

CONTROL OF THE FLUIDITY OF HIGH-ALUMINA MATRIX SYSTEMS WITH VARIOUS TYPES OF DEFLOCCULATING AGENTS

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Translated from *Novye Ogneupory*, No. 5, pp. 68 – 74, May, 2022.

Original article submitted March 3, 2022.

This article presents the results of a comparative analysis of the dispersing effect of Russian and imported deflocculating agents based on polyphosphates and polycarboxylate ethers (PCEs) on the rheological properties of high-alumina matrix systems and their mineral components. As demonstrated, in terms of deflocculating characteristics, Russian PCEs are not inferior to analogous imported products and can be used to control the rheological behavior of low-cement refractory casting masses.

Keywords: functional matrix mixtures, deflocculating agents, polycarboxylate ethers, fluidity, viscosity, thickening factor, low-cement casting masses

INTRODUCTION

Recently, in Russia and other countries, heat-engineering low-cement refractory casting masses of a new generation, according to the accepted international classification low cement castable, have become widespread [1, 2]. Such masses contain 1.0 – 2.5 wt.% CaO or 4.0 – 9.0% high-alumina cement (HAC).

From the perspective of materials science, such casting masses can be considered heterogeneous binary composite materials in which the matrix system has the property of continuity and consolidates polyfractional filler grains into an integral whole. A distinctive characteristic of the matrix system is the limiting volume concentration achieved because of the dense packing of polydisperse particles of the solid phase [3]. The matrix, which constitutes 25%–35% of the composite material, has a decisive influence on the rheo-technological and operational properties of unshaped refractories. The incorporation of highly dispersed mineral particles into the matrix systems necessitated the development and use of special dispersing deflocculating agents to provide the necessary technological mobility of mixtures with a simultaneous decrease in the amount of water.

Currently, the main deflocculating agents widely used for refractory casting systems are polyphosphates [4 – 7],

polyacrylates [5, 8 – 11], polycarboxylate esters (PCEs) [4, 5, 8 – 11], dispersing aluminum oxides [12, 13], and their combinations. Among polyphosphates, the most common are sodium hexametaphosphate (HMP) $\text{Na}_6[(\text{PO}_3)_6]$ and sodium tripolyphosphate (TPP) $\text{Na}_5\text{P}_3\text{O}_{10}$. Polyphosphate anions are well adsorbed by particles with a negatively charged surface, increasing the charge value, which provides an electrostatic dispersion mechanism. However, orthophosphates are formed at increased temperatures in an alkaline environment, which increases the viscosity of casting masses over time [14].

Sodium polyacrylates represent a group of organic deflocculating agents with the general formula $[-\text{CH}_2-\text{CR}(\text{COONa})-]_n$ and a molecular weight of 1000 – 20,000. The deflocculating effect of polyacrylates depends on the length and nature of the *R* radical [14, 15]. PCEs have a comb structure and comprise a main polycarboxylic chain as well as side polyester chains (Fig. 1). The ionic charge of the main chain is required for the adsorption of polycarboxylate on the surface of mineral particles and the creation of an electrostatic effect, while the side chains of the polymer macromolecule provide a steric dispersion effect. The efficiency of PCE-based dispersants depends on the charge density of the main chain as well as the chemical structure and the length and number of side chains [14].

In the refractory industry, PCEs of various grades have already become widely applied to manufacturing high-quality casting refractory masses for various purposes. However,

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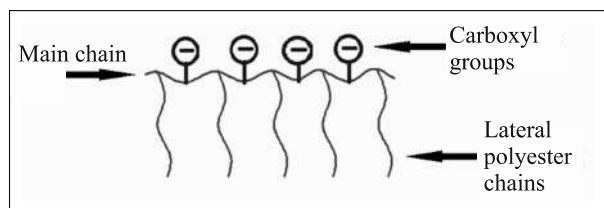


Fig. 1. Scheme of a fragment of a PCE macromolecule.

