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Investigation of Peculiarities in the Hardening Process of Portland Cements with Active Additives out of Waste

Jadvyga ŽVIRONAITĖ [*], Ina PUNDIENĖ, Valentin ANTONOVIČ, Valdas BALKEVIČIUS

Vilnius Gediminas Technical University, Scientific Institute of Thermal Insulation, Linkmenų 28, LT-08217 Vilnius, Lithuania

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This study deals with the impact of waste fluid cracking catalyst (FCC) and cupola dust (CD) on hardening process of various Portland cements (CEM I 42.5 R (PCR), CEM I 42.5 N (PCN), CEM II/A-S 42.5 N (PCSN)). The plain cement pastes and waste modified pastes (with cements replacement for 10 % of additives), also pastes with the well-known pozzolana additive, microsilica (MS), were investigated. The studies of development of hardening structure by ultrasound wave velocity (UWV) measurement method, of change of mineral composition and physical-mechanical properties were carried out. Impact of additives is subject to both the own properties (chemical, mineral composition and fineness) of additive and of cement. FCC accelerates noticeably the development of initial structure of finer cement (PCR) only. Beyond 24 h development of structure in all FCC modified pastes is going somewhat slower, nevertheless, after 28 days their structure is already more compact than that of plain pastes. The strength properties are changing accordingly. The development of initial structure of CD modified PCR, and especially PCSN, pastes is going faster, PCN - almost does not change. Beyond 24 h compacting of structure of all CD modified pastes was slower. For the early strength of cements, the impact of CD was negligible, after 28 and 90 days the strength of CD modified PCR and PCN pastes was lower than that of plain pastes. In the case of PCSN, the slag is activated by alkali and the strength increased. CD additive, like MS and FCC, decreases the OH^- concentration in the liquid phase of hydrating cement suspensions at the initial period (till 3 h), however further in the course of 28 days it was growing and became higher than that of plain cement suspensions.

Keywords: Portland cement, fluid cracking catalyst, cupola dust, pozzolana, hardening structure, ultrasound wave velocity tests, mechanical tests.

INTRODUCTION

The production of the most used cement binder – Portland cement (further PC) – is related to the extremely high input of fuel and the great emission of CO_2 into environment. It is calculated that the emission of CO_2 by the production of cement makes 5 % – 7 % of total CO_2 emission. Therefore, recently the research into composite cement binders and their concretes, in which a part of PC could be replaced by other active additives, particularly by wastes, has intensified. Especially promising is the waste with pozzolana properties, which can improve the resistance of cement products to alkaline and sulphate corrosion, modify their hardening process, structure and increase the strength, etc. [1 – 5]. Such waste as fly ash, silica fume and granulated blast-furnace slag is widely used in the production of cement binders and concretes and for its investigations a lot of studies are dedicated. The sphere of investigations expanded greatly along with growing amounts of waste and appearance of new sorts of waste.

Recently the most investigated is the use of fluid cracking catalyst (FCC), which is famous for its zeolite structure, for the production of cement binders and concretes [1, 6 – 11]. It is established that in hardening cement mixtures with FCC the content of not combined portlandite (CH) decreases and the amount of cementing phases (calcium hydrosilicates and hydroaluminates) increases, i. e. the well-known pozzolanic reactions take place. This has positive influence on strength and other properties of hardened PC compositions. According to [1, 12] an addition of FCC increases greatly the strength of mortars, due to remarkably better cohesion of aggregates and cement paste, but the impact of FCC on strength of cement paste is insignificant or unfavourable. The better strength of mortars with FCC is indicated in [6, 8, 10] along with the fact that FCC worsens the workability of cement mortar, and that its ability to increase the strength is subject greatly to water/binder ratio, properties of plasticizer, as well as to type of cement. According to [9], FCC suits best for cements of type R where replacement of cement may constitute 10 % – 15 %. With a lesser amount of water, only superplasticizers are essential to obtain proper workability of cement mortars [9, 11]. In the study [13] it is found, that FCC in the mixture of lightweight concrete behaves as an active filler aggregate and increases the compressive strength (~ 4 %). FCC, as a fine additive, also was tested in a refractory castable [14 – 16]. It is found, that FCC accelerates hydration of aluminate cement in the mixture of castable, but decreases its flow and reduces the working time. In many studies it is stressed that only ground FCC of higher fineness is effective [9, 15].

