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Investigating the hydration of deflocculated calcium aluminate cement-based binder with catalyst waste

Valentin Antonovič · Marius Aleknevičius · Jadvyga Kerienė · Ina Pundienė · Rimvydas Stonys

Bretsznajder Special Chapter

Abstract The hydration of the binder, consisting of calcium aluminate cement (CAC) with Al_2O_3 >70% and with or without the additives of the FCC catalyst waste and polycarboxylate deflocculant Castament FS-20, is investigated. The methods of calorimetry, thermal analysis, XRD, electrical conductivity, SEM, as well as ultrasonic wave velocity measuring and bending strength evaluation are used. The results of the investigation show that the FCC catalyst as well as polycarboxylate deflocculant, are active additives, influencing the CAC binder's hydration process. In the structure of the hardened binders, certain amounts of unhydrated CAC minerals, CA, and CA_2, and the hydration products, such as CAH_{10}, C_2AH_8 and the amorphous AH_3, are found. However, in the binder with a deflocculant, there are smaller amounts of CAH_{10} and the amorphous AH_3, though the amount of C_2AH_8 is higher than that in the binder without a deflocculant additive. It has been found that in the case, when the FCC catalyst and deflocculant are simultaneously used, the FCC catalyst produces a positive effect on the formation of the CAC binder's structure, increasing its mechanical strength. The results obtained in this article allow to predict that the FCC catalyst and deflocculant simultaneously used as the additives to the CAC binder, will enable to control the hydration process of the binder.

Keywords Calcium aluminate cement · Hydration · FCC catalyst waste · Polycarboxylate deflocculant

Introduction

The spent catalyst waste from the fluidized catalytic cracking unit (FCC) of the oil refinery can be used in different cementitious materials (binders, mortars, and concretes) as an active additive [1–16].

Calorimetric studies of Portland cement [1, 2] and calcium aluminate cement [3, 4] pastes show that the FCC catalyst remarkably influences the hydration of various cements. It has been found that, in the compositions with Portland cement, the FCC catalyst reacts with calcium hydroxide (a product of cement hydration) as a pozzolanic material [5–7]; in the compositions with calcium aluminate cements the FCC catalyst is an active additive [8].

The authors, investigating Portland cement—FCC catalyst mixtures, noticed that, due to the pozzolanic reaction of the FCC catalyst additive, the compressive strength of the material increased [2, 5, 9, 10]. However, in some applications, the pozzolanic activity of the FCC catalyst was not sufficient [11]. Dweck et al. [12] found that, in some cases, the compressive strength of the cement stone containing the FCC catalyst was lower than that of the material made only of cement and water. The authors noticed that some elements in the spent catalyst composition, such as nickel and vanadium, might have limited its pozzolanic activity. The authors concluded that when cement was partially substituted with the FCC catalyst, the mechanical property of the modified cement stone had to be determined for each case because it did not necessarily increase even when the catalyst demonstrated pozzolanic activity.

Zornoza et al. noticed [13] that larger amounts of waste in Portland cement compositions required larger water–cement

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V. Antonovič (✉) · M. Aleknevičius · I. Pundienė · R. Stonys
Scientific Institute of Thermal Insulation, Vilniaus Gediminas Technical University, Linkmenų str. 28, 08217 Vilnius, Lithuania
e-mail: valentin@centras.lt

J. Kerienė
Department of Chemistry and Bioengineering, Vilniaus Gediminas Technical University, Saulėtekio al. 11, 10223 Vilnius, Lithuania

ratio to assure good workability of cement paste. With a smaller amount of water, only super plasticizers (deflocculants) can help to obtain proper workability of the cement paste. In Portland cement–FCC catalyst compositions having plasticizers a positive effect of the catalyst additive on the strength properties of the material can be observed in addition to water content decrease [2, 14]. The mechanical characteristics of a cementitious material with the FCC catalyst additive can be improved, increasing the FCC catalyst pozzolanic activity by its grinding [9, 15] or adding the particular chemical compounds, such as $CaCl_2$ and Na_2SO_4 [11].