Russian consumers mainly use imported polycarboxylate deflocculating agents, such as Castament FS20, FS30, FS40, and FS60 made by BASF (Germany) and Peramin® Al 200 and Peramin® Al 300 manufactured by Kerneos (France). At the same time, a sufficient range of polycarboxylate dispersants made in Russia, which are similar to products manufactured in other countries, are offered in Russia [16].

This work aimed to perform a comparative analysis of the efficiency of the dispersing action of additives based on polyphosphates and PCEs toward the rheological properties of high-alumina functional matrix systems and their mineral components.

RESEARCH METHOD

Submicron activated α -aluminum oxide (SMALOKS-A) and modified HAC (MHAC) were used to prepare model functional matrix mixtures (FMMs). SMALOKS-A was obtained by dry grinding from high-alumina raw materials in α -form [17], and MHAC was prepared by grinding high-alumina clinker obtained by roasting a powder mixture of CaCO_3 and Al_2O_3 at 1450°C [18].

The physicochemical characteristics of the FMM initial components were analyzed using analytical scanning electron microscopy (Quanta 200 3D and Quanta 600 FEG, FEI, Netherlands) and x-ray phase analysis (Ultima IV diffractometer, Rigaku, Japan). The chemical composition of the materials was determined through x-ray spectral microanalysis using a Quanta 200 3D electron-ion focused-beam microscope combined with an EDAX energy-dispersive x-ray detector. The particle-size distribution was determined on a Microtrac S3500 particle size analyzer (Microtrac Inc., USA) using laser diffraction. Distilled water (for SMALOKS-A) and ethanol (for MHAC) were used as the dispersion medium. The specific surface area of the powders was determined using the method of low-temperature adsorption and thermal desorption of nitrogen (automated unit TriStar II 3020, Micromeritics, USA).

SMALOKS-A is a non-aggregative white powder well distributed in the aquatic environment. Figure 2a shows the integral curve and the distribution diagram of SMALOKS-A particles. The SMALOKS-A sample has a pronounced bimodal nature of particle distribution. The mode 1 and mode 2 maxima are within the range of $0.3 - 0.4 \mu\text{m}$ and $2.0 - 3.0 \mu\text{m}$, respectively.

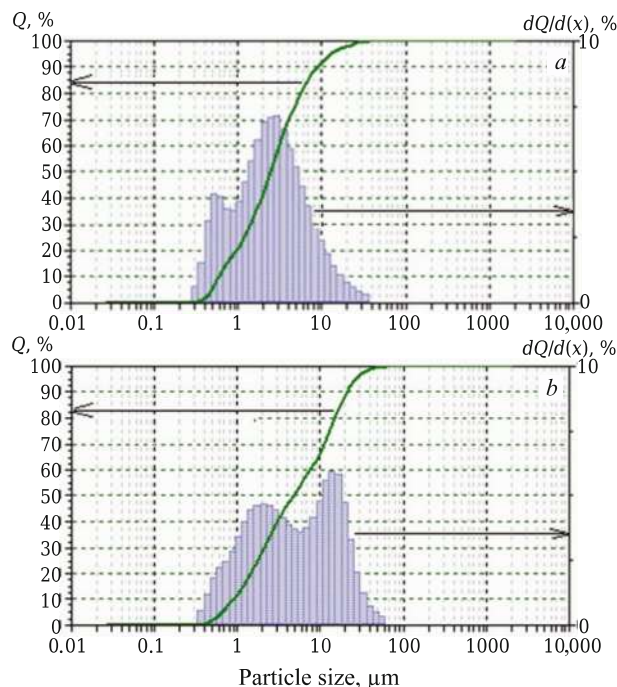


Fig. 2. Particle-size composition of FMM components: a) SMALOKS-A; b) MHAC.

In scanning electron microscopy (SEM) images of finely dispersed submicron activated α -aluminum oxide (Figs. 3a and 3b), two types of particles are clearly differentiated, differing not only in morphology but also in size. Larger crystallites ($3 - 4 \mu\text{m}$) have a three-dimensional prismatic shape. The type 2 particles constitute the submicron α - Al_2O_3 fraction ($<1 \mu\text{m}$) and are represented by isometric plates. The particles are isolated; no tendency to aggregate is noted. The SEM data (Figs. 3a and 3b) agree well with the results for the particle-size distribution of the SMALOKS-A sample (Fig. 2a), confirming the bimodal nature of the particle distribution.

