SPENT FCC CATALYST FOR IMPROVING EARLY STRENGTH OF PORTLAND CEMENT

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ABSTRACT

Spent catalytic cracking catalyst (FCC) from petrol industry has proven to be a very active pozzolanic material. This behavior leads to an additional increase in the strength of the mortar that contains this catalyst. Pozzolanic effects tend to be considered for periods above three days, whereas in shorter times, the influence of pozzolan is usually negligible. However, reactivity of FCC is so high that both pozzolanic effects and acceleration of cement hydration are evident in short curing times. This paper presents a study of the effect of the presence of FCC on cement hydration and the reaction products in the first 48 hours of curing time, carried out by determining flexural and compressive strength of mortars with 3 different tests (substitution, addition and with accelerator). For the FCC behavior comparison, limestone, mullite and andalusite were used. Finally the characterization of hydrates was performed by thermogravimetry.

Keywords: Pozzolan, spent catalytic cracking catalyst, mechanical strength, thermogravimetry.

INTRODUCTION

Due to the advantages offered, the use of pozzolans is a common practice in the preparation of mortars and concrete. These advantages can be classified as: technological (improvements in strength, durability, workability, shrinkage and hardening, among others); economical (low cost and high performance); ecological (energy consumption, CO_2 emissions, disposal elimination); and social (employment generation sources). Moreover, in some cases, pozzolans have been used as a replacement of high percentages of Portland cement[1] and for obtaining geopolymers[2]. However, the most significant advantage of the use of pozzolans is an increase in mechanical strength over long time periods. This can be explained as a consequence of the formation of additional amount of hydration products due to the reaction between the pozzolan and the calcium hydroxide (released by the hydration of the cement)[3, 4]. It has also been shown that the inclusion of pozzolans in mortars and concretes produces an increase in durability against chemical aggressors. This is due to a reduction in permeability as a result of the location of the products generated from pozzolanic reaction in the pores [5, 6, 7, 8].

Studies carried out on spent catalytic cracking catalyst (FCC) highlight its behavior as a very active pozzolan, explained by its high mechanical strength obtained and by the fixation of significant amounts of calcium hydroxide released in cement hydration[9, 10, 11, 12, 13]. Studies in mortars have shown that mortars substituted with FCC decrease workability with respect to the control mortar. This can be explained based on the zeolitic type structure of the spent catalyst, which results in a greater water demand[9, 14]. Payá et al[10] reported that initial and final setting times are noticeably influenced by the presence of FCC in cements, being shorter probably because the sulphate content in blended cement decreased with the FCC content by dilution effect. Conventional pozzolans act as inert during short curing times, because the reaction between calcium hydroxide and the pozzolan is slow. Unlike conventional pozzolans, the specific characteristic of FCC is its high reactivity in short time periods because the fixation of calcium hydroxide is very fast, producing an increase in mechanical strength[15, 16, 17]. The FCC behavior with respect to mechanical strength in short and medium curing times has been extensively studied [9, 10, 11, 15]. Mortars with FCC also demonstrated high reactivity at extended curing times, being obtained higher mechanical strength than the control specimens with substitutions up to 15% [9, 14, 18]. However, literature that studies FCC behavior in very short curing times (during the first 48 hours) barely exists. This period is of especial interest because of the potential applications in precast components, which need to reach a specific strength within a short time period. Therefore, the aim of this work is to study the aforementioned behavior in the first 48 hours of curing time at room temperature for different water/binder ratios and with the presence of accelerating agents for setting and hardening. For FCC comparison purposes, limestone, mullite and andalusite were used and three different mortars tests were performed, namely: (1) with pozzolan as a cement replacement, (2) with the addition of pozzolan; and (3) with accelerator.

RESEARCH SIGNIFICANCE

Several studies on the use of spent FCC catalyst as a mineral addition in Portland cement binder have been reported. Those studies have mainly focused on some properties of fresh and hardened binder. Nevertheless, none of them have reported the evolution of mechanical strength and pozzolanic activity in very short curing times (the first 48 hours). This is relevant because the high pozzolanic reactivity reported for this mineral addition can be utilized in precast applications where high strength is required in very short times.

