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***EFFECT OF SILICOALUMINOUS POZZOLANS ON THE HYDRATION
PROCESS OF THE PORTLAND CEMENT CURED AT LOW TEMPERATURES***

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ABSTRACT:

Hydration processes in cement pastes and mortars both with the addition of silicoaluminous pozzolans (metakaolin MK and catalyst used in catalytic cracking FCC) have been studied in this paper. Additionally, the amount of hydrates and portlandite in cured pastes from 5 to 20° C and for 3 to 28 days curing time has been determined. The microstructural study by using thermogravimetric analysis in pastes has shown that, at low temperatures (5-10°C) the FCC acts mainly as a pozzolan, whereas MK also produces acceleration in the hydration of cement. The influence from the mechanical point of view is a relative increase in the compressive mechanical strength of mortars

cured at 5° C for both pozzolanic materials, through replacement of cement and through replacement of aggregate. These two pozzolans are effective materials to compensate for setting and curing conditions at low temperatures, especially in aggregate replacement mortars.

Keywords: pozzolan, spent catalytic cracking catalyst, metakaolin, low temperature curing, cement replacement, aggregate replacement.

1. INTRODUCTION

The influence of curing temperature on the properties of concrete is of great importance. The manual of concrete practice recommended by the ACI [1], defined cold weather as the period exceeding 3 consecutive days, where the average temperature is below 5° C and the temperature is above 10° C in most of the midday. The EHE08 [2] provides the 5° C as the minimum temperature to be mixed in cold weather, since at low temperatures the cement hydration is delayed. Besides at temperatures below 0° C, mortars and concretes can experience problems because of freezing water.

Several examples [3-11] about the influence of temperature and curing time of concrete and mortar have been reported. The expected behavior by lowering the curing temperature for the mortars compared to those cured at 20° C, is a reduction of strength in the early ages of curing, maintaining or decreasing slightly at longer curing times.

The delay in the process of hydration of cement at low temperatures directly affects the production of portlandite, but also the pozzolanic reaction may be modified in these conditions. Some research [12-14] dealing with the issue of low temperature curing in cementitious matrices in the presence of pozzolanic materials has been reported, concluding that depending on the type of pozzolan, the curing behavior at low temperature may vary. The work carried out by Escalante-Garcia et al [12-13] shows the behavior of mixtures of cement with three mineral additions: PFA (Pulverized fuel ash),

VA (volcanic ash) and GGBFS (ground granulated blast furnace slag), for curing of 10, 20, 30, 40 and 60° C. In general terms, it has been observed that, for all mixtures with mineral additions in the early curing times, the compressive strengths are higher with increasing curing temperature. For mortars with PFA, the pozzolanic reaction is slower for the temperature of 10° C, without obtaining improvements in mechanical properties, for all ages and curing temperatures. In the case of the addition of VA, the mechanical strength has been improved when curing at 40 to 60° C, but not when curing at low temperature. Finally, the addition of GGBFS showed that at 10° C, the contribution of such addition to the strength gain was very low even at 28 days curing time.

A study on the influence of silica fume in mortars with different cements cured at 0, 27 and 60° C was made by Chakraborty et al [14]. Strength values were higher than their respective controls for all curing temperatures. These authors concluded that the rate of hydration of C_3S is heavily influenced by lower curing temperature, greatly affecting the strength obtained in the early curing times. The loss of strength is compensated with the replacement of cement by silica fume in a percentage of between 8 and 12%.

The aim of this paper has been the study of two silicoluminous pozzolans in order to observe their evolution in pastes and mortars cured at different temperatures, by using thermogravimetric techniques and mechanical strength behavior. The silicoaluminous pozzolans, selected for the research are metakaolin (MK) and spent catalyst for catalytic cracking (FCC). The metakaolin (MK) is a synthetic product, obtained by calcining kaolin; it is a very fine material and with high pozzolanic activity, which improves both mechanical strength and durability of mortar and concrete to which is added [15-18]. The catalyst used in catalytic cracking (FCC) is a silicoaluminous zeolite, which is generated as waste in the process of catalytic cracking

for obtaining naphas. This material has proved to behave as an excellent pozzolan from early curing times, improving both durability and mechanical behavior of the cementitious matrices that contain it [19-25].

Thus, the main objective is to analyze the behavior of the pastes and mortars of Portland cement partially replaced by MK and FCC, at different temperatures and curing times to evaluate their behavior under such conditions.

2. EXPERIMENTAL SECTION

2.1 Materials, equipment and methodology

The materials used were Portland cement type CEM I 52.5 R, supplied by the company Cemex S.A, spent catalytic cracking catalyst from BP Oil, S.L refinery from Castellón (Spain), and finally, commercial metakaolin supplied by the ECC International company, under the name Metastar. The chemical composition of these materials is shown in Table 1. The limestone filler used (CA) was supplied by the company Cemex. The used accelerant, Sika Rapid 1, came from the Sika S.A Company with a recommended dosage between 0.5-2% of cement weight. The superplasticizer Sika-ViscoCrete 5-700, was also supplied by the SIKA SA Company. This is a polycarboxylic based superplasticizer whose recommended dosage is in range 0.5-1.5% in respect to the weight of cement or binder.