Figure 2b presents the integral curve and a particle distribution diagram for MHAC. The bimodal nature of the particle distribution has been established. The mode 1 maximum corresponds to $2.0 \mu\text{m}$, and the mode 2 maximum is recorded in the range of $10.0 - 11.0 \mu\text{m}$. The morphology of MHAC grains is presented in Figs. 3c and 3d. The structure of a HAC sample is formed by volumetric, well-formed isometric grains varying in size from $1.5 - 5.0 \mu\text{m}$ (Figs. 3c and 3d). The peculiarities of the particle morphology include the presence of sharp-cornered chips formed during grinding. There are also type 2 particles, characterized by a plate-like isometric shape (size up to $1.0 \mu\text{m}$). The characteristics of SMALOKS-A and MHAC are presented in Table 1.

Two types of polyphosphates, namely, HMP of Budal Na 623 (Chemische Fabrik Budenheim KG, Germany) and TPP (Alfakhim Plus, Russia), as well as chemically pure grade citric acid (Reakhim, Russia) were used as deflocculating

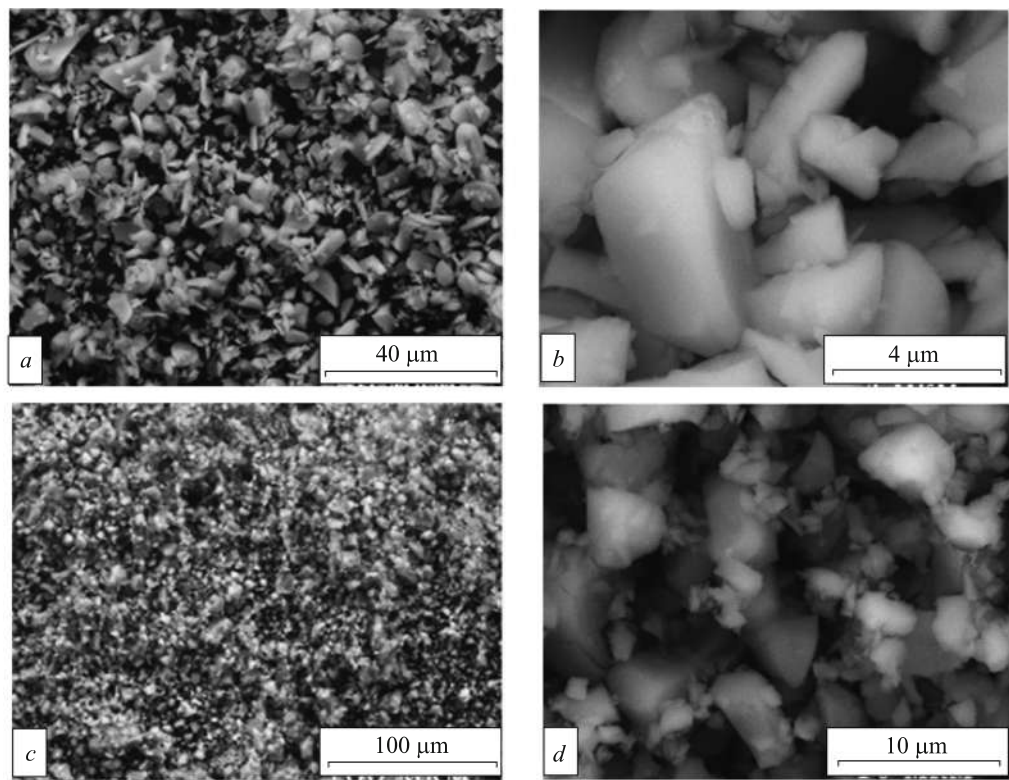


Fig. 3. Scanning electron microscopy images of SMALOKS-A (a, b) and MHAC (c, d) samples.