EXPERIMENTAL INVESTIGATION

The spent catalyst (FCC) was supplied by BP OIL España S.A. (Castellón-Spain). It is basically an aluminosilicate material whose composition is similar to metakaolin. Table 1 shows the FCC composition along with that of the cement used (ordinary Portland cement, OPC). In preparation for the experiments, FCC was ground for 20 minutes in an alumina ball mill (Gabrielli Mill-2) to increase its reactivity (FCC20). This drastically reduces particle size (see Figure 1) and transforms its original spheroidal form into irregular particles (Figures 2a and 2b). Spanish Portland cement CEM I-52.5R was used to prepare the pastes specimens and mortars. This was supplied by Cemex (Buñol-Spain), which also supplied the limestone filler, with a high degree of purity (93.3% in $CaCO_3$). Mullite ($Al_6Si_2O_{13}$) and andalusite (Al_2SiO_5) were also used to establish a comparison with FCC20. These selected crystalline materials (non-reactive), with similar chemical composition to FCC20, allow us to study the dilution effect. The percentages of the aluminum and silicon oxides present in these materials, respectively, are: 71.8% and 28.2% for mullite, and 63% and 37% for andalusite. Average mean particle diameters for the cement and mineral additions studied are: cement = 15.01 μm, FCC = 19.96 μm, mullite = 37.36 μm, andalusite = 31.05 μm, and limestone filler = 23.38 μm.

For thermogravimetric studies, three cement pastes were prepared with a water/binder ratio of 0.5: the control paste (only cement), and two pastes, cement/FCC20 and cement/limestone, with a 9:1 ratio. After mixing, the samples are kept in closed containers at 20 °C (68 °F). During the first 48 hours, portions of the samples are taken and pulverized with an agate mortar. Hydration is interrupted with acetone. The solid is filtered and dried at 60 °C (140 °F) for half an hour. These samples are studied by means of the thermogravimetric analysis, using Mettler-Toledo TGA850 equipment with a horizontal furnace. Sealed aluminum crucibles of 100 µL ($2.64x10^{-5}$ gal) were used with a lid that has a microhole to create a water vapor self-generated atmosphere. The analysis was carried out in a dry nitrogen atmosphere with a flow of 75 mL/min (0.0198 gal/min), a heating rate of 10 °C/min (50 °F/min), and a temperature interval of 35-600 °C (95-1112 °F).

The mortar preparations were carried out according to UNE norm 83.452-88[19], mixing 450 g (15.87 oz) of either plain Portland cement or a cement/mineral addition mix, 1350 g (47.62 oz) of siliceous aggregate, water, and superplasticizer, if necessary. The superplasticizer is of new generation (superplasticizer additive of high range water reduction to generate high mechanical strength), commercialized under the name of Viscocrete, supplied by Sika, S.A. (Ctra. de Fuencarral, 72. 28108 – Alcobendas, Madrid). Additionally, a commercial accelerator named Oleoplast was added (supplied by the same company). Mortar specimens of 40x40x160 mm (1.57x1.57x6.30 in) were prepared according Spanish norm UNE-EN 196-1[20]. Once the specimens were prepared, these were kept in a wet chamber at 20±1 °C (68±1.8 °F), until 15 minutes before the specified curing time. Then, they were demoulded and mechanical compression and flexural tests were carried out in the established curing times. Each test, for a given curing time, consists of three prismatic specimens that are tested in bending mode. The six pieces generated from this test are finally tested in compression mode.

EXPERIMENTAL RESULTS AND DISCUSSION

Mortars with FCC20 substitution

A first experiment was carried out with two purposes: to study the effect of FCC20 on cement mortars during the first few hours of curing time, and to study the role of the water/binder ratio (binder being the sum of cement plus pozzolan) in the development of the mechanical strength. Substitution mortar specimens of 40x40x160 mm (1.57x1.57x6.30 in) were prepared with and without cement substitution for 15% FCC20, with different water/binder ratios (0.35-0.50), and cured for 8, 16, 24, 36 and 48 hours. To ensure that consistency in mortars was adequate in the mixes with lower ratios of water/binder, variable amounts of superplasticizer were added.

Table 2 shows the conditions under which the mortars were fabricated. If we represent the index of strength activity (R_{fi}/R_{fo} for flexural strength, or R_{ci}/R_{co} for compressive strength, being R_i the strength for substituted mortar and R_o the strength for control mortar at the same age and with the same w/b ratio), we obtain the graphs shown in Figures 3 and 4. Considering that we are making mortars with a 15% substitution, if FCC20 behaves as inert, the index should be 0.85.

Regarding the index of flexural strength activity values (fig. 3), we observe that they are very disperse, showing no clear trends. However, only the values at 36 hours of curing time increase as the w/b ratio increases. It is also noteworthy that the w/b=0.5 ratio is the only that presents all values above 0.85.

With respect to the index of compressive strength activity from figure 4, we can state the following:

- In general, the index increases as the w/b increases.