Table 1. Chemical Composition of cement, FCC y MK (%)

FCC residue undergoes a milling process in order to improve its reactivity, as previous studies have shown that pozzolanic behavior increases with the decrease in the particle size. The grinding time determined as optimal by Payá et al [19] was 20 minutes, since

longer grinding time did not improve the reactivity of the material. Figure 1 shows the particle size distribution of the FCC and MK pozzolans, and limestone filler, CA

Figure 1. Distribution particle size of FCC, MK and CA.

The average diameter in ground FCC is 19.73 μm , 22.03 μm in the CA and finally, 5.84 μm in the MK.

The thermogravimetric equipment used is a 850 TGA Mettler-Toledo module. The heating interval was 35-600° C, with a continuous 75mL/min flow of N₂ and a heating rate of 10° C/min applied to the analyzed samples. Aluminum crucibles with a pinhole lid were used and then sealed with the aim of achieving a water vapor self-generated atmosphere.

For thermogravimetry studies, pastes were prepared with 0.5 water /binder ratio, and a replacement of cement by MK or FCC of 15% percentage. The selected curing temperatures were 5, 10, 15 and 20° C, which was achieved by storing them in a water bath with temperature control. Samples were taken and curing times were 3, 7, 14 and 28 days. For each selected curing time, part of the paste was extracted and the hydration process stopped by the addition of acetone. Subsequently the mixture was filtered and dried in a furnace at 60° C for 30 minutes.

To study mechanical strength behavior, two types of mortars were manufactured.

Firstly, mortars were made with replacement of a part of cement. These mortars were prepared by the mixing method proposed in UNE-EN 196-1 [26], with a 3:1 ratio aggregate/cement and water/binder of 0.5. The replacement percentage of cement by pozzolan was maintained at 15%, like the one used in cement pastes. The curing temperatures were 5 and 20° C and mechanical strengths were measured at 1, 2, 7 and

28 days curing time. In a second phase, mortars were produced by the addition of mineral admixture, by replacing a part of the aggregate by the pozzolan, maintaining the water-cement ratio constant and decreasing the water/binder ratio. The percentage of aggregate replacement by pozzolan was 10%. In these last mortars, the addition of a superplasticizer was necessary. The curing temperatures and times were those used in the previous phase, that is, 5 and 20° C.

In all experiments, the materials used for mixing were maintained at the required curing temperature for 24 hours before use, so that when they were mixed at the same temperature, they were subsequently cured.

3. RESULTS AND DISCUSSION

3.1 Thermogravimetric Studies

The reaction of Portland cement with water results in the formation of hydration products including portlandite. This portlandite released in the presence of active pozzolanic materials, may lead to the formation of new hydration products similar to those formed in the hydration of cement, and in order to study the formation of hydration products, cement/pozzolan pastes were prepared as described in the experimental section. These pastes were analyzed by the thermogravimetric technique, where the change in mass of a sample placed in a controlled atmosphere is continuously recorded [27]. Thus, during the heating of hydrated cement pastes, dehydration processes, and water loss associated with compounds formed in the reaction of Portland cement hydration, took places [28].

The temperature range of 35-600° C was chosen because the processes of dehydration of products formed and the dehydroxylation of portlandite are represented in that temperature range.

In the thermogravimetric curve (TG) of hydrated cement paste, a continuous loss of mass in the range 80-250° C is observed. The derivative curve (DTG) of the TG curve, allows us to identify different decomposition processes as Figure 2a shows.

Additionally, loss of mass for the combined water of hydrated calcium silicates (peak 1), ettringite (peak 2), and hydrated calcium aluminates and aluminum silicates (peak 3) are identified. The loss of mass that occurs in the temperature range 520-600° C (peak 4) is that corresponding to the dehydroxylation of portlandite. Figure 2a depicts the DTG curves for control, MK and FCC pastes cured at 5°C for 7 days. Figure 2b depicts the DTG curves of FCC pastes cured for 3 days at different curing temperatures (5-20° C).

Figure 2. DTG curves for control, MK y FCC pastes.

a) DTG curves for pastes cured at 5° C and 7 days curing time.

b) DTG curves for FCC pastes cured at different temperatures, for 3 days curing time.

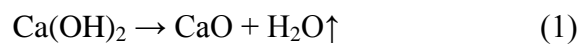
For DTG curves in figure 2, we can conclude that:

- From DTG curves of the control, FCC and MK pastes for 7 days and 5° C curing, it is observed the presence of hydrated calcium silicates and ettringite (peaks 1 and 2) in all of them. The peak corresponding to the hydrated calcium aluminates and calcium aluminosilicates (peak 3) is clearly present in the FCC and MK pastes, which shows that the pozzolanic reaction develops a greater amount of hydration products of this type than control paste.

- ✚ When the same paste at the same age is evaluated for different curing temperatures, it is observed that as the temperature increases, a greatest amount of hydration products occurs. As shown in Figure 2b, the peak corresponding to the hydrated calcium aluminates and aluminum silicates calcium only appears for 15 and 20° C.