TABLE 1. Characteristics of SMALOKS-A and MHAC

Indicator	SMALOKS-A	MHAC
<i>Chemical composition, wt.%</i>		
Al ₂ O ₃	99.68	70.50
SiO ₂	0.02	0.28
Fe ₂ O ₃	0.03	0.15
CaO	0.02	27.85
TiO ₂	—	0.12
Na ₂ O	0.12	0.17
<i>Phase composition, wt.%</i>		
α-Al ₂ O ₃	Not less than 98	—
Monoaluminate (CaO·Al ₂ O ₃)	—	65.0
Dialuminate (CaO·2Al ₂ O ₃)	—	35.0
<i>Particle-size composition, μm</i>		
D ₂₀	0.9	1.5
D ₅₀	2.4	5.4
D ₉₀	8.3	22.5
Submicron fraction (≤1 μm), %	22.5	7.5
<i>Textural characteristic:</i>		
S _{sp} (Brunauer – Emmett – Teller surface area), m ² /g	1.4	0.41

agents. PCE-based deflocculating agents were provided for testing by the Sintez OKA Group of Companies. Table 2 presents the physical and chemical parameters of different grades of deflocculating agents. In addition, we used imported deflocculating agents based on PCE Peramin® AL 200 and Peramin® AL 300 (P-AL 200 and P-AL 300) at a ratio of 1:1.

The quantitative composition of the model functional matrix mixture, i.e., the ratio of SMALOKS-A to MHAC, was calculated as follows. The content of the matrix in refractory casting masses is 25% – 35% and that of coarse filler grains is 65% – 75%, i.e., on average, 30% and 70%, respectively [3, 19]. At the same time, the average HAC content in low-cement masses is 6%. Hence, the FMM model, considering the above data, was adopted by us in a mass ratio of SMALOKS-A to MHAC of 80:20.

According to the literature data [6, 7, 11, 14], the polyphosphate content in casting masses averages 0.03 – 0.05%, which corresponds to approximately 0.1% of the matrix mixture mass. In this work, the polyphosphate dosage is taken as 0.1% and 0.25% of the FMM mass. In all cases, citric acid (C₆H₈O₇·H₂O) of chemically pure grade was added to the casting masses in the amount of 0.1%.

According to the literature, the optimal amount of PCE in refractory casting masses is 0.10 – 0.15%, which corresponds to approximately 0.34 wt.% for the matrix mixture [4, 5, 20]. In this study, considering the literature data and

TABLE 2. Physical and Chemical Parameters of PCE

PCE grade (abbreviation)	Content of the main substance, wt. %	Density, g/cm ³ , at 20°C	Viscosity, cPs, at 20°C	pH at 20°C
Sinteflow Mega 50 (M 50)	50 ± 1.0	1.10 ± 0.01	500 ± 100	3.5 ± 1.0
Sinteflow Mega 52 (M 52)	50 ± 1.0	1.10 ± 0.01	450 ± 100	3.5 ± 1.0
Sinteflow Mega 70 (M 70)	50 ± 1.0	1.10 ± 0.01	500 ± 100	3.5 ± 1.0
Sinteflow DUO 60 (DUO 60)	50 ± 1.0	1.10 ± 0.01	500 ± 100	3.5 ± 1.0
Sinteflow Sensitive 50 (S 50)	50 ± 1.0	1.10 ± 0.01	500 ± 100	3.5 ± 1.0
Sinteflow DM 50 (DM 50)	95.0 ± 1.0	350 – 450	Not more than 5	5.0 – 7.0

the recommendations of the Sintez OKA Group, the following PCE concentrations of different grades are adopted:

PCE grade (abbreviation, Table 2)	PCE dosage, %
M 50	0.17 and 0.34
M 52	0.17 and 0.34
M 70	0.17 and 0.34
DUO 60	0.19 and 0.38
S 50	0.21 and 0.42
DM 50	0.17 and 0.34
P-AL 200 + P-AL 300	0.17 and 0.34

Notably, deflocculating agents DM 50 (dehydrated form M 50) and P-AL 200 + P-AL 300 were dry powders, while the remainders were aqueous PCE solutions.