- At 16 hours of curing time, the mortar with FCC20 behaves better than the mortar containing only cement (indices much greater than 0.85). This highlights the fact that FCC20 experiences a pozzolanic reaction at a very short curing time.
- Starting at 24 hours, and compared to the high values obtained at 16 hours, the index values decrease considerably for all w/b ratios.

In summary, the behavior observed in figure 4 is due to the fact that in a first interval (8-16 hours) FCC20 immediately consumes the portlandite ($Ca(OH)_2$) released in cement hydration. Between 24 and 48 hours, a greater hydration is necessary to proceed with the pozzolanic reaction, this can easily be achieved with a greater w/b ratio.

Mortars with FCC20 addition

In this section, we study the effect of FCC20 on mortars where the cement content remains constant and the pozzolan is added (added mortars). To this end, a first experiment was designed to analyze compressive strength reached by mortars with a w/c ratio=0.45, at 16 hours of curing time, with a 10 % substitute of aggregate with other additions. To obtain an adequate workability, 0.5% of superplasticizer was added. The additions were FCC20, limestone filler, and two crystalline products whose chemical composition is similar to the catalyst: mullite and andalusite (aluminum silicates).

Compressive strength values obtained are shown in Figure 5. On one hand, if the ratio of the mechanical strength of added mortars and control mortar is calculated, the dilution effect of the inert materials could be observed (this ratio would range between 0.92-0.93). On the other hand, FCC20 clearly shows a pozzolanic reaction (the ratio value is 1.28). This is a considerable gain in compressive strength for a short curing time of 16 hours.

Next, and in light of these results, a second experiment was designed to study the effect of the FCC20 addition on curing time. Table 3 shows the conditions under which the mortars were fabricated.

As observed in Figure 6, values of flexural strength show no definite trend. However, notice that at 48 hours, FCC20 mortar is the one that presents the greatest strength value. With regards to compressive strength, it can be seen that for all curing times, the FCC20 mortar shows the highest values compared to mortar without addition (control). Additionally, the difference between mortar with FCC20 added and the control mortar increases with curing time, suggesting the high reactivity of this pozzolan in this curing time range.

Influence of activators

In order to study the influence of some chemical activators on the behavior of this pozzolan, a liquid concrete accelerator was used, whose effect is to shorten initial setting time. Two types of mortars, with and without accelerator, were prepared for the analysis with a w/c=0.55. Given that speeding up the setting process considerably decreases mortar workability, a 0.5% superplasticizer was dosified. The first types of mortars contain only cement (control specimen). The second types of mortars were prepared with the addition of 10% FCC20. Dosage of mortars is shown in Table 4 and results of mechanical strength are summarized in Table 5. From Table 5, it should be noted that, for flexural as well as for compression strength, the mortars with FCC20 present greater values than the control mortars. This indicates that the pozzolan plays an important role under the effect of the accelerator. Notice that very high values, above 30 MPa (4351.13 psi), are reached at 24 hours. The effect of the accelerator is shown in Figure 7 by means of the indexes of strength activity, R_{ci}/R_{co}, of mortars with added 10% FCC20, prepared with (w/c ratio = 0.55) and without setting accelerator (w/c ratio=0.50). Observe that these indexes are clearly higher in mortars prepared with accelerator, whose values are above 1.7 in all cases, making it very interesting for production in prefabricated products and high strength concretes in very short times.

Studies in pastes

Reactivity of FCC20 with respect to calcium hydroxide fixation released in cement hydration has been studied through thermogravimetric analysis at longer curing times [21, 22]. However, it is of interest to study the evolution of lime fixation in the first 48 hours. Results are discussed in this section. Control cement pastes were prepared with w/c=0.5. In addition, pastes with cement/FCC20, and with cement/limestone were prepared in a 9:1 proportion (keeping w/c=0.5). Curing times were 8, 16, 24, 32, 40 and 48 hours. Figure 8 shows DTG curves in a 35-300 °C (95-572 °F) interval, and curing times of 8 hours (Figure 8a) and 48 hours (Figure 8b). It is well known[23] that in the hydration of Portland cement at short curing times, the presence of gypsum (dehydrate form), calcium silicate hydrates (CSH) and calcium hydroxide (CH), is observed. Also, the amount of CH and CSH increases with time while the amount of gypsum decreases. In Figure 8a, two ranges are identified. The loss observed in the range between 100-180 °C (212-356 °F) is attributed to dehydration of CSH and the ettringite formed (Af_T, $(CaO)_3(Al_2O_3)(CaSO_4)_3 \cdot 32H_2O$), together with the first dehydration of the remaining gypsum (Cs_1, $CaSO_4 \cdot 2H_2O$). In the range between 180-200 °C (356-392 °F), a peak is observed towards 196 °C (384.8 °F), attributed to the second dehydration of the gypsum (Cs_2). As the curing time increases for these pastes, the peaks corresponding to gypsum disappeared and a new peak emerges around 202 °C (395.6 °F), as is observed in Figure 8b. This is attributed to the dehydration of calcium aluminosilicate hydrates (CASH) and calcium aluminate hydrates (CAH). This figure also shows how the presence of FCC20 in the paste produces an increase in the formation of ettringite, due to the presence of aluminum in FCC20, resulting in a better resolution of the first range. The presence of ettringite and CSH has been corroborated by electron microscopy, as shown in Figure 9, where ettringite can be observed in the form of needles along with amorphous products, which were identified as CSH.