Other waste containing very fine SiO_2 is the waste from the production of rock wool – cupola dust. This waste, witch accumulates in the filters for air out coming from cupola, is very dispersive, with strong sorption properties [17, 18]. In the scientific literature we did not find any data related to potential applications of this waste in cement products. Nevertheless, basing on the researches as to impact of additives of high fineness on cement mixture,

[*]Corresponding author. Tel.: +370-5-2752535; fax.: +370-5-2752629.
E-mail address: *jadvyga.zvironaite@vgtu.lt* (J. Žvironaitė)

we can maintain that very fine CD containing nanoscale size particles will exert influence on hardening processes of cement and perhaps will suit as a filler of high activity or as a pozzolana additive. It is know [19], that both to chemical nature as well as fineness are important with regard to the PC hydration process and strength development. In the investigations [20] of early hydration and structure development of cement system that contain micro and nanoscale pozzolanic additives it is found, that the particles of nanoscale size (colloidal silica) accelerate early hydration reaction by providing large amount of reactive siliceous surface with serves as a site for early C-S-H precipitation. In the work [21] the high amorphous compound was obtained then hardened cement paste with sodium silicate solution ($Na_2O{\cdot}nSiO_2$ nanodispersion) was used.

The purpose of this study is to find out the peculiarities of impact exerted by the fine dispersive waste with pozzolana properties (FCC and CD) on hardening processes of different cements, by fully investigating their impact on formation of initial hardening structure and further its compacting, as well as investigating the changes in mineral composition including those of OH^- concentration in liquid phase and the physical-mechanical properties. Basing on these investigations, a possibility to use CD for cement products will be evaluated and the most suitable cements for further investigations will be selected.

MATERIALS AND TEST METHODS

Cements. Several types of PC with strength class 42.5 according EN 197-1 were used: CEM I R, CEM I N and CEM II/A-S N. Clinker mineral composition was: C_3S – 57.5 %, C_2S – 15.5 %, C_3A – 8.5 %, Ca_4AF – 10 %. Quantity of slag in CEM II/A-S was ~15 %. The codes and fineness characteristics of cements are given in Table 1.

Table 1. Fineness characteristics of cements

Cement		Residue on 90 μm sieve, %	Specific surface, m^2/kg
Type	Code		
CEM I R	PCR	1.2	370
CEM I N	PCN	3.5	345
CEM II/A-S N	PCSN	2.5	350

The following materials were used as additives:

– cupola dust (CD). The X-ray diffraction tests show that cupola dust is dominated by amorphous compounds; it contains as well small amounts of crystalline dolomite, halite and sylvine (Fig. 1). From the index of electric conductivity of suspension, one can judge about not a few soluble compounds contained there. The average size of particle is 24.4 μm, 50 % particles are smaller than 11.2 μm, 3.9 % particles are of smaller than 100 nm.

– fluid cracking catalyst (FCC). This is a mixture of microspheres of different sizes (20 μm – 100 μm), average particles size is ~42 μm. X-ray diffraction analysis of FCC showed that it is Y-zeolite.

– microsilica RW-Füller (MS) produced by RW Silicium GmbH. Granular MS was used, the particles from 10 μm to 40 μm dominated in it (> 50 %), average particle size 18.7 μm.

The chemical composition, pH and electric conductivity of additives are given in Table 2, the particle size distribution – in Fig. 2.

At the beginning, PC with the additives was mixed in dry condition. For mixing of cement paste the planetary mixer according to EN 196-1 was used. The plasticity of paste was measured using a Southard viscometer. Specimens ((40 × 40 × 40) mm cubes) were moulded by vibration. The specimens were cured for 1 day in moulds in the moist room, then in water at temperature of (20 ±1) °C. They were tested after 2, 28 and 90 days.