Unlike Portland cement [17–19], pozzolanic materials, combined with calcium aluminate cement (CAC), do not undergo a pozzolanic reaction. This is because free calcium hydroxide is not produced during the hydration of CAC [20]. In work [8], the influence of the FCC catalyst on the hydration of CAC, having about 40% of Al_2O_3 (CAC40) and 70% of Al_2O_3 (CAC70), was studied. Based on the obtained results, it is concluded that the FCC catalyst influences the hydration of CAC depending on the type of cement, as well as the quantity of the introduced waste. It has been found that the influence is more effective in the case of using CAC40 than CAC70. The XRD results confirmed the reactivity of the FCC catalyst. The new aluminum silicate hydrate of the C-A-S-H type, described as the strätlingite C_2ASH_8, was identified in the presence of the catalyst. The study of the compressive strength of the CAC40 cement mortars containing the FCC catalyst indicated that the compressive strength after the period of 6 h considerably decreased compared to that of a composition without the FCC catalyst. However, after a long-term period (150 days), the strength was increased, which allowed the authors of the work to suppose that the increase in the compressive strength was caused by the formation of strätlingite. The authors of the work [8] also noticed that, in the applications of the FCC catalyst additive, the optimum amount of this material and some additional substances, e.g., a superplasticizer (deflocculant), had to be added.

The added deflocculants, acting as dispersing agents [21], can be used in the CAC-FCC catalyst compositions for improving the flow characteristics, as well as physical–mechanical properties of the material, similar to the case of using Portland cement compositions. The recent investigations of the researchers from BASF Construction Chemicals show [22–24] that polyelectrolytes of the polycarboxylate ether type are very efficient dispersants for CAC-based products because they provide super workability to the material in the fresh state and excellent physical properties in the hardened state. The authors of [22] have found that, depending on the chemical structure, polycarboxylate ethers influence the hydration process of CAC. They can either retard or accelerate cement hydration, which can result either in the prolonged workability of the material or in more rapid strength development.

Some efforts were made to use the FCC catalyst in the refractory concrete produced on the basis of CAC40 with the additive of the Castament FS-40 type polycarboxylate ether deflocculant [25]. In this way, a positive effect of the waste catalyst on the strength of concretes could manifest itself. The FCC catalyst (in the amount of 2 and 5%), combined with microsilica and reactive alumina and the Castament FS-20 type polycarboxylate ether deflocculant increased the compressive strength of the refractory concrete up to 25% [26]. These recent investigations clearly show an accelerating effect of the FCC catalyst on CAC40 hydration and a positive effect on the compressive strength of concretes.

For producing refractory concretes, CAC70 is also widely used. Using the FCC catalyst waste in the refractory concrete produced on the basis of CAC70, it is important to investigate the process of hydration of the CAC70 based binder with the additives of the FCC catalyst and the deflocculant and, based on the results obtained, to clarify the possibility of getting a binder with the required properties. The solution of these problems is the aim of the present study.

Experimental

Materials

Testing was performed with calcium aluminate cement Gorkal 70 produced at the enterprise “Gorka” in Trzebinia (Poland). The content of Al_2O_3 in cement was $\geq$70%, the main mineral phases were as follows: CA ($CaO \cdot Al_2O_3$) and CA_2 ($CaO \cdot 2Al_2O_3$), bulk density of 1,100 kg/m^3, and the specific surface by Blaine method of 454 m^2/kg. The chemical composition (in mass%) of the FCC catalyst waste was as follows: Al_2O_3—39.4, SiO_2—50.1, Fe_2O_3—1.3, SO_x—2.3, CaO—0.5, MgO—0.49, Na_2O—0.2, K_2O—0.07, Mn_2O_3—0.06 and the ignition loss—5.4% [25]. Polycarboxylate deflocculant Castament FS-20 is a commercial product of the company BASF Construction Chemicals, Germany.

Cement and FCC catalyst pastes were prepared using the distilled water.

The binder’s compositions with a deflocculant (BD-type) and without it (B-type) were investigated (Table 1). In all compositions, the W/C* ratio was 0.35 (*cement + additives).