To calculate the fixed lime in the pozzolanic reaction, provided that the proportion of cement affects the percentage of fixed lime in pastes, we should consider the reduction in the cement content by the addition of the mineral admixture. Thus, the percentage of fixed lime can be calculated according to the following equation[24]:

$$\%Fixed\ lime = \frac{\left[(CH)_c * C_{\%}\right] - (CH)_i}{\left[(CH)_c * C_{\%}\right]} * 100 \qquad (1)$$

where $(CH)_c$ is the lime present in the control paste for a given curing time, $(CH)_i$ is the amount of lime in the paste with the addition at the same curing time, and $C_{\%}$ is the proportion of cement present in the substituted paste (in our case 0.9).

Results of fixed lime are shown in Figure 10. FCC20 shows a first important fixation of lime at 8 hours. Later, there is a period of time where the "particle" effect is predominant over the pozzolanic reaction (16-24h). This effect causes an acceleration of the hydration reaction of cement, and, as a consequence, negative values for fixed lime are obtained (in the pozzolan paste, the hydration of the cement is quicker than that in the cement paste). Finally, between 32 and 48 hours, there is a new period in which the percentage of fixed lime increases gradually.

CONCLUSIONS

1. Substituting cement by FCC20 produces a maximum increase of Rc in the period between 8-16 hours, compared to mortars without substitution; however, this effect is less pronounced between 24-48 hours.
2. The effect of FCC20 during the first 48 hours of reaction time is of pozzolanic type. In this period, this effect is more evident in mortars with ratios w/c=0.5. This suggests that reaction speed is boosted if there is an increase in the liquid phase.

3. The effect on the mechanical strength, which occurs in mortars with FCC20, is observed in a greater extent if FCC20 is dosified as an addition to the cement. This effect takes place especially during the period between 24-48 hours, when the amount of portlandite released by the hydration of cement begins to increase considerably.
4. The use of accelerators boosts FCC20 activity, increasing mechanical strength during the first 48 hours considerably.
5. Thermogravimetric analysis data show that FCC20 produces lime fixation in a short curing time (8 hours). Later, there is a period of time where the "particle" effect is predominant over the pozzolanic reaction (16-24h). After 24 hours, the pozzolanic effect is once again activated.

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Table 1 – Chemical composition of Portland cement and FCC20 (%)

Parameter	FCC	OPC
L.O.I.*	1.50	3.02
CaO	<0.01	62.87
SiO_2	48.20	20.21
Al_2O_3	46.00	4.94
Fe_2O_3	0.95	2.85
MgO	<0.01	1.05
K_2O	<0.01	0.95
Na_2O	0.50	0.10
SO_3	0.30	3.54

* Loss on ignition

Table 2 – Mix formulations of substitution mortars

Curing time (hours)	w/b ratio	Types of Mortars	% Superplasticizer
16, 24, 36, 48	0.35	1. Control, 2.Substitution15% FCC20	1.0
	0.40		0.5
	0.45		0.5
	0.45		--
	0.50		--

Table 3 – Mix formulations of mortars with additions

Curing time (hours)	w/c ratio	Mortar types	% Superplasticizer
8, 16, 24, 36, 48	0.5	1. Control 2. Addition 10% FCC20 3. Addition 10% limestone filler	-

Table 4 – Mix formulations of mortars with FCC20 addition and accelerator

Curing time (hours)	w/c ratio	Types of mortars	% Superplasticizer	Accelerator (with reference to the total water mass)
8, 16, 24, 32, 40, 48	0.55	1. Control 2. Addition 10% FCC20	0.5	1/6