Table 2. Chemical composition of additives, expressed in mass percentages

	CD	FCC	MS
SiO_2	49.8	50.1	96.1
Al_2O_3	3.4	39.4	0.2
Fe_2O_3	7.3	1.3	0.1
CaO	2.5	0.5	0.3
MgO	11.8	0.5	0.4
SO_3	0.72	2.3	0.4
Na_2O	5.6	0.2	1.2
K_2O	4.6	0.1	0.1
Cl	0.4	–	–
LOI	11.4	5.4	0.4
Conductivity, μS/cm	3100	200	400
pH	9.5	6.3	8.1

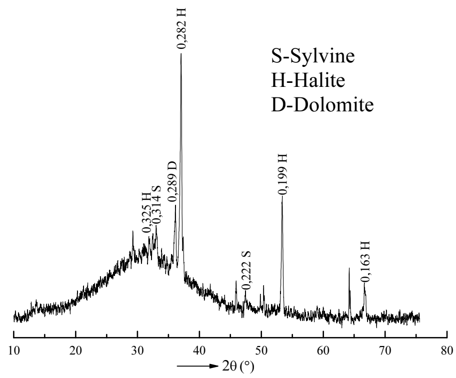


Fig. 1. The X-ray diffraction pattern of CD

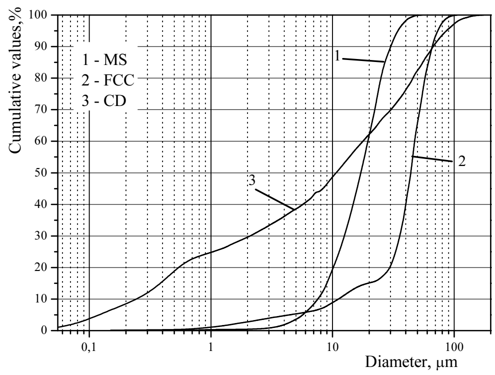


Fig. 2. Particle size distribution of not pre-dispersed additives

The variation of OH^- concentration in liquid phase of hydrating cement (or cement with additives) was investigated using suspensions with water/solid ratio 10 : 1. After a certain period, the suspensions were filtered and the OH^- ion content was determined in filtrates by titrating with 0.1 N hydrochloric acid.

For investigation of hardening structure development in cement pastes by measurement ultrasonic wave velocity (UWV) the Schleibinger Geräte GmbH datalogger with the Pundit 7 ultrasonic pulse indicator was used. Fresh paste (following 10 min from start of stirring) or specimen cube was set between two ultrasonic transducers operating at 10 pulses per second and frequency of 54 kHz. The ultrasonic transition time changes, i. e. ultrasonic sound speed increases according to the setting of cement pastes and development its hardening structure.

The X-ray phase analyses were carried out using a DRON-7 diffractometer (anticathode – cooper, anode voltage 30 kV, anode current 8 mA). The phase composition was identified using reference data from ICDD database. For thermoanalytical studies a STA PT-1600 (Linseis Germany) thermoanalyser was used at heating rate of 10 °C min^{-1}, sample mass of 10 mg. The TG, DSC, curves were registered. pH and electrical conductivity of suspensions (distilled water and FCC, or CD, or MS at ratio of 10 : 1) was measured with a Mettler-Toledo apparatus. Particle size distribution was determined using Cilas 1090 Liquid (France).

RESULTS AND DISCUSSION

Composition and spreadability of cement pastes. The plain and waste modified PC pastes (with PC replacement for 10 %) were investigated. Water/binder ratio (W/B) was constant (0.3). The flow diameters of pastes according Southard viscometer are given in Table 3. As can be seen, the spreadability of all cement pastes is most reduced by CD, then by MS and FCC.