Methods

The amount of heat released during the binder’s hydration and the heat release rate were measured by the calorimeter TONICAL III (Toni Technik GmbH). The mixtures (35 g

Table 1 Dry composition of binders in mass%

Component	Composition							
	B-type binder				BD-type binder			
	B1	B2	B3	B4	BD-1	BD-2	BD-3	BD-4
CAC	100	95	90	85	100	95	90	85
FCC catalyst	0	5	10	15	0	5	10	15
Deflocculant[a]	0	0	0	0	0.2	0.2	0.2	0.2

[a] Over 100% of cement and catalyst mixture

of water or 35.2 g of water with the dissolved deflocculant and 100 g of a solid substance) were studied for 48 h at the operating temperature of 20 °C, and the heat evolution curves were registered. Then, the samples hydrated in the calorimeter were investigated by XRD and by a thermal analyzer. The XRD analysis was performed, using the diffractometer DRON-7 (with a copper anticathode, a nickel filter, anode voltage—30 kV, anode current—12 mA and goniometer apertures—0.5, 1.0 and 1.5 mm). For decoding the peaks, the ICDD database was used. The TG and DTG curves were registered by a thermal analytical instrument Linseis STA PT-1600 up to 1,000 °C (with the temperature rise rate 10 °C/min, air as the heating environment for samples in the furnace, the weight of the samples—75 mg).

For investigating the electrical conductivity of the suspensions of the binders in water, the device MPC 227 of the company METTLER TOLEDO (the electrode InLab 730 with the measurement range of 0–1,000 μS/cm) was used. The solid-water ratio of 1:5 was constant.

For SEM, ultrasonic and mechanical investigation, the paste of binders was prepared in a special mixer of forced mixing of the Hobart type with the capacity of 5 l. First, dry components of the mixture were mixed for 2 min, then, the water, making 3/4 of the total water content, was poured into the dry mixture and mixed for 2 min (56 rpm), and, finally, the remaining water was poured and mixed further for 3 min.

The prisms (6 pieces of 40 × 40 × 160 mm) manufactured under slight vibration (5 s) were kept for 48 h in wet conditions at the temperature of 20 °C. Then, the ultrasonic and SEM tests were performed and the bending strength of the hardened binders was investigated.

The ultrasonic wave velocity (UWV) was measured by the device Pundit 7 with the frequency of 54 kHz, using two standard cylindrical heads of the UWV device. The measuring surface of the heads was pressed against the binder's specimens at two strictly opposite points. Vaseline was used to insure a good contact. The velocity of the ultrasonic wave (V in m/s) was calculated by the following formula: $V = S/t \times 10^6$ (S is distance in meters; t is time in microseconds). The bending strength of the binders (5 prism pieces) was determined, using the compressive test machine Tinus Olsen.

The microstructure of the hardened binder was investigated by SEM JSM-7600F (JEOL).

Results and discussion

Calorimetric and electrical conductivity tests

The hydration of CAC includes three stages. After the contact with water, the cement grains dissolve and release Ca^{2+} and $[Al(OH)_4]^-$ ions into the solution, which quickly becomes saturated. Then, the incubation period begins, when the nuclei are forming. At the third stage, germs are built up and massive precipitation of the hydrates occurs [27].

These three stages can be clearly observed in the curves showing heat release rate during the hydration of the binders in the calorimetric tests. In Fig. 1, the heat release rate curve characteristic of all binders' compositions is shown. During the first stage of cement hydration, the first small maximum of heat release rate, developing due to the dissolution of anhydrous cement grains and wetting of

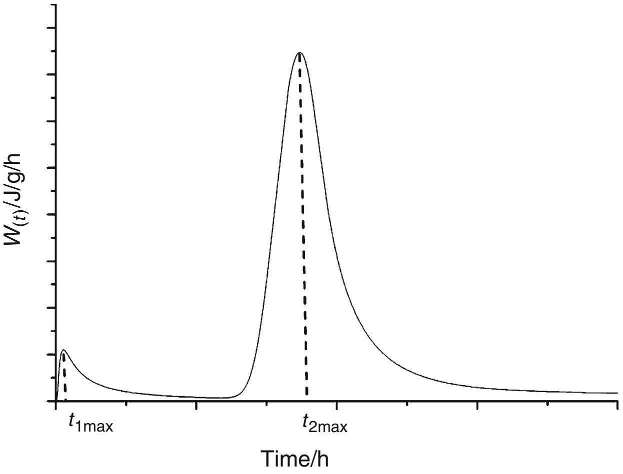


Fig. 1 Heat release rate curve characteristic of all binders: t_{1max} and t_{2max} denote the time of heat release rate maximums

cement and catalyst particles, can be observed. At the incubation stage, the heat release rate does not change. The second considerable maximum observed in the curve is related to the crystallization of the hydration products.