Table 5 – Mechanical strength of mortars with FCC20 and accelerator added. w/c ratio = 0.55

Curing time (hours)	Rf (MPa)		Rc (MPa)	
	Control	10% FCC20	Control	10% FCC20
8	0.7 [0.12]	2.2 [0.09]	2.5 [0.12]	9.5 [0.75]
16	2.8 [0.21]	6.0 [0.35]	12.5 [1.39]	27.4 [2.41]
24	4.6 [0.31]	7.0 [0.16]	17.8 [5.11]	33.9 [2.46]
40	5.3 [0.15]	7.3 [0.17]	21.4 [4.81]	39.4 [3.74]
48	5.8 [0.36]	6.8 [0.26]	23.9 [3.51]	40.9 [1.26]

Note: Numbers in brackets are standard deviations in MPa (1 MPa= 145 psi).

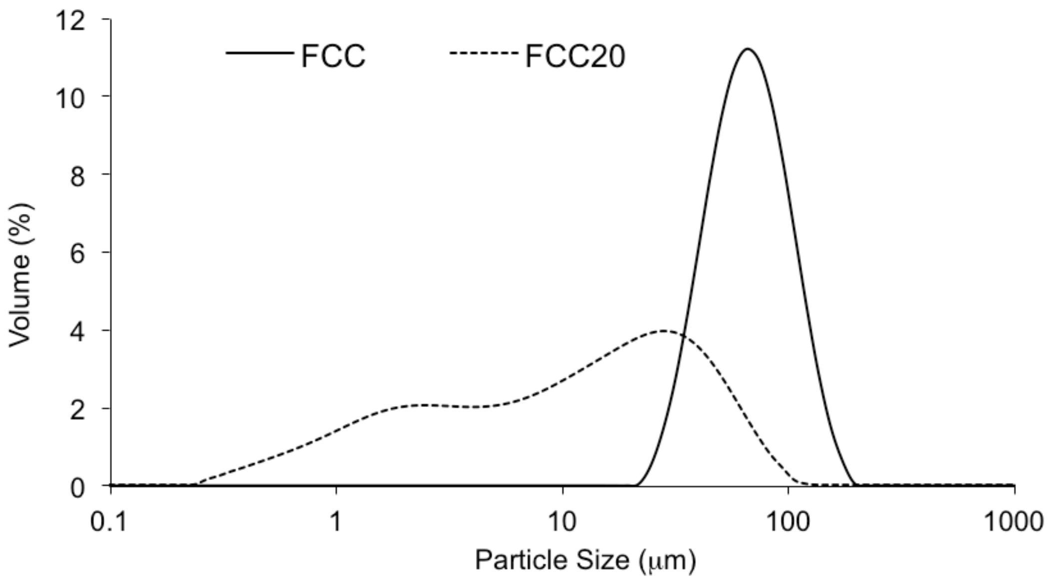
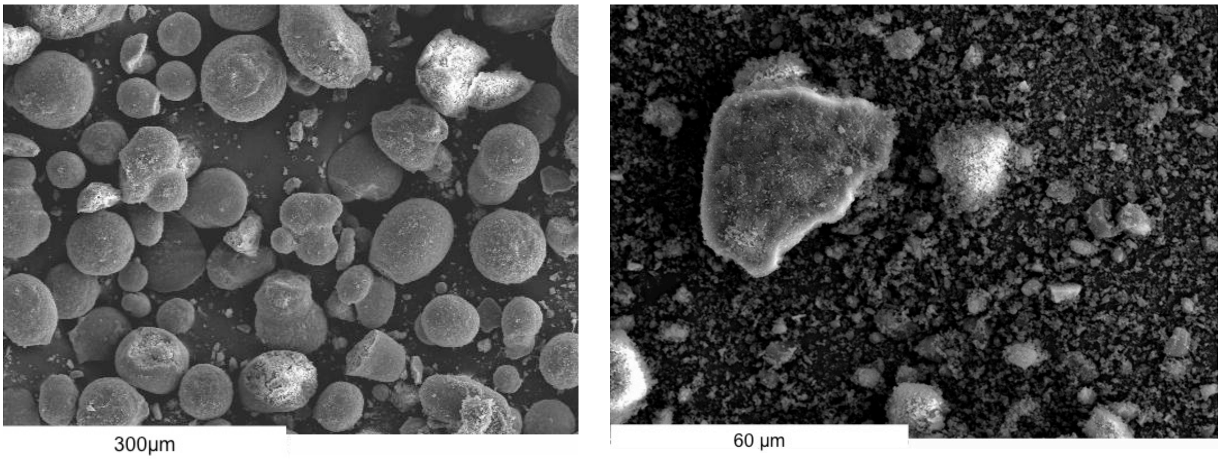


Fig. 1– Particle size distribution of FCC before and after 20 min of grinding. (Note: 1μm = 3.94×10^{-5} in)



a) b)

Fig. 2– SEM photographs a) original FCC b) FCC20.