Table 3. Flow diameter of plain and modified cement pastes

Cement code	Flow diameter, mm			
	–	CD	FCC	MS
PCR	80	50	60	55
PCN	100	65	80	70
PCSN	115	70	90	75

X-ray and thermogravimetric tests. We performed the X-ray investigations of CD modified pastes only (Fig. 3), because many studies are dedicated to investigations of mineral composition of hardening cement mixtures with FCC [7, 9, 11]. The content of hydration products (ettringite, CH, CSH) and not yet hydrated C_3S, was evaluated by ratio of the main diffraction peaks of them and of mineral MgO (which practically does not participate in the process of hydration and is distinguished for stability of main diffraction peak intensity). As seen from Fig. 4 (which presents the changes in CH, C_3S and CSH only), in cement PCR of higher fineness, the additive CD practically does not stop the hydration of C_3S, while its hydration in cements PCN and PCSN was going slower in the course of 2 days. The content of detectable by X-ray crystalline CH and CSH in all cement pastes with CD is lower than in plain pastes. The same tendencies of variation in mineral composition are also observed in the pastes, which were hardened for 28 days.

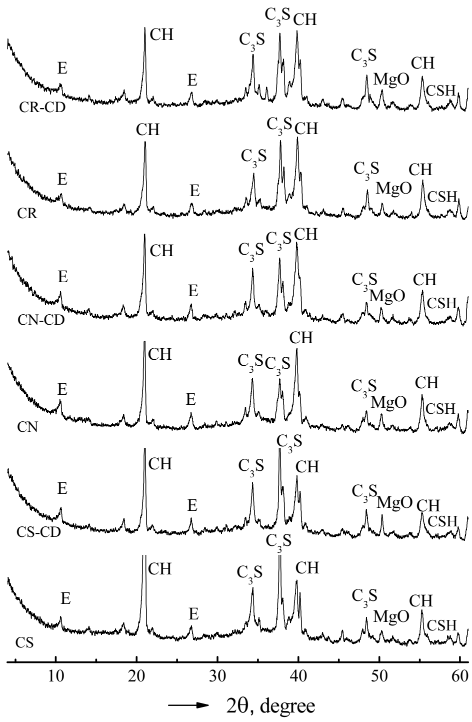


Fig. 3. X-ray diffraction patterns of plain and CD modified cement pastes. Hardening time – 2 days

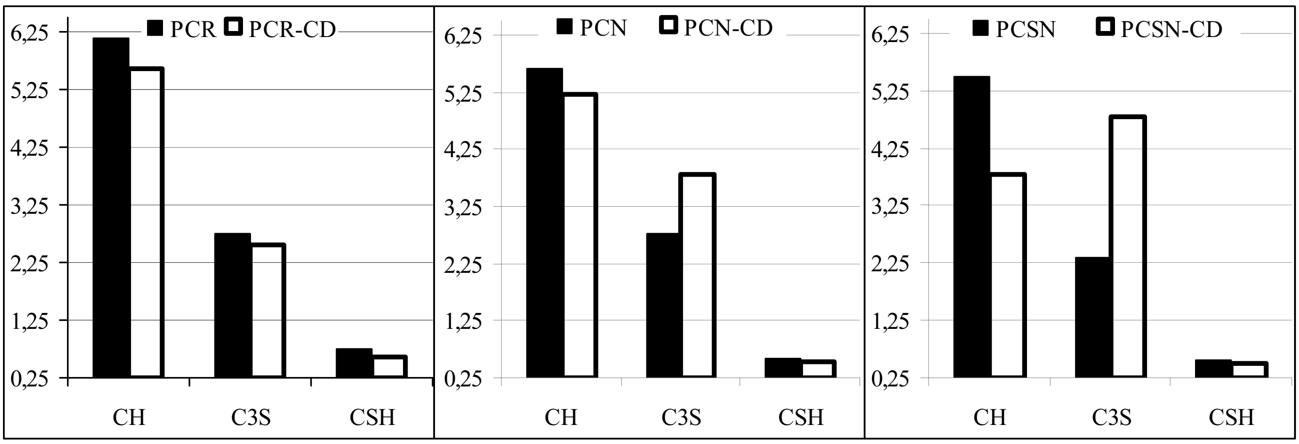


Fig. 4. The comparative intensity of CH, CSH ir C_3S main diffraction peaks in plain and CD modified cement pastes. Hardening time – 2 days

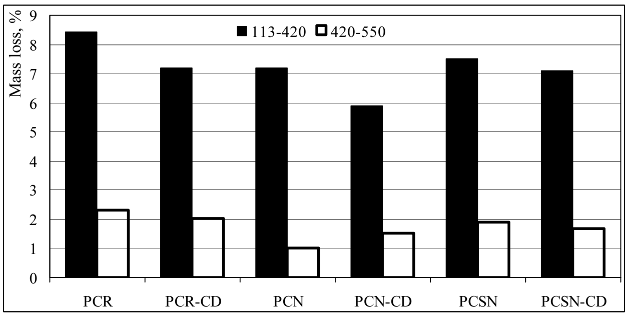
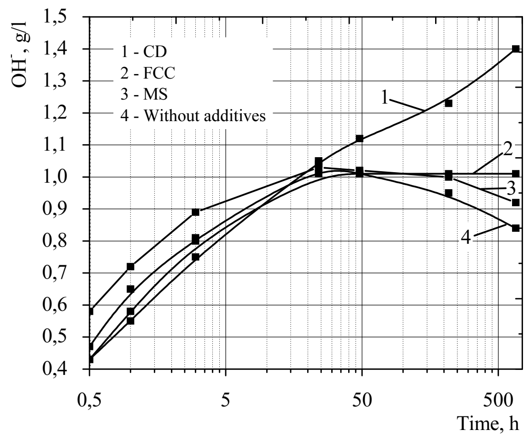
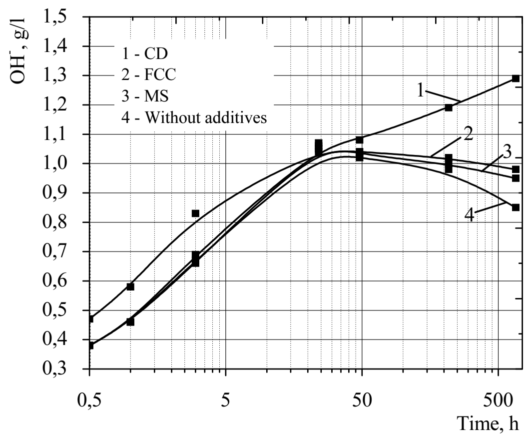


Fig. 5. Mass loss of plain and CD modified cement pastes. Hardening time – 2 days

Investigations of OH^- concentration in liquid phase of cement suspensions. The variation of OH^- concentration in liquid phase of cement suspensions during the initial and middle period of cement hydration reflects the process of CH formation and its transit into liquid phase. Many researchers assume that during this period the pozzolanic reactions are not yet going, they start later [2, 22, 23]. The lower initial CH concentration in cement mixtures with pozzolana are conditioned by the pozzolanic impact blocking C_3S hydration, what is manifested by formation of envelope on surface of C_3S grains [20, 23]. The results of variation of OH^- concentration in the period of 28 days in PCR and PCSN suspensions (Fig. 6) show that the CD impact differs from that of FCC or MS.



a



b

Fig. 6. The variation of OH^- concentration in cements suspensions during 28 days: a – PCR; b – PCSN

At the beginning all additives reduce the OH^- concentration, after 3 h the OH^- concentrations in the suspensions with FCC and MS and plain suspensions are almost the same, in the suspensions with CD the OH^- concentration is still lower. In all suspensions the OH^- concentration was increasing for 24 h, further in the suspensions with MS and FCC and plain suspensions was gradually decreasing. After 28 days in the suspensions with FCC and MS it is lower than in those of plain cement (except for the suspension of PCR with FCC). MS reduces the OH^- concentration more effectively. The impact of FCC may be influenced by impurities contained in it, as well as by the zeolite structure of FCC particles. Meanwhile in the suspensions with CD the OH^- concentration was constantly increasing in the period of 28 days. The chemical properties of CD could have accounted for that, i. e. presence of soluble alkaline impurities, Na and K salts, as well as nanoscale size particles. It was established by the study [20], that nanoscale size particles tend to absorb Ca^{++} ions and to increase the content of OH^- in liquid phase of hardening cement. The impact of additives on cements PCR and PCSN of different mineral composition and different fineness carries a similar character (the lower OH^- concentration at the beginning in PCSN suspensions is due to lower content of clinker in this cement

Ultrasonic wave velocity (UWV) tests. For investigation of hydration and hardening structure development process, the UWV methods are used recently more and more [24, 25]. Researchers in [26] suggested that UWV can be used very effectively to monitor the hydration and formation of microstructure of cement pastes. Authors proposed to description the hydration and hardening process in 3 steps. 1 – when UWV not change – beginning of hydrate formation (induction period, normally (3 – 4) h); 2 – UWV sharply increases – massive precipitation of hydrates with a progressive transition from amorphous to crystallized forms, the mixture stiffens (quickly structure compaction period, untill 24 h); 3 – UWV slowly increases and became stable, then cement skeletons approaches its final stiffness (slowly structure compaction period, follow up 24 h).

The UWV tests of plain and modified cement pastes show that the impact of additives on development of structure is subject both to own properties of the additive and to mineral composition and fineness of cement (PCR and PCN mineral composition is the same, only fineness differs). It should be mentioned that the UWV values of pastes with fine additives are influenced by entrapped air as well. In the studies [27, 28], it was established that though MS accelerates the processes of hydration and setting of aluminate cement pastes, nevertheless, the UWV values are lower due to entrapped air.

As can be seen (Fig. 7) FCC impact is similar to that of MS, only less expressed. After 10 min. since mixing, the UWV values of both pastes are similar (~600 m/s) and lower than those of plain cement pastes (~900 m/s) due to higher content of entrapped air. FCC shortens the induction period in PCR from 3.8 h to 3 h (MS to 2.7 h), practically does not change that of PCN (MS shortens inconsiderably), extends that of PCSN from 3 h to 4.2 h (MS to 3.8 h). The FCC impact on the early stage of development of hardening structure (setting and compacting of structure) is also weaker than that of MS. The impact of CD is other. The initial UWV values of CD modified pastes are practically the same as those of plain pastes, i. e. considerably higher than those of FCC or MS modified pastes despite the fact that its rheological properties and homogeneity are poorer. This shows the sudden interaction between CD and cement particles and the formation of denser microstructure. CD influences the induction period contrarily to FCC or MS: in PCR it does not change, in PCN – prolongs, in PCSN – shortens. Such differing impact of CD could be also conditioned by its chemical properties, especially the presence of soluble salts and alkaline pH, as well as nanoscale size particles. These particles become centers for new formations of hydration and, due to their abundance: many small new formations appear causing a denser and homogenous microstructure [20]. Speaking about the impact of properties of cement, one can see that in the case of finer cement PCR, all additives shorten or do not change the duration of inductive period and accelerate the compacting of structure during the early and middle period of hydration (till 24 h). The existence of slag in cement also changes the character of performance of additives, e.g. in PCSN the impact of MS and FCC is weaker and that of CD is much more effective.

The further UWV investigations of hardened pastes showed that during the third period of formation of structure (following 24 h), the impact of CD and FCC undergoes changes (Fig. 8). The development of structure in all pastes with CD slowed down and their UWV values after 2 days (in PCSN somewhat later) were already lower than those of plain pastes and remained such in the period of 90 days. The UWV values for FCC modified pastes after 2 days were lower; nevertheless, further the development of structure intensified and after 90 days the UWV values of theses pastes were higher than plain pastes.

Investigations of strength and density of cement pastes. The results of investigations as to impact of FCC and CD on compressive strength of cement pastes are given in Fig. 8. Upon evaluation of results, it is necessary to take in consideration the fact that the significantly poorer consistency of pastes with additives could have affected the homogeneity of hardened samples and at the same time the strength indices. This is reflected in the dispersion of compressive strength values: the variation coefficient of these values for plain pastes was within 2 % and 13 %, for pastes with FCC – within 5 % and 20 %, with CD – within 7 and 22 %. The density of pastes with additives was also lower: with CD – (1735 – 1775) kg/m^3, with FCC – (1765 – 1780) kg/m^3, plain pastes – (1800 –1810) kg/m^3. It is known [9, 11], that the optimal impact of fine dispersive additives on cement mixtures is achieved only upon selection of the appropriate W/B and using plasticizers. Therefore, the results of these investigations reflect only the character of development of strength of cement stone when a part of cement is replaced by FCC or CD.

The development of strength in all three cement pastes with FCC was similar: during the early period of hardening (after 2 days) the strength was lower, after 28 days almost the same, while in 3 months already higher than that of plain pastes. The authors who investigated the impact of FCC on strength of cement mortars (where its impact is considerably more effective) established that FCC practically not influences the early strength, but increases it after 28 and especially after 90 days and that the impact of FCC is greatly subject to fineness of cement and that it most suits for cements of type R [9]. In our investigations no higher impact for cement PCR was established, however, this could have been conditioned by poorer rheological properties of CD modified PCR paste.

The impact of CD in pastes of various cements differed. The strength of PCR pastes with CD in all periods of hardening was lower than plain pastes, however, the difference between strengths decreased over time and after 90 days the values of strength were similar. Having in mind the extremely poor rheological properties of PCR paste with CD, one can maintain that the strength of this paste would be higher with appropriate consistency of paste, e. g. using plasticizers. The early strength of PCN paste with CD was higher than plain paste; nevertheless, further the difference decreased and after 90 days they became already lower than plain paste. It is know [29] that the alkaline elements increase the early strength of PC, but decrease the latest strength. As it is seen (Fig. 6), the OH$^-$ concentration in all suspensions with CD after 2 days was

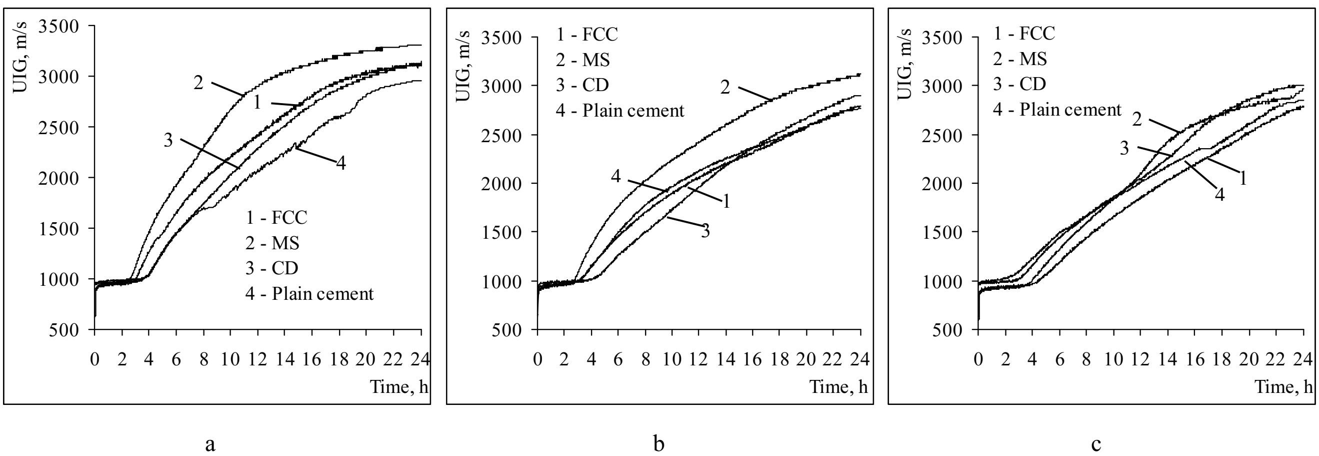


Fig. 7. The variation of UWV in plain and modified cement pastes. a – PCR; b – PCN; c – PCSN

somewhat higher and after 28 days noticeably higher than in the plain or FCC containing suspensions. It should be noted that the strength of PCR and PCN pastes with CD is only slightly less (3 % – 10 %) than that of plain pastes, most likely due to poor rheological properties of pastes. One can suggest that the negative impact of OH^- on latest strength is compensated by presence of very fine amorphous SiO_2 in the CD additive. CD influenced best the strength of PCSN paste: the early strength did not change, while after 28 and 90 days was already higher than that of plain paste. In this case the alkaline impurities contained in CD exert rather a positive than negative influence, since they activate slag component.