The analysis of the first maximums of the heat release rate (Fig. 1, $t_{1\max}$, first hydration stage) in the binders without a deflocculant (B-type binders) and those with a deflocculant (BD-type binders) shows (Fig. 2a) that the addition of the FCC catalyst to the B-type binders prolongs the time of the first heat release maximum ($t_{1\max}$) very slightly, while the FCC catalyst added to the BD-type binders significantly prolongs the time of the first heat release maximum. The results presented in Fig. 2a also show that the time of the first heat release maximum is more prolonged due to the deflocculant's addition in the case of the BD-type binder.

As mentioned above, at the first hydration stage, the release of ions into the solution occurs. The results of the electrical conductivity test (Fig. 2b) presents a picture of ions' concentration in the solution and gives the essential information on the stage of dissolution in the hydration reaction between cement and water.

Figure 2b presents the times ($t_{c\max}$), after which the maximum values of electrical conductivity in the binder's suspensions are reached. These times correspond to the times of cement hydration in the respective suspensions, after which the balance of the released ions is reached. Comparing the values provided in Fig. 2, we can see that the times of maximum electrical conductivity in the suspensions of the binders (Fig. 2b) are very close to the times of heat release rate maximums in the paste of the respective binders (Fig. 2a). Thus, the results of the above tests show that the deflocculant Castament FS-20 influences the kinetics of the hydration process by slowing down the dissolution of anhydrous cement particles. According to [27], the retarding mechanism consists of blocking dissolution of the cement particles by deflocculant adsorption on the cement grain surface and/or its combination with calcium ions. The retarding effect caused by the deflocculant is greater when the additives of the FCC catalyst are used. Probably, this can be accounted for by the release of ions from the FCC catalyst particles and its influence on cement hydration.

The FCC catalyst, like other zeolite materials, is a good absorber. After mixing the FCC catalyst with water, a noticeable quantity of heat is released due to absorption (Fig. 3a, Curve 1). The addition of the deflocculant to water used for mixing causes an increase in the heat release rate value (Fig. 3a, Curve 2).

In all cases of the investigated binders, the amount of heat, released during absorption, is summed up with the heat, released during the dissolution of the anhydrous cement grains and wetting of cement particles. It considerably affects the maximum value of the heat release rate in binders, i.e., the higher the content of the FCC catalyst, the greater the heat release rate reached (Fig. 3b). It should be

Fig. 2 The time of the first maximum ($t_{1\max}$) of the heat release rate in binders (**a**) and the time ($t_{c\max}$), when the maximum electrical conductivity is reached in the binder's suspensions (**b**)

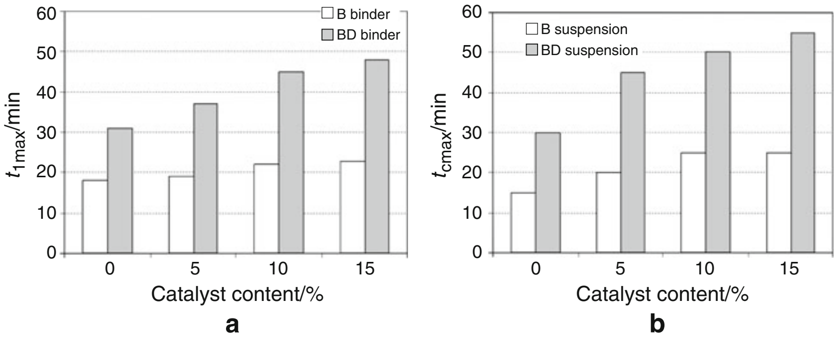
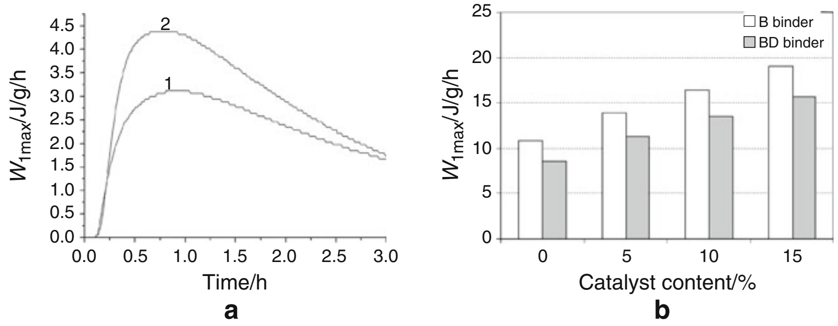


Fig. 3 Heat release rate in FCC catalyst and water mixture (*1* without and *2* with a deflocculant) (**a**) and the maximum value of heat release rate at the first stage of binders' hydration (**b**)



noted that, regardless of the fact that the heat release rate was higher in mixing the FCC catalyst with the deflocculant-containing water than when it was mixed with pure water (Fig. 3a), a decrease in the heat release rate in cement binders with deflocculants can be observed, compared to that found in cement binders without the deflocculant (Fig. 3b).

The analysis of the time of the second maximum (Fig. 1, $t_{2\max}$, third hydration stage), show that the FCC catalyst accelerates the third hydration stage (more rapid crystallization of hydrates can be observed), and these results (Fig. 4, Curve 1) correlate with the results presented in [3, 4], confirming in the hypothesis made in [3, 8] that the FCC catalyst grains act as nucleation centers.

Polycarboxylate deflocculant Castament FS-20 slows down the process of hydration by 1.5–3 h, depending on the content of the FCC catalyst, while this effect manifests itself at all phases of hydration (Figs. 2, 4). This shows that it is possible to regulate the hydration process of binder by selecting a proper amount of these two additives.

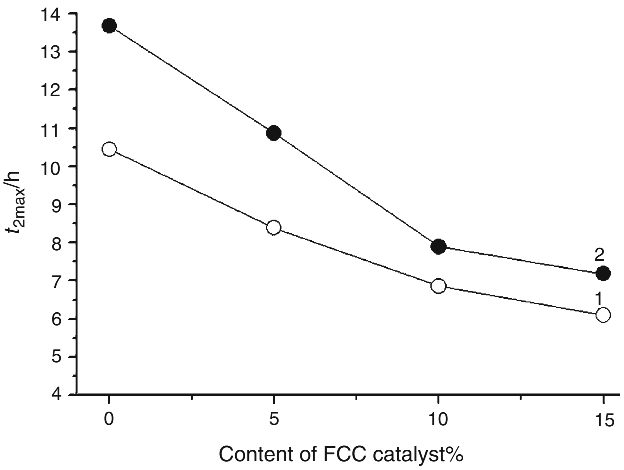


Fig. 4 Maximum value of heat release rate at the third stage of binders' hydration: *1* without and *2* with a deflocculant

XRD and thermal analysis

The XRD results of cement binders provided in Fig. 5 show that the structure of the binder B1 includes the remaining unreacted cement minerals (CA, CA_2) and that the hydrates CAH_{10} and C_2AH_8 have formed. The same hydration products were identified in the binders with a catalyst additive or a deflocculant additive.

Analyzing peak intensities of the main hydrates CAH_{10} and C_2AH_8 in the compositions with the same concentration of cement and FCC catalyst, we can see (Fig. 6) that in the binders with a deflocculant, a smaller amount of the hydrate CAH_{10} and a larger amount of the hydrate C_2AH_8 have formed compared to the compositions without a deflocculant.