For the evaluation of the mass loss related to the portlandite decomposition, an integration form was used suggested by Taylor [29], because the baseline does not remain horizontal during the heating process. Straight lines tangential to the curve of mass loss at the beginning, the turning point and the end of the process of dehydration of portlandite are drawn. The cut off points of the tangents define the initial and final temperatures of the process to calculate the mass loss involved in the process. In the case of a paste with a pozzolan, this peak represents the unreacted portlandite with it. From this data, we can determine the amount of portlandite fixed by the pozzolan in the paste [30].

The portlandite dehydroxylation (CH) is related to the reaction:



To know the percentage of fixed lime (%CH), the following equation is used:

$$\%CH = \frac{|CH_c * C\%| - CH_p}{|CH_c * C\%|} * 100 \quad (E1)$$

where CH_c is the amount of CH in the control paste for a given curing time, CH_p is the amount of CH present in the pozzolan paste at the same age and $C\%$ is the proportion of cement present in the pozzolan paste (in per unit). CH_c and CH_p determinations are made by stoichiometry from the following equation:

$$CH_p = \frac{H}{PM_H} * PM_{CH} \quad (E2)$$

where H is the water loss in the range evaluated previously noted, PM_{CH} is the molecular weight of $Ca(OH)_2$ and PM_H is the molecular weight of H_2O .

To perform the evaluation of the thermogravimetric curve, a total percentage of mass loss (P_T) of the sample from 35° C to 600° C is calculated and a mass loss due to the dehydroxylation of portlandite (P_{CH}) in the temperature range of 520-600° C is measured. If we subtract the percentage of loss due to portlandite decomposition to the percentage of total mass loss (P_T), we will obtain the percentage of water associated with the rest of the hydrates (P_H).

$$P_H = P_T - P_{CH} \quad (E3)$$

The results of portlandite present (CH_p) and the percentage of combined water in hydrates (P_H) in pastes with 15% substitution of cement by FCC and MK, are shown in Table 2. With data from portlandite present in the control paste and FCC and MK pastes, and using the equation E1, fixed lime percentage values are calculated for pastes with FCC and MK. The results are plotted in Figure 3.

Table 2. Portlandite percentages, CH_p and combined water in hydrates P_H for the control, FCC and MK pastes.

Figure 3. Fixed lime percentage (%CH) in FCC and MK pastes at 5, 10, 15 and 20° C.

From the analysis of fixed lime percentages by the FCC and MK pastes we conclude that:

- ✚ At 5° C, the FCC is able to fix portlandite for all curing times. In contrast, the MK, for the earliest curing times (3 and 7 days), negative fixed lime

percentages are presented because an acceleration in the hydration process of cement is produced, which is more important than the pozzolanic reaction. It is up to 14 days curing time when the pozzolanic reaction begins to be more important. At this temperature, the particle effect produced by the MK particle is very important, thus accelerating the cement hydration process.

✚ At 10° C, a similar behaviour is shown; the MK in the earliest curing times accelerates the cement hydration process but in this case, fixed lime data are less negative than those found as for the previous temperature. In the FCC pastes always positive fixed lime percentages are obtained and higher than those found for 5° C.

✚ MK paste at 15° C begins to have positive fixed lime percentages from 7 days, reaching over 30% for the 28 days curing time. The FCC's behavior is similar to lower temperatures, namely, positive values of fixed lime are always obtained, although at 28 days it is slightly below the MK value paste.

✚ Finally, at 20° C both pozzolan pastes yield positive fixed lime percentages for all curing times. For this curing temperature the particle effect is lower compared to the pozzolanic reaction.

✚ Therefore, we can conclude that at low temperatures the FCC has more pozzolanic reactivity than the MK, although the latter has a smaller

particle size. Thus, especially at 5 and 10° C, positive lime fixed percentages can be obtained, even at short curing times.

To analyze the index of hydration products formation in the pastes containing pozzolans, the difference values of the percentage of combined water in pozzolan pastes compared to those for the paste control, are calculated by using the equation:

$$\Delta PH = (PH)_{puz,T,t} - (PH)_{con,T,t} \quad (E4)$$

where $(PH)_{puz,T,t}$ the value of the percentage of water combined for pozzolan pastes for every curing time (t) and temperature (T) analyzed, and $(PH)_{con,T,t}$ the percentage of combined water value for the control paste at these same ages and temperatures.

The data obtained by applying the E4 equation are shown in Figure 4, which represents ΔPH versus curing time.

Figure 4. Differences in the percentage of hydration products versus curing time

Figure 4 shows that, at short curing time and low temperatures, combined water percentages for pozzolan pastes are lower than those obtained by the control paste.

However, with increasing curing time, this trend changes and high positive values ΔPH are obtained due to the presence of more hydration products resulting from the pozzolanic reaction. After 14 days of curing time, except for 5° C, ΔPH values are always positive. For 20° C test, always positive values are reached, which indicate that the decrease in the temperature not only affects the cement hydration reