ethers-based deflocculants appeared and so far their properties comparing to those of common deflocculants are little investigated. This study compares the effect of content of two deflocculants, sodium tripoliphosphate and polycarboxylate ethers Castament FS-20, on hydration of aluminate cement, on structure forming during it, pH and electrical conductivity in suspensions. It was established that the increase in content of deflocculant sodium tripoliphosphate, as a binder, in the suspension results in a considerable increase of electrical conductivity, while the increase of Castament FS-20 reduces pH values. The study also evaluates the effect of content of deflocculants on duration of exothermal reaction in paste of castable with medium content of cement, on formation of structure, physical-mechanical properties of castable. It was established by means of compressive strength tests that the increase in content of sodium tripoliphosphate affects the strength properties of castables, meanwhile Castament FS-20, on the contrary, increases the compressive strength of castable samples. Dilatometric test helped to establish a different effect of deflocculants on processes of linear changes at high temperatures.

Keywords: different type deflocculants, pH, electrical conductivity, EXO reaction, UWV (ultrasound wave velocity), MCC-type castable (medium cement content castable), linear changes.

1. INTRODUCTION

Deflocculants, otherwise called plasticizers or dispersants and in general modifiers of rheological properties, are used for improving the rheological and physical-mechanical properties of refractory castables. Due to a high effectiveness of these additives, a great attention is being paid for their investigation [1 – 8]. The principle of action of deflocculants, largely used for production of refractory castables, such as sodium polyphosphates [9 – 17] and polyacrylates (Darvan D811) [10, 12, 18 – 19], is electrostatic dispersion (strong shift of potential ξ of cement particles to negative field). However, after formation of initial cement hydrates in castables, the plasticizing effects of these deflocculants disappear. Recently polycarboxylate ethers-based deflocculants (Castament FS-20) were developed for production of castables, since these deflocculants create a double effect of dispersion, electrostatic and spatial. Their action is much more effective than that of common deflocculants [4, 13, 20 – 21].

In the laboratory there were performed the investigations of pH and electrical conductivity (EC) of aqueous solutions of well-known deflocculants, such as inorganic sodium tripoliphosphate (NT) and organic sodium polyacrylate Darvan D811, as well as recently developed organic deflocculants, such as polycarboxylate ethers Castament FS-10 and Castament FS-20 (FS) (Table 1). The deflocculants of different pH and EC will act differently on the above-mentioned indices of the castable paste, on the process of cement hydration and physical-mechanical properties of the castable hardened.

The researches as to the use of new deflocculants in castables of different types are not numerous, and a few of them are reported in [15 – 17] relating to MCC-type castable and with different content of NT [22 – 24]. It was established that increase in NT content speeds up the cement hydration, however, it increases the shrinkage of castable after burning at high temperatures and reduces the strength characteristics [15 – 16, 19]. There was also assessed a possibility to use the low-quality microsilica in MCC-type castable together with the deflocculant FS [15 – 16, 18, 25]. It was established that by increasing the content of FS, the rheological properties of castable paste can be improved. However, the influence of both deflocculants on hydration and physical-mechanical properties of castable, both after natural hardening and after thermal treatment, remained unexplained. Therefore, the purpose of this study was to consider the effect of content of two deflocculants of different types (a well known inorganic and newly developed organic), NT and FS, on properties of suspension of binder, as well as on hydration of MCC-type castable paste and on physical-mechanical properties of castables.

2. MATERIALS AND TEST METHODS

The present study deals with two different type of deflocculants – sodium tripolyphosphate ($Na_5P_3O_{10}$, molecular mass 367.86, TU 2148-095-43499406-98) (NT), cristalline material dissolving in water; and Castament FS-20 (made by BASF, Germany) (FS), is belonging to

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polycarboxilathers, a powder dissolving well in water, dispersive polymer. We have used microsilica (RW-Füller)

Table 1. pH and EC values of aqueous solutions of deflocculants (0.1 g deflocculant solved in 100 g water)

Name	pH	EC (μS/cm)
Sodium tripoliphosphate	9.4	950
Darvan D811	8.15	390
Castament FS-10	7.6	140
Castament FS-20	4.35	250