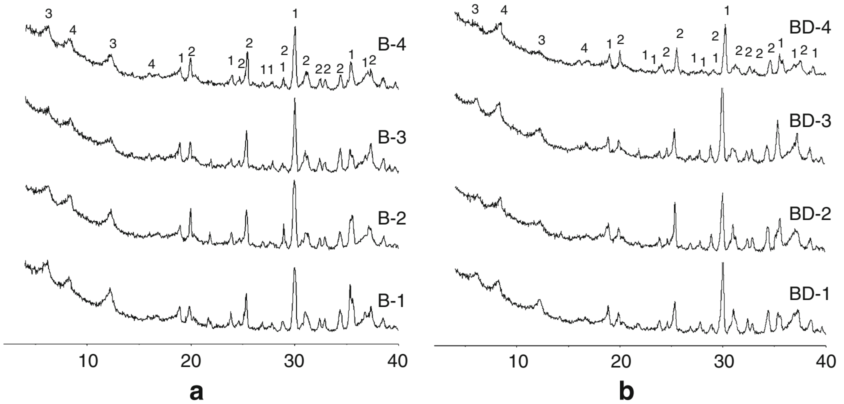
The hydrated phases of the binder's hardening were estimated by analyzing the curves TG (Fig. 7) and DTG (Fig. 8), registered in the thermographic tests of the binders.

During the burning, free and hydrate-linked water, as well as organic admixtures contained in the catalyst, are removed. It was established that the mass loss after the burning of the binders at the temperature of 1,000 °C was the smallest (about 25.1%) in the composition B1 without any FCC catalyst or deflocculant and in the composition BD-1 (about 22.2%) without the FCC catalyst additive, but having the deflocculant additive.

In the compositions with the FCC catalyst, more water remains in the hardened binder, compared to the compositions without FCC catalyst, probably due to water retention in the catalyst (zeolite) particles. Therefore, after burning, the mass loss in the compositions containing the FCC catalyst is higher, reaching 25.2–28.0%.

The water from the cement stone is removed in stages, during which the maximums can be observed in the DTG curve (Fig. 8): 107–117, 180–183, and 271–278 °C. As shown in [28], within the temperatures interval of 100–130 °C, CAH_{10} is dehydrating, while within the intervals of 170–200 and 210–300 °C C_2AH_8 and the amorphous

Fig. 5 XRD patterns of binders after 48 h hardening: **a** without and **b** with deflocculant (1-CA; 2-CA_2; 3-CAH_{10}; 4-C_2AH_8)



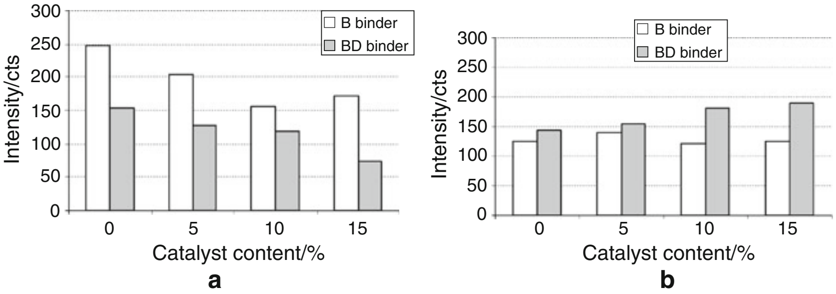


Fig. 6 XRD results of the hydrated binder: **a** intensity of CAH_{10} the main peak ($2\theta = 6.2632$), **b** intensity of C_2AH_8 the main peak ($2\theta = 8.2564$)