Suspensions of MHAC, SMALOKS-A, and FMMs were prepared as follows. The weighed portions of the powders, calculated considering a suspension volume of 120 – 150 cm³, were placed in a 300 cm³ plastic cup and stirred for 1 min. Next, the required amount of the deflocculating agent solution or powder was added and stirred for 3 min until a homogeneous suspension was obtained. When a dry deflocculating agent was added to the matrix suspension, its predetermined amount was preliminarily triturated with a small part of the FMM powder or its components.

The fluidity of the MHAC, SMALOKS-A, and FMM casting systems was evaluated using an outflow time of 100 mL of the suspension on an Engler viscosity meter with an outlet diameter of 5.4 mm. Before testing, the suspension was stirred with a glass rod, poured into the viscosity meter vessel, and held at rest for 30 sec. Then, the outlet was opened and the time of the outflow of 100 mL of the suspension (τ_0) was recorded using a stopwatch. At least three parallel measurements were performed. Similar tests were performed for the suspension after holding in the viscosity meter for 30 min, and the fluidity 2 (τ_{30}) was determined. Then, the thickening factor was calculated as $TF = \tau_{30}/\tau_0$.

DISCUSSION

As shown above, deflocculating agents do not affect the entire composition of the refractory casting mass but only its finely dispersed components. Therefore, the study of fluidity and the effect of various types of deflocculating agents was conducted in relation to FMMs as well as individual compo-

nents of finely dispersed activated alumina (SMALOKS-A) and MHAC.

In stage 1, the fluidity of MHAC suspensions with a moisture content of 22%, deflocculated with PCE-based dispersing agents, was evaluated. A reference suspension with the same moisture content does not have fluidity. The test results are presented in Fig. 4. The addition of polycarboxylate deflocculating agents of all grades reduces the viscosity of MHAC suspensions to varying degrees. The histogram (Fig. 4) shows that for all dispersants, the viscosity decreases as their concentration increases, with the exception of S 50. For S 50, τ_0 at a dosage of 0.42% is 1.8-fold higher than at a dosage of 0.21%. The best fluidity was achieved by introducing the deflocculating agent M 52 (71 sec) at a dosage of 0.34%. For M 50 and its dehydrated analogue DM 50 at a concentration of 0.17%, the value of τ_0 is almost equal.

Notably, after 30 min, because of the sedimentation of solid particles, a dense precipitate was formed, and the fluidity of the MHAC suspensions was almost impossible to measure. Samples of MHAC with the addition of polyphosphates in combination with citric acid at a given humidity, as well as the reference suspension, do not have fluidity.

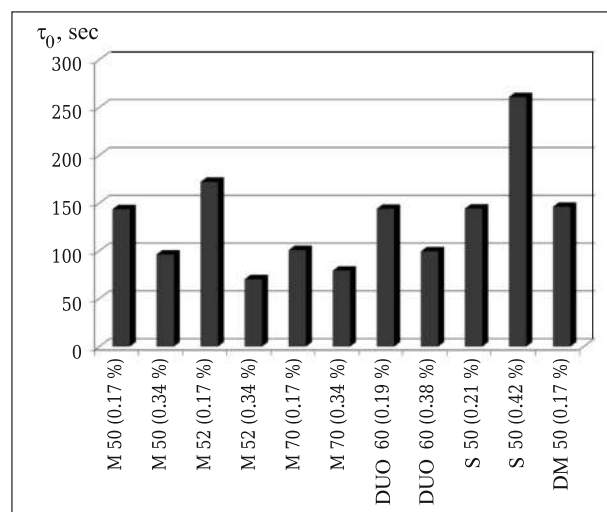


Fig. 4. Fluidity of MHAC suspensions deflocculated with PCE additives.