(SR) that is a product from RW silicium GmbH (SiO_2 content 96 % ±1.5 %, bulk density of (330 – 360) kg/m^3). In our research alumina cement „Gorkal 40"(AC) manufactured in Poland was applied. The content of Al_2O_3 in it is not less than 40 %. The main mineral phases are: CA, ferrite phase, $C_{12}A_7$, C_2AS; bulk density of 1160 kg/m^3, refractoriness not lower than 1280 °C. Chamotte aggregate is made by crushing chamotte bricks, the density of which is 1920 kg/m^3.The amount of Al_2O_3 is 33 % – 38 %; the bulk density of chamotte aggregate (fr. 0 mm – 10 mm) is 1260 kg/m^3. Dispersive chamotte of 80 % is made up of <0.08 mm fraction. Bulk density was 1100 kg/m^3.

For investigating of the electrical conductivity and pH of suspension, a device MPC 227 of the firm METTLER TOLEDO (electrode InLab 410, measurement precision of 0.01 pH and electrode InLab 730, measurement range of (0 – 1000) μS/cm was used. The measurements of pH and electrical conductivity in solution and suspensions were performed every 10 min. since their mixing with water total testing 2 h. Deflocculants and suspensions were made with distilled water. Deflocculants concentration in solution changes was from 0.1 % to 1.0 %. In suspensions solids-water ratio being constant as 1 : 2. The SR – AC ratio in suspensions was 1 : 2, while the content of deflocculant was also calculated in per cent of solids content.

The temperatures of exotermic effects during the binding and hardening of castable paste were registered according to the methodology devised by company Alcoa [26].

The cube shape samples of the size (70×70×70) mm were formed for 6 compositions of a castable. After 3 days of normal curing, the samples were dried at a temperature of 105 °C ±5 °C for 48 hours in an electric furnace. After they were kept for 5 hours at each of the investigated temperatures (800 °C, 1000 °C, 1200 °C) in an electronic controller furnace and cooled. The ultrasonic pulse velocity ((LST EN 1402-7) after 3 days of normal curing) and compressive strength (LST EN 1402-6) of castable were investigated.

Dilatometric analyses of the mortar samples were performed in a Linseis apparatus L76 with a heating rate of 5 °C/min in specimens of 5 mm diameter and 50 mm length.

3. RESULTS AND DISCUSSION

To the aim of assessing the effect of deflocculant content on pH and EC values, the aqueous solutions of both deflocculants were prepared and in these solutions the content of deflocculant varied from 0.1 % to 1 %. These amounts were chosen whereas the producers [27 – 28] in contrast to the researchers widely used deflocculants in range (0.03 % – 1 %). The researchers tipically use only one for the studies selected deflocculant amount [3, 12, 20, 29] or a very narrow range [9 – 10, 25].

As shown by measurements performed (Fig. 1), the increase in content of either deflocculant NT or FS does not influence practically pH values of solution. The main difference was observed between EC of solutions. In the NT case, EC varies from 300 μS/cm to 2000 μS/cm, and in the solutions with FS from 70 μS/cm to 600 μS/cm. This shows that the increase in NT content can more influence EC of solution and by this to exert a greater effect on the process of cement hydration.

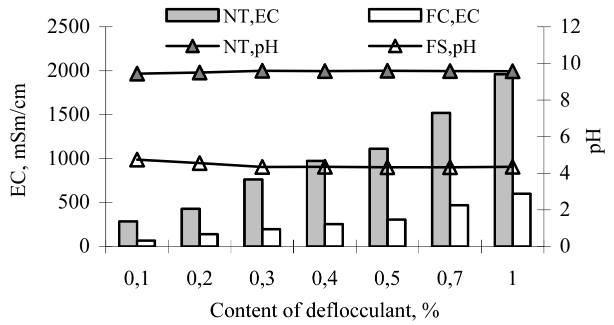

Fig. 1. Effect of deflocculant NT and FS content on pH and EC values of aqueous solutions

Since in MCC-type castable one of the main binder components is SR, the effect of deflocculant content (from 0.125 % to 0.5 %) on pH and EC changes in the suspensions of castable binder (AC and SR) was assessed (Figs. 2, 3). The initial measurements showed that with the NT increase in suspensions, the EC values increase as well (from 306 μS/cm, when NT content is 0.125 %, to 1970 μS/cm, when NT content is 0.5 %). The results confirm the investigators [3] data, showing that the deflocculant additive lead to increasing of cement suspension EC.

After 2 hours these differences decrease and the EC changes are as follows: 1520 μS/cm when NT content is 0.125 %, and 2500 μS/cm, when NT content is 0.5 %. At the beginning pH values differ considering NT content, within 9.6 and 11, and after 2 hours these differences disappear and pH varies between 11.6 and 11.8 in all suspensions. Investigators [30] also indicate that deflocculant NT has alkaline properties and increasing of deflocculant content can significantly speed up setting time of low-cement refractory castables.

In the suspensions with FS, the values of EC are notably lower, i. e. 213 μS/cm when FS content is 0.125 %, and 775 μS/cm, when FS content is 0.5 %. As in the case with NT, with the lowest concentration of deflocculant, EC grows most and after 2 hours reaches 2330 μS/cm, and in the suspension with the highest content of FS, the value of 1000 μS/cm is reached. In contrast to the NT case, pH differences are notably higher, 8 and 5.8 at the beginning of measurements, and after 2 hours – 10.46 and 7.00. It shows that using FS, one can regulate the pH values and, applying NT, the EC values in the suspensions.

In order to determine the effect of content of both deflocculants on physical-mechanical properties of MCC-type castable, we prepared 8 compositions of castable (Table 2), one half of them with NT, the other half with FS. In both cases the content of deflocculant was kept the same (0.125 %; 0.2 %; 0.35 %; 0.5 %) as in the suspensions. The compositions with NT, considering the content of deflocculant, were marked in the following way: B1; B2; B3;B4. The compositions with FS were marked accordingly: F1; F2; F3; F4.

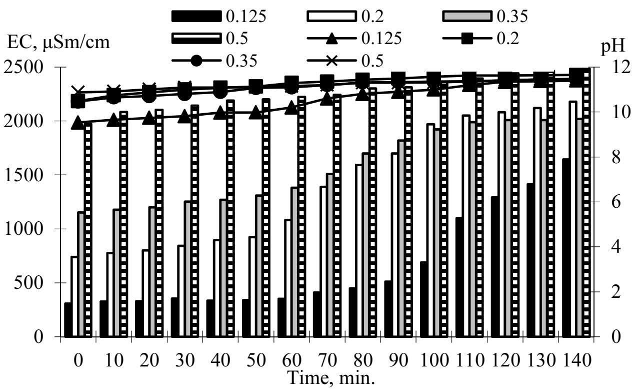

Fig. 2. EC (columns) and pH (lines) changes in the suspensions of binder with different NT content within 2 hours

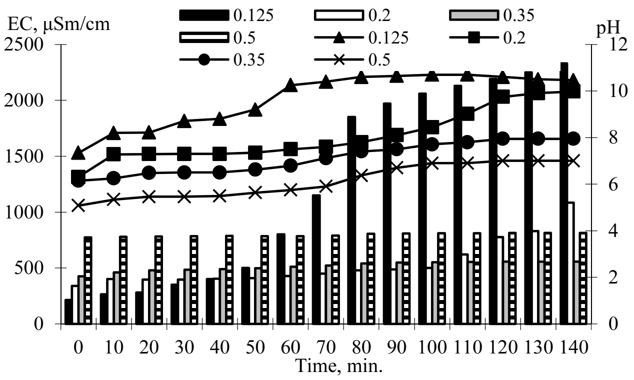

Fig. 3. EC (columns) and pH (lines) changes in the suspensions of binder with different FS content within 2 hours

To assess the effect of deflocculants on the course of MCC-type castable paste hydration, the investigations were carried out as to the EXO reaction of castable pastes with different content of NT and FS. The changes in temperature of paste fixed during the investigation showed (Fig. 4) that the increase in NT content speeds up the EXO reaction. With the minimal NT content, the EXO maximum was fixed after (380 – 400) min. and with maximal content, after (250 – 280) min. The effect of NT content on EXO maximum temperature was observed, 43 °C with minimal NT content, 34 °C with maximal one. That the increase in NT content speeds up the EXO reaction was confirmed by japanese scientists [31] study, where has been tested NT deflocculant amount impact on the Ca ions concentration in the alumina cement suspension. When NT amount increase in suspension, Ca ions concentration increase too. Its accelerate the formation of chelate and speed up Ca ions precipitation.

With the minimal FS content (Fig. 5), the EXO maximum was fixed after 900 min. (longer by 2.5 times than with the same NT content), while with the maximal one after 3500 min. (longer by 12 times than with the analogical NT content). Here the EXO maximum temperature is lower than with the NT deflocculant, and in the case of F1 reaches 40 °C and of F4 – 29 °C. Such a great difference in temperature and duration of hydration shows that each deflocculant influences the process of cement hydration quite differently. This was confirmed by other researchers studies. As pointed in [32], citric acid additives reduces pH values in alumina-cement suspension from 5 to 3 and significantly slow down the cement hidratation process. In the studies [13, 18] was investigated polyacrilate, polymetaphosphate and polycarboxylate ethers with different side chain length as deflocculants in microsilica-containing high cement castables. It was established that the shorter the side chain, the more retarded is the cement hydration. The maximum heat of hydration shifts from 12 h for deflocculant with the shorter side chain to 95 h for deflocculant with the longest side chain. In contrast, the samples prepared with polyacrilate or polymetaphosphate exhibit also a quick hydration of the cement at 12 h.

Table 2. Composition of MCC-type castable, in mass %

AC	SR	Dispersive chamotte	Chamotte aggregate (fr. 0 – 10 mm)	Water above 100 % of dry matter
12	2	16	70	12

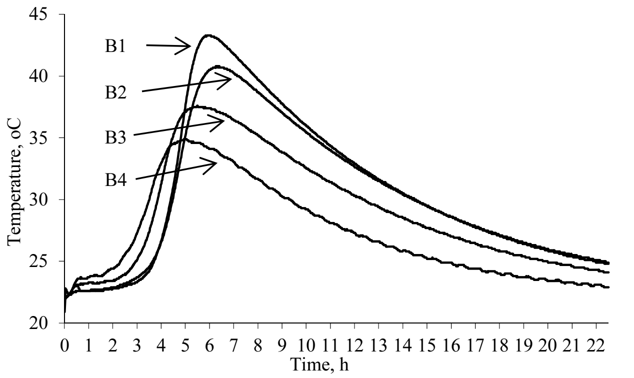

Fig. 4. Kinetics of EXO reaction in MCC-type castable paste considering NT content: B1 – 0.125 %, B2 – 0.2 %, B3 – 0.35 %, B4 – 0.5 %

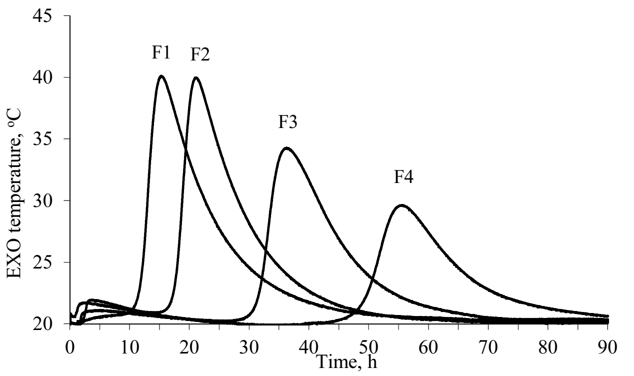

Fig. 5. Kinetics of effect of EXO reaction in MCC-type castable paste considering FS content: F-1 – 0.125 %, F-2 – 0.2 %, F-3 – 0.35 %, F-4 – 0.5 %

Upon comparison of the results of the EXO investigations with the data of suspensions investigation, one can suppose certain relationships (Fig. 6). We can see that the increase of NT makes a lesser effect on both EXO maximum time and pH values than that of FS. Most likely, the acidic properties (lower pH values), the original structure and a low EC of deflocculant FS considerably stop the processes of cement hydration and transit of ions Ca^{2+} into solution, while the alkaline properties of NT and high EC stimulate a more rapid cement hydration.

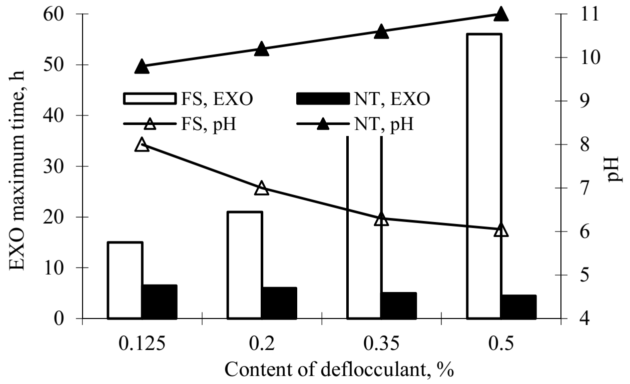

Fig. 6. Relationship between EXO maximum time of MCC-type castable paste and pH values in the suspensions of binder considering the content of NT and FS deflocculants

Applying the method of UWV testing, the investigations of structural changes in castable samples after 3 days hardening were carried out. Upon comparison of data of EXO maximum time and UWV measurements (Fig. 7), we can see that the increase in NT content in the composition of castable influences but little both the UWV values and EXO maximum time. While increasing the FS content in the composition of castable, the hydration is running on longer (the EXO maximum time increases) and UWV decreases in the samples (3300 m/s with minimal FS content and 1300 m/s with maximal FS content), what shows that the speed of formation of structure is decreasing. It can be also said that the UWV values in the castable samples with deflocculant FS are lower than those with NT.

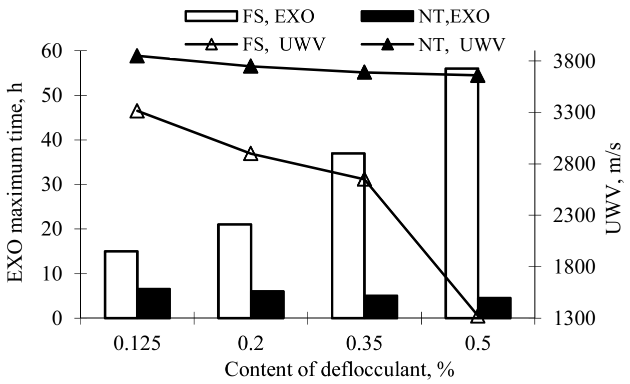

Fig. 7. Relationship between EXO maximum time and UWV values in MCC-type castable paste after 3 days hardening, when considering the content of deflocculants NT and FS in castable samples

By the investigations of the effect of deflocculant content on physical-mechanical properties of a castable after treatment at different temperatures, it was established that the increase in content of NT deflocculant does not result in higher compressive strength (CS) values either after hardening and drying or after burning at temperatures of 800 °C – 1200 °C (Fig. 8). The increase in content of NT only stimulates more the decrease of compressive strength after burning at 1000 °C.

The sintering processes after burning at 1200 °C increase the compressive strength values; however, the pattern of the deflocculant NT effect remains. The further thermal treatment stimulates the castable destruction processes, which are the greater, the higher NT content in the sample; and this fact confirms the data of compressive strength.

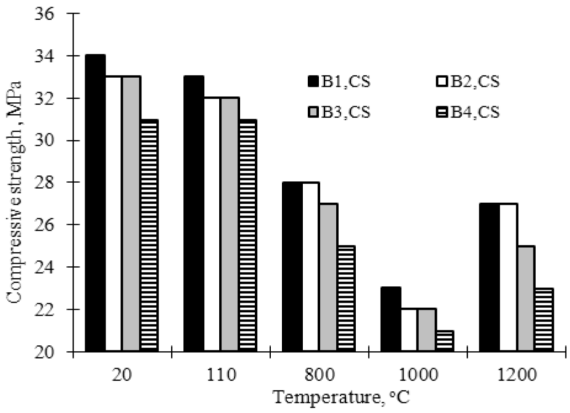

Fig. 8. Changes in compressive strength in NT castable samples treated at different temperatures