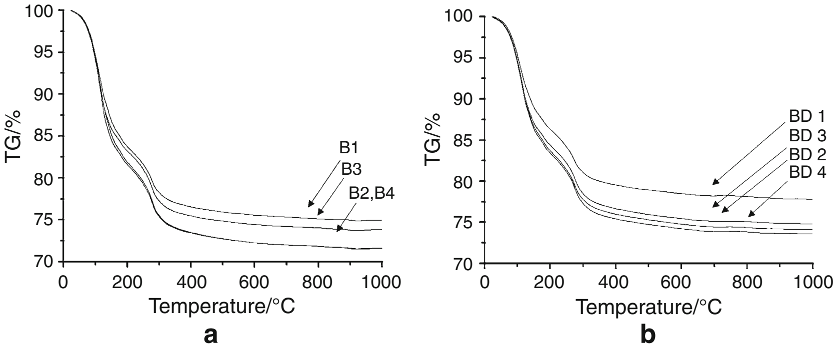


Fig. 7 TG curves for binders: **a** without and **b** with a deflocculant

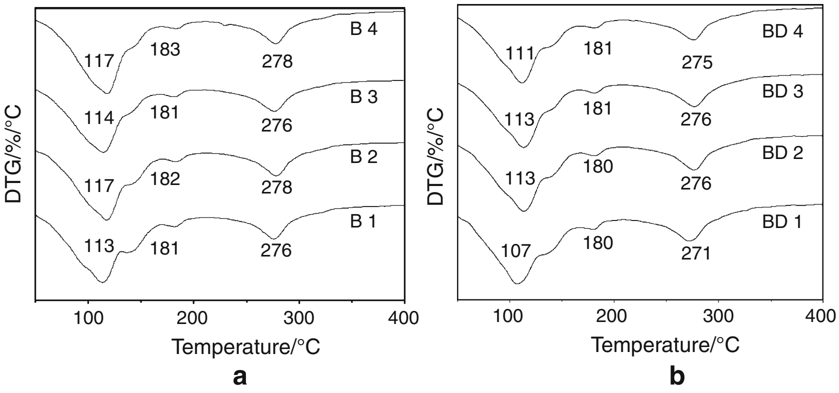


Fig. 8 DTG curves for binders: **a** without and **b** with a deflocculant

AH_3 are dehydrating accordingly. One can observe (Fig. 8) that the temperature of the first and second maximums slightly increases (by 1–4 °C) with the increase of the amount of the FCC catalyst in the compositions. In the compositions with a deflocculant, the temperatures of the maximum are somewhat lower than those in the binder without a deflocculant (increasing by 1–6 °C). This means that the FCC catalyst additive and the deflocculant additive produce a certain effect on the ratio of the formed hydrates CAH_{10} and C_2AH_8. The third maximum corresponds to dehydration of the amorphous AH_3. In this case, a slight decrease in the intensity of the maximum can be observed in the compositions with a deflocculant, compared to the compositions without this additive. The XRD analysis results show (Fig. 6) that in the binders with the deflocculant, a smaller amount of hydrate CAH_{10} and a larger amount of hydrate C_2AH_8 have formed, compared to the compositions without the deflocculant. This means that with the addition of the deflocculant the relation between the contents of CAH_{10} and C_2AH_8 changes. A slight decrease of the amount of the amorphous AH_3, following the addition of the deflocculant (Fig. 8), is related to this change.

Mechanical tests and the analysis of microstructure

The results of tests on the bending strength in the samples of binder after hardening of 48 h (Fig. 9a) show that the bending strength of the B-type binder with the FCC catalyst (Fig. 9a, Curve 1) is lower than that of the control pure cement binder B1. In the BD-type binder with a deflocculant (Fig. 9a, Curve 2) the bending strength is higher, when the content of the catalyst additive is higher, i.e., it increases from 8.5 MPa (without the catalyst additive) to 13.6 MPa (with 15% catalyst additive).

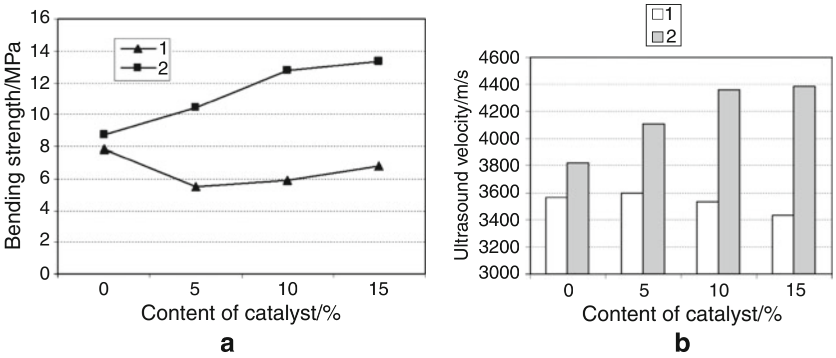


Fig. 9 The binder's bending strength after 48 h hardening (**a**) and ultrasound wave velocity in binders (**b**): *1* without and *2* with a deflocculant

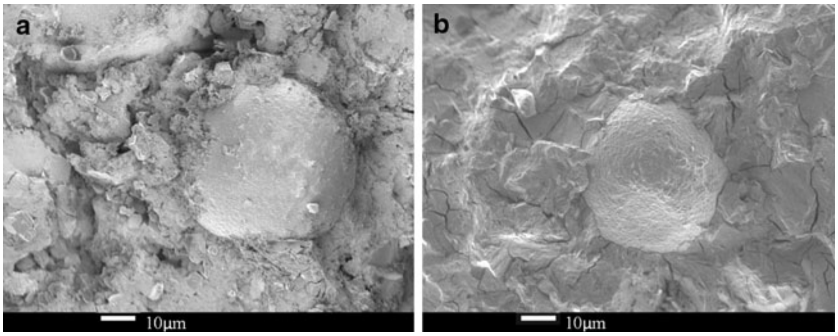


Fig. 10 SEM images of the binder's microstructure: **a** the binder B-3, **b** the binder BD-3

Analyzing the results of the tests based on the ultrasound wave velocity (UWV) measuring (Fig. 9b), one can observe that a combination of the deflocculant and catalyst additives causes the compaction of the binder's structure, i.e., the ultrasound wave velocity, depending on the content of the FCC catalyst additive, increases by 300–480 m/s in the samples of the BD-type binder. Meanwhile, in the case of the B-type binder without a deflocculant, the addition of 5% catalyst slightly increases the compactness of the binder's structure (UWV is increased by 40 m/s). However, a higher amount of catalyst negatively affects the structure of the material (UWV is decreased).

The microstructure of binders was estimated by SEM. Fig. 10 provides the photos of microstructures characteristic of the B and BD-type binders. We can see that the material formed during the hardening of the B-type binder without a deflocculant is very porous (Fig. 10a), meanwhile, the material of the BD-type binder with a deflocculant is compact (Fig. 10b), but contains microcracks developed due to shrinking of the material in the course of hardening.

The above results obtained in SEM and UWV tests are in agreement with each other, showing that, when a deflocculant is added, the structure of the catalyst-containing CAC binder is much more compact, compared to that without a deflocculant. The compactness of the CAC binder structure is increased upon the addition of the deflocculant because the deflocculant (superplasticizer) acts as a dispersing agent: it is adsorbed on the cement particles, causing mutual electrostatic repulsion of particles. As a result, the cement paste fluidity is improved [21, 29–31] and self-compacting of concrete occurs [31]. The polycarboxylate ether-type deflocculant Castament FS-40 used in this study is a very efficient dispersant [22–24], therefore, the catalyst containing CAC binder with the deflocculant additive has a much more compact structure, compared to that of the binder without a deflocculant. These results correlate well with the data of the mechanical tests, implying that the more compact the binder's structure, the better its mechanical properties.

Conclusions

(1) The results obtained in investigating the hydration of the binder, consisting of calcium aluminate cement ($Al_2O_3 > 70\%$) with the additives of the FCC catalyst waste and deflocculant Castament FS-20, show that the FCC catalyst, like a polycarboxylate deflocculant, is an active additive, influencing the hydration process of the binder.

(2) In the structure of the hardened binder, without or with additives added separately or together, certain amounts of unhydrated CAC minerals, CA and CA_2, and the hydration products, such as CAH_{10}, C_2AH_8, and amorphous AH_3, were found. However, smaller

amounts of CAH_{10} and amorphous AH_3, and a larger amount of C_2AH_8 were found in the binder with a deflocculant, than those observed in the binder without a deflocculant additive.

(3) It has been found that in the case, when the FCC catalyst and deflocculant are simultaneously used, the FCC catalyst produces a positive effect on the formation of the CAC binder's structure, increasing its mechanical strength.

The results obtained in this article allow to predict that the FCC catalyst and deflocculant, simultaneously used as the additives to the CAC binder, will enable to control the hydration process of the binder.

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