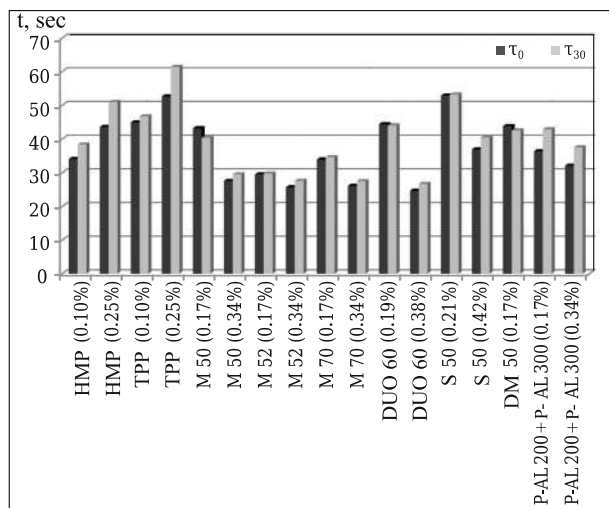


Fig. 5. Fluidity of SMALOKS-A suspensions deflocculated with polyphosphate agents and PCE additives.

Similarly, the influence of deflocculating agents on the fluidity of aqueous suspensions of reactive alumina SMALOKS-A with a moisture content of 20% was assessed (Fig. 5). As a reference, the fluidity of the SMALOKS-A suspension without a deflocculating agent was taken, obtaining τ_0 and τ_{30} values of 211.9 sec and 377.8 sec, respectively (TF = 1.78). The introduction of polyphosphate additives and polycarboxylate deflocculating agents substantially improves the fluidity of SMALOKS-A suspensions (by a factor of 4 – 8.5). Among the polyphosphate deflocculating agents, HMP exhibits the greatest effect. For HMP and TPP, increasing the amount of additives introduced does not have a noticeable effect on fluidity. Thus, for HMP with concentrations of 0.10% and 0.25%, τ_0 is 34 sec and 44 sec, respectively.

In general, PCE additives showed greater efficiency compared to polyphosphates. The maximum and comparable effect of improving fluidity was achieved using PCE grades M 52 (0.17%), M 52 (0.34%), M 70 (0.34%), and DUO 60 (0.38%), namely, τ_0 is 29 sec, 26 sec, 26 sec, and 25 sec, respectively. Increasing the dosage of M 50 and DUO 60 reduces the viscosity of SMALOKS-A suspensions by 1.6- and 1.8-fold, respectively. For other PCE-based deflocculating agents, the amount of additive introduced does not have a substantial effect on fluidity. PCE P-AL 200 + P-AL 300 showed the highest fluidity at a concentration of 0.34% (τ_0 = 32 sec), which is approximately 1.3-fold lower than that of PCE grade DUO 60 (0.38%).

Notably, the TF values of all studied PCE grades are close to unity (Fig. 6). This result indicates complete rheological stabilization of SMALOKS-A suspensions with a moisture content of 20% for 30 min in the range of all studied SCE concentrations.

Next, we studied the effect of all grades of deflocculating agents on the fluidity of matrix suspensions of the composi-

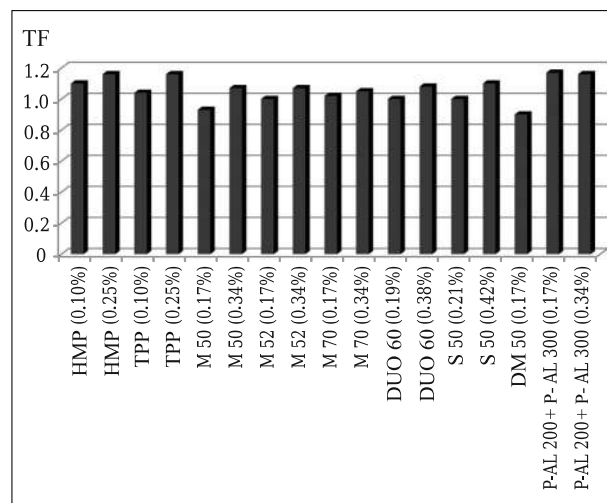


Fig. 6. TF of SMALOKS-A suspensions deflocculated with polyphosphate agents and PCE additives.

tion of SMALOKS-A and MHAC with a moisture content of 20% at a ratio of 80:20. FMM samples prepared without adding dispersants were high-viscosity pastes. Figure 7 presents the deflocculating effect on the fluidity of aqueous matrix suspensions of polyphosphates with a moisture content of 20% and different grades of PCE. As shown, HMP and TPP additives exhibit a thinning potential with respect to matrix suspensions. For HMP, an increase in concentration to 0.25% increases fluidity by 1.5-fold (from 60 sec to 41 sec). The suspension with the addition of TPP in the same concentration range has approximately the same values of τ_0 (53 sec and 52 sec, respectively), and the maximum thickening occurs at a dosage of 0.25% (TF = 3.2, Fig. 8). For HMP at a similar concentration, the TF of the suspensions is also increased (up to TF = 1.2).

The results of study of the dispersing ability of PCE showed a higher deflocculating effect of all additives in relation to matrix suspensions with a moisture content of 20%, where the values of τ_0 vary within the range of 22.2 – 28.8 sec. Notably, the concentration of additives has a considerable effect on the TF (Fig. 8). Thus, at low deflocculating agent concentrations, the TF changes in a narrow range (3.0 – 3.6). An increase in the amount of additives prevents the suspension thickening within 30 min, which is confirmed by a decrease in the TF of the matrix suspensions. The maximum value of the TF is obtained in FMMs with DUO 60 (0.19%) and C 70 (0.17%) additives, and it amounts to 3.59 and 3.59, respectively. An increase in the concentration of these deflocculating agents to 0.34% causes a decrease in the TF to 2.1 (by 1.7-fold). In general, a similar dependence is noted for all PCE grades.

Samples with different concentrations of P-AL 200 + P-AL 300 showed comparable efficiency compared to PCE produced in Russia. The fluidity values of FMMs almost remain constant with increasing deflocculating agent

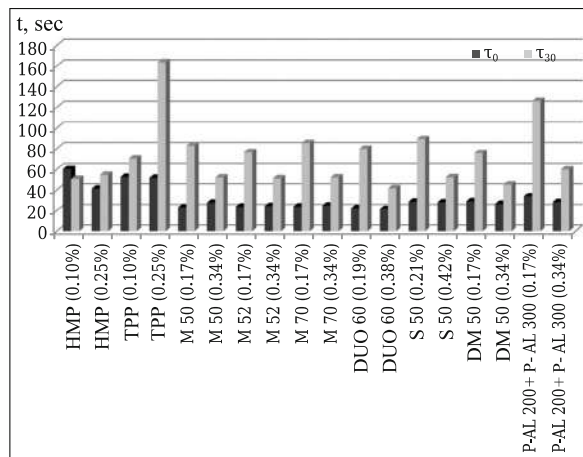


Fig. 7. Fluidity of FMM suspensions deflocculated with polyphosphates and different grades of PCE.

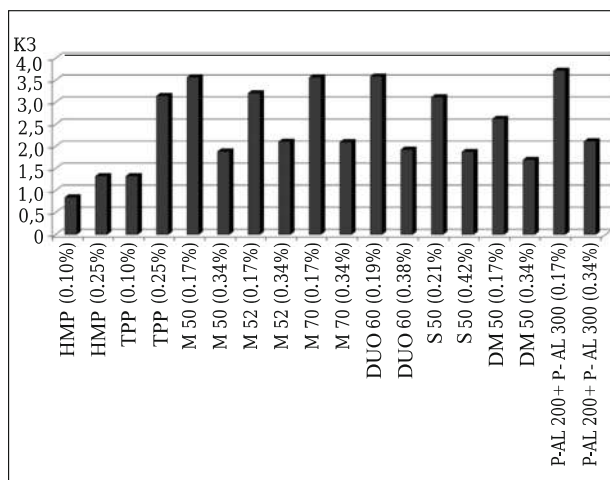


Fig. 8. TF of FMM suspensions deflocculated with polyphosphates and various grades of PCE.

concentration. The τ_0 index for suspensions with the additions of P-AL 200 + P-AL 300 of 0.17% and 0.34% is 25 sec and 29 sec, respectively. The TF increases with the amount of additive.

A comparative analysis of the deflocculating action of polyphosphates and PCE revealed that polycarboxylate dispersants have a stronger dispersing effect on matrix suspensions (by 1.8 – 2-fold). For example, τ_0 for FMS with additions of DUO 60 (0.38%) and HMP (0.25%) is 22 sec and 41 sec, respectively. In general, the best diluting effect was noted with DUO 60, where at concentrations of 0.19% and 0.38%, τ_0 was 22.2 and 21.5 sec, respectively.

In suspensions of MHAC and SMALOKS-A, almost no thickening occurs with the addition of PCE for 30 min. This result is due to the complete stabilization of casting systems in the studied range of PCE concentrations. The thickening

of matrix suspensions is largely associated with the hydration processes of the calcium aluminate cement that interacts with water, resulting in the formation of highly dispersed hydrated phases. This process leads to the coagulation of the hydrodispersions. PCE macromolecules, upon contact with an aqueous medium, dissolve, adsorb on fine mineral particles of SMALOKS-A and MHAC, and block the interaction of cement with water. In this regard, hydrate phase formation and the coagulation process proceed more slowly.

For matrix mixtures at a SMALOKS-A-to-MHAC ratio of 80:20, most of the interfacial surface is accounted for as SMALOKS-A particles. Considering the specific surface area of each component, the contribution of SMALOKS-A to the total surface area in the matrix system is approximately 90%. Therefore, when PCE is dissolved, its molecules are adsorbed to a greater extent on SMALOKS-A particles; the remaining molecules are sorbed on MHAC particles. At high PCE concentrations, deflocculating agent molecules are sufficient to block cement particles. Thus, the hydration processes of MHAC slow down and the thickening decreases during the first 30 min. At low PCE concentrations, the deflocculating agent molecules are no longer sufficient to completely block the cement particles, which intensifies hydration and subsequent coagulation. This latter process explains the increase in the thickening factor of such systems. For matrix suspensions deflocculated with polyphosphates, in which the molecules have a linear structure and only the electrostatic mechanism of dispersion is implemented, the above regularities are not observed.

CONCLUSION

– Additives of all studied commercial grades of deflocculating agents reduce the viscosity of finely dispersed alumina suspensions to varying degrees, while a good dispersion of MHAC suspensions is achieved only with the addition of PCE.

– A higher deflocculating effect of PCE additives has been established in relation to suspensions of matrix mixtures based on SMALOKS-A reactive alumina and HAC. The complex imported deflocculating agent Peramin® AL 200 + Peramin® AL 300 showed comparable efficiency with PCE produced in Russia.

– In MHAC and SMALOKS-A suspensions deflocculated with PCE additives, almost no thickening occurs within 30 min. This result is due to the almost complete aggregative stabilization of casting systems in the studied range of PCE concentrations.

– Increased values of the thickening coefficient of FMM suspensions at PCE concentrations of 0.17 wt.% are due, in our opinion, to the intensive hydration processes of calcium aluminate cement. Thus, highly dispersed hydrated phases are formed, and coagulation of hydrodispersions occurs. With the introduction of 0.34 wt.% PCE, deflocculating agent molecules are sufficient for adsorption on

SMALOKS-A particles and the obstruction of cement particle surfaces. Consequently, the hydration processes of MHAC slow down and thickening decreases during the first 30 min.

The work was performed at Belgorod State National Research University with financial support from the Ministry of Science and Higher Education of the Russian Federation under agreement No. 075-11-2020-038, dated December 14, 2020, on the implementation of a comprehensive project "Creation of an import-substituting production of components for matrix systems and thermal engineering composite materials of a new generation based on them," in accordance with the Decree of the Government of the Russian Federation No. 218 of April 9, 2010.

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