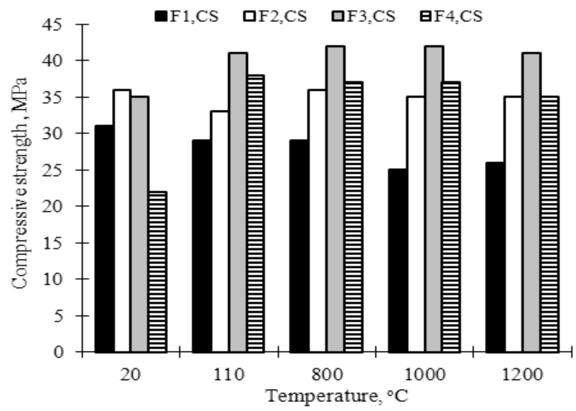

Fig. 9. Changes in compressive strength in the castable samples with FS deflocculant treated at different temperatures

Unlike the NT case, in the castables with the deflocculant FS (Fig. 9) one can observe the optimal content of deflocculant, 0.2 % – 0.35 %. With such a content of deflocculant, the compressive strength after hardening reaches 35 – 36 MPa, and with 0.5 %, it drops down to 22 MPa. This drop can be accounted for an extended period of hydration (Figs. 5 – 7). After drying, the compressive strength in the castables with a higher FS content notably grows up. The effect of different defflocculants influence on the physical-mechanical properties of refractory castable have been studied by [3, 12, 33]. The early compressive strength after 24 h curing for low-cement castable samples with polyacrilate defflocculant was 0 MPa, while for samples with polycarboxylate ether with different side chain length 4, 27 MPa and 32 MPa respectively. It was mentioned that the longer the side chain, the higher the compressive strength of castable samples. After drying compressive strength for samples with polyacrilate defflocculant was

82 MPa, whereas for samples with polycarboxylate ether 111 MPa. After firing at 1500 °C compressive strength for samples with polyacrilate was 198 MPa, for samples with with polycarboxylate ether 298 MPa.

The strength characteristics of the samples with FS burned at 800 °C, 1000 °C and 1200 °C are notably higher (averagely by 15 MPa) than those of samples with analogical NT content; and after burning at 800 °C – 1000 °C, these samples, unlike those of NT, do not loose strength. The performed investigations show that the strength properties of castables, regardless of FS content; are higher within the whole interval of temperatures than those of castable samples with an analogical NT content.

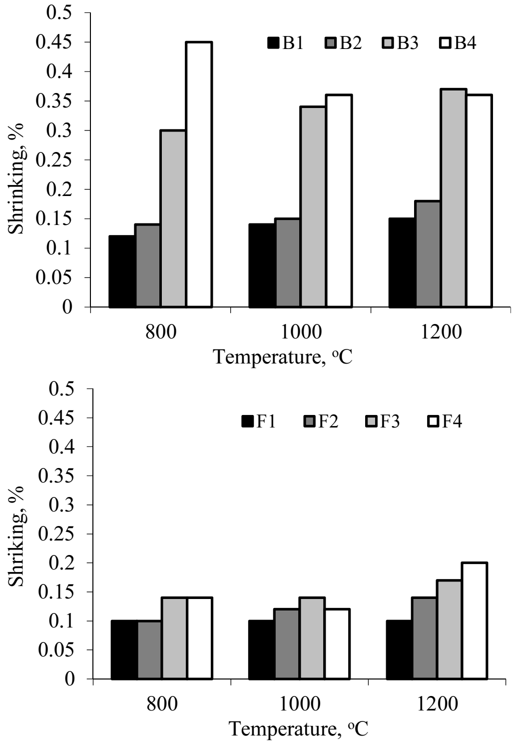

Fig. 10. Shrinking of castables with FS and NT additives after burning at different temperatures