It is obvious that the CD impact on strength is subject both to fineness of cement and its mineral composition. The CD additive is best suitable for slag-containing cement and perhaps for finer cements of type R. Basing on the experience related to application of fine dispersive additives in mortars and concretes, as well as on the results of researches in this field, one can predict that thank to selected appropriate W/B and use of plasticizers, the replacement of a part of cement by the CD additive would increase the strength of cement mortars and concretes.

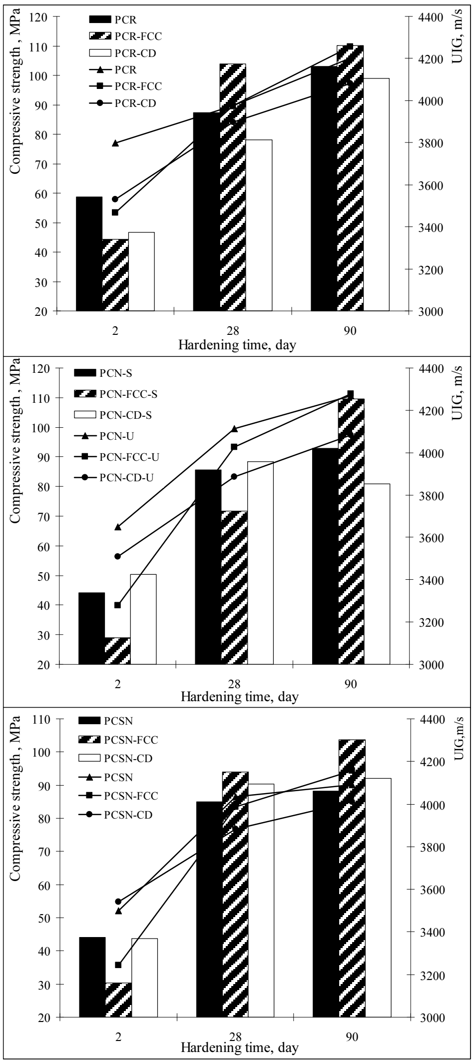


Fig. 8. The compressive strength and UWV values of plain and modified cement pastes

CONCLUSIONS

It is established that the CD additive, through its grading (~50 % of particles of 0.1 μm – 10 μm size, as well as nanoscale size particles) and chemical composition, most reduces the cement paste spreadability among all additives investigated.

The impact of CD and FCC on development of structure in cement pastes differs; furthermore, it is subject to fineness of cement and its mineral composition. FCC accelerates the development of PCR initial structure, exerts no influence on that of PCN and in PCSN slows down it. Further (follow-up 24 h) the structure of all cement pastes with FCC is developing slower for some time, but later this process is accelerating and after 90 days their structure is already more compact than that of plain cement pastes. CD most activates the development of initial structure of PCSN paste. During the further periods of hardening the development of structure in all cement pastes with CD additive is going slower than that of plain pastes. Mostly this process slows down in PCN, least in PCSN pastes.

In the early period of hardening (till 2 days) the CD impact on mineral composition of hardening cements corresponds to that of well-known fine dispersive pozzolana additives: at the beginning the hydration of C_3S is going slower, with less CH and CSH compounds formed, and they are finer, the initial OH^- concentration in liquid phase decreases and in the course of 3 h – 4 h it remains lower. Nevertheless, in the further period of hardening the CD impact already differs from that of MS or FCC. After 2 and 28 days the content of CSH compounds was lower, that of not hydrated C_3S higher (in the case of PCR cement the same content) than in the plain pastes and OH^- concentration in liquid phase does not decreased, but contrarily, is grew further and after 28 days was much higher than in the analogical mixtures with FCC and MS.

The development of strength in pastes with FCC and CD was also different. The early strength of pastes with FCC (after 2 days) was lower, but, further their strength was growing more rapidly and after 90 days was already higher than that of plain pastes. The CD impact on early strength of all cement pastes is not significant; however, further it slows down the development of strength in PCR and especially in PCN pastes, while in PCSN pastes accelerates. It seems likely that such impact was determined by alkaline elements contained in CD (cations K^+ and Na^+), which increase the early strength of PC cements and decrease the later strength. In case of PCSN the alkali activated slag and the strength grew. According this tentative investigations CD additive is most likely suitable for slag containing cements.

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