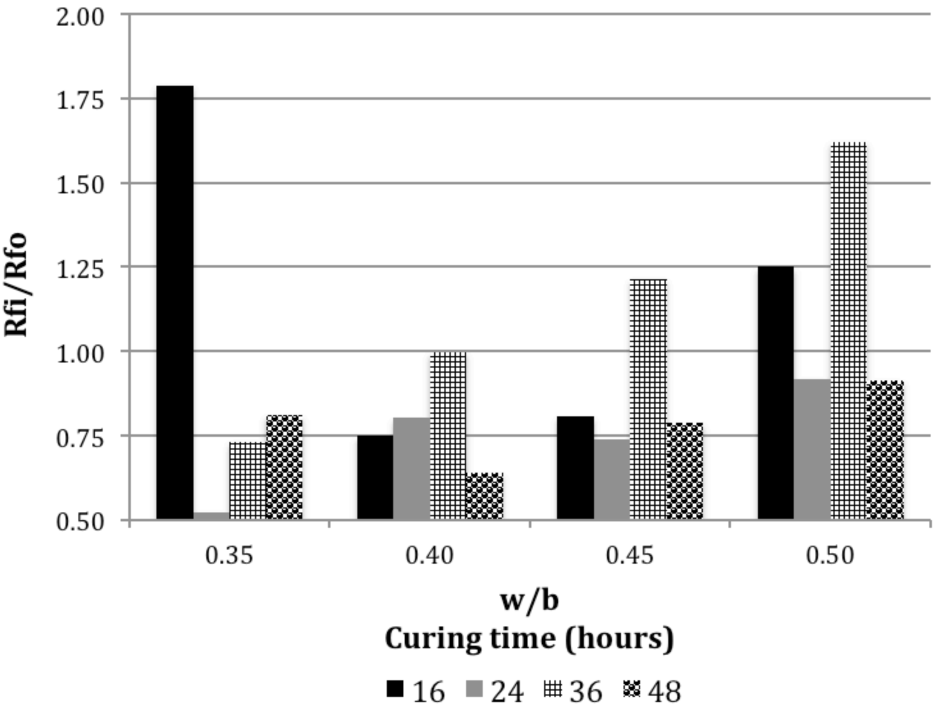


Fig. 3 – Index of flexural strength activity as a function of curing time and w/b ratio for substituted mortars.

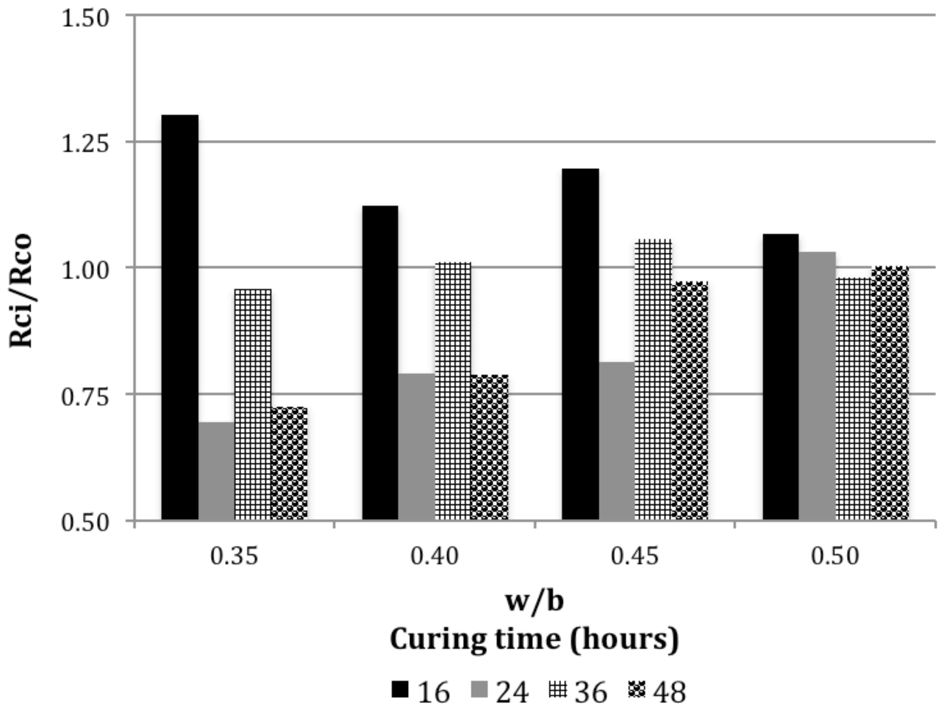


Fig. 4 – Index of compressive strength activity as a function of curing time and w/b ratio for substituted mortars.