By the investigations of shrinking, it was established that along with increase of NT content in the composition of castables, the shrinking of samples is increasing after burning at 800 °C – 1200 °C (Fig. 10). It is mostly observed at NT content of 0.35 % and 0.5 % in the composition of castables, the shrinking being of 0.3 % – 0.45%. Along with the increase of FS content in a castable, the shrinkage is also growing; however, with the maximal FS content, the reached shrinkage is only of 0.2 %. In order to assess the effect of deflocculants on linear changes of samples during heating, the dilatometric investigations were performed with castable mortar compositions B3 and F3 (the maximal size of filling grain being 1.25 mm) (Fig. 11). As early as the process of dehydration (at 100 °C – 200 °C), the sample F3 expanded more than the sample B3. During further heating at 400 °C -800 °C, this tendency remained. The maximal shrinking values were observed at 1000 °C: in the sample B3 – 0.5 %, in the sample F3 – 0.25 %. Upon burning at maximal temperature of 1200°C, the shrinking of sample B3 reached 0.4 %, that of sample F3 – 0.15 %. The results are rather interesting and show that the deflocculant FS influences the shrinking of samples considerably less than that of NT. Another observation: the samples of castable with NT, distinguished for a higher shrinking, do not have better strength characteristics.

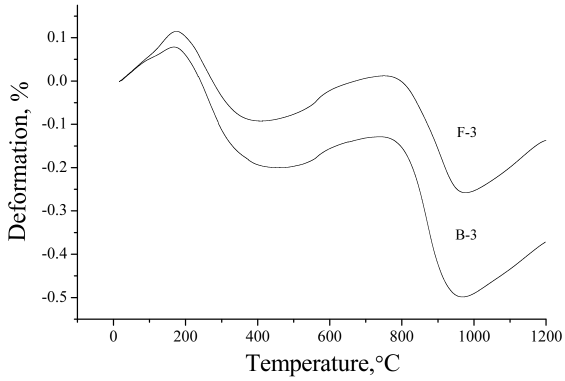

Fig. 11. Effect of heating temperature on linear changes of samples (B3 with deflocculant NT, F3 with deflocculant FS)

4. CONCLUSIONS

1. The investigations of changes in electrical conductivity and pH in the binder suspension considering type and content of deflocculant (from 0.125 % to 0.5 %) showed that the deflocculant NT increases the alkalinity of suspension and ensures the high initial values of electrical conductivity, meanwhile the deflocculant FS decreases pH along with an insignificant increase of electrical conductivity in the suspension of binder. By increasing the FS content in the suspension, one can regulate pH indices and by increasing the content of NT additive – the electrical conductivity.

2. It was established that the increase of deflocculant NT in castable speeds up the processes of cement hydration, and that of deflocculant FS, on the contrary, stops them. In both cases of increasing the content of deflocculant in castable the temperature of EXO reaction is notably decreasing.

3. A relationship was assessed between the content of deflocculant, the pH values in the suspension, the maximal time of EXO reaction in castable mixture and the speed of structure formation (UWV values after 3-days hardening) in a castable. The increase of NT content does not exert any greater influence either on the pH indices in the suspension of binder or on the maximal time of EXO reaction or on the changes in the UWV values in castable. The increase of FS content decreases the pH indices in the suspension of binder; therefore, the time of EXO reaction in castable mixture elongates by 2 – 3 times (from 15 h to 55 h) and the speed of structure formation decreases (UWV decreases by 2.5 times, from 3300 m/s to 1300 m/s).

4. The dilatometric investigations of mortars with the same content (0.35 %) of deflocculants NT and FS showed that the shrinking of sample with deflocculant NT during heating exceeded approximately twice than that of analogical sample with FS.

5. Unlike the case of deflocculant NT, the application of deflocculant FS enables to establish its optimal content in the castable (0.2 % – 0.35 %), which allows receiving castables of highest physical-mechanical properties. By comparing the effect of both deflocculants on strength properties of castables and shrinking after thermal

treatment, it was found that the compressive strength of castable samples with deflocullant FS exceeds almost twice than that of samples with analogical content NT; and shrinkage is almost 3 times lower than that of analogical samples with additive FS.

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