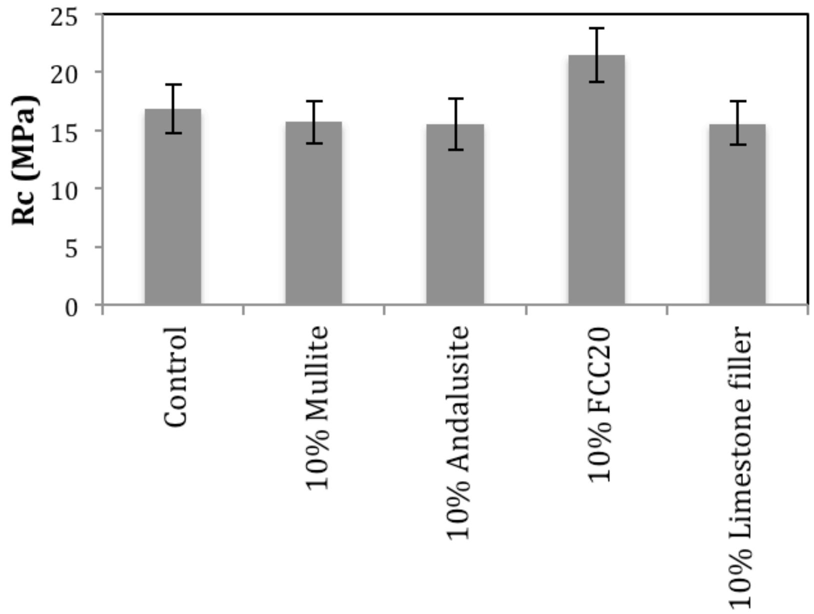
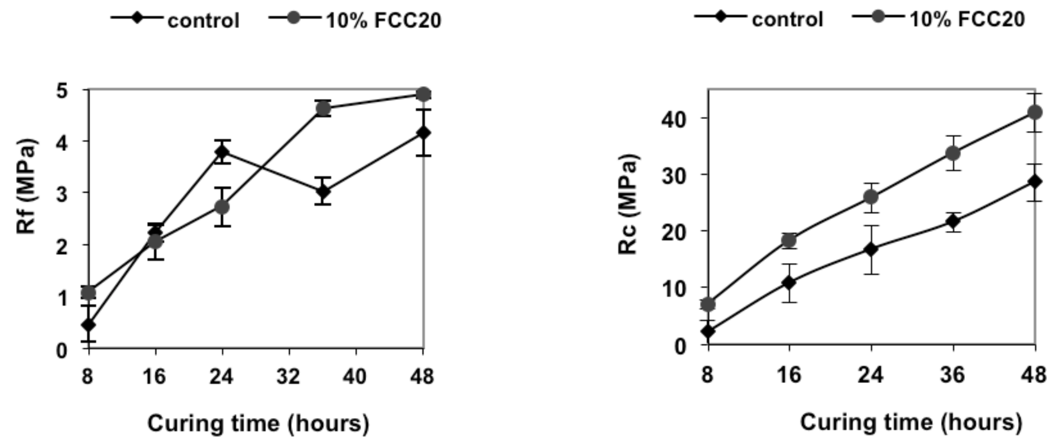


Fig. 5 – Compressive strength of mortars with additions, cured for 16 hours and with a w/c ratio=0.45. (Note: error bar means standard deviation, 1 MPa= 145 psi).



a) b)

Fig. 6 – Mechanical strength of control mortars and mortars with FCC20 addition. a) Flexural strength; b) Compressive strength. (Note: error bar means standard deviation, 1 MPa= 145 psi).

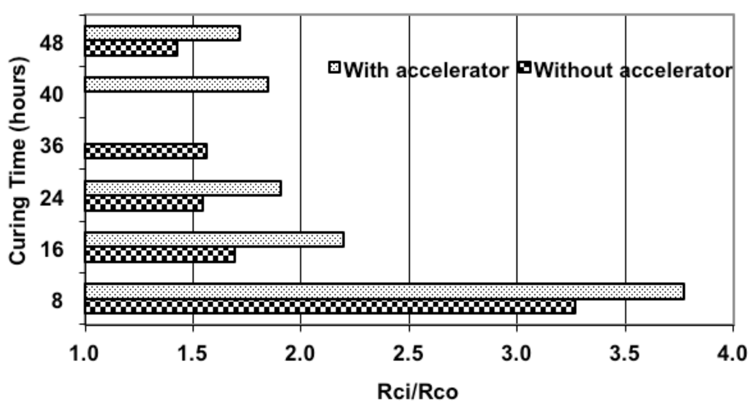
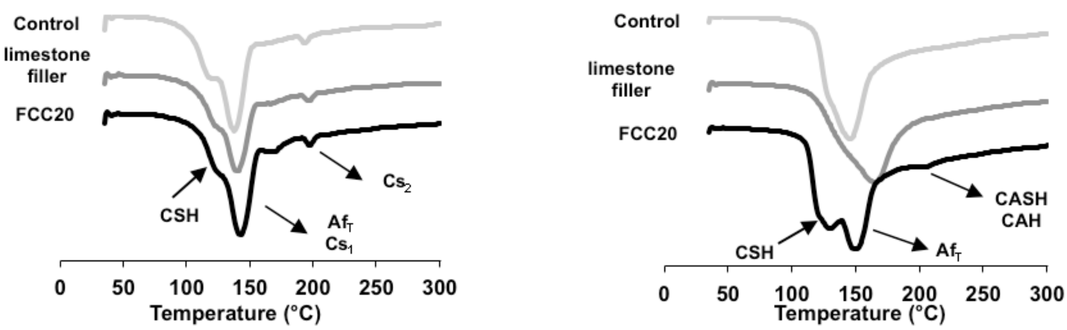


Fig. 7 – Index of strength activity, R_{ci}/R_{co} of mortars with added 10% FCC20, prepared with (w/c ratio = 0.55) and without setting accelerator (w/c ratio=0.50).



a) 8 hours b) 48 hours

Fig. 8 – DTG curves for control, 10% limestone filler and 10% FCC20 pastes, cured at different ages. (Note: ºF=1.8xºC+32).

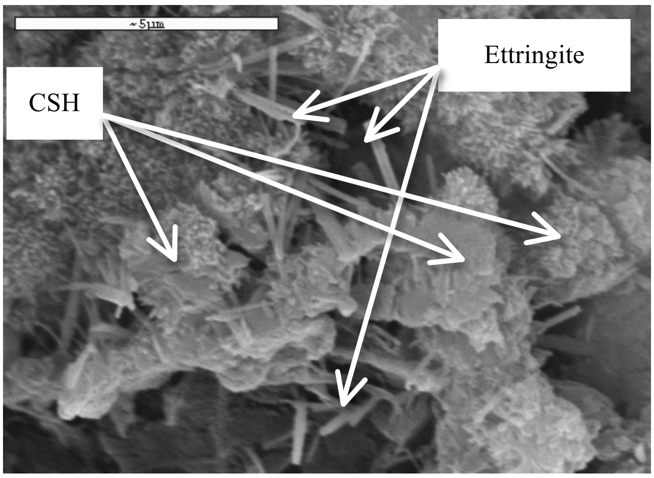


Fig. 9 – SEM of cement FCC20 9:1 paste cured for 24 hours.

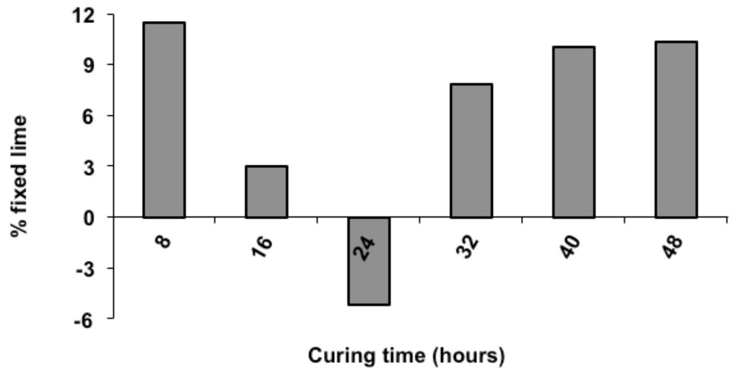


Fig. 10 – Percentage of fixed lime in cement pastes with 10% of FCC20. Evolution with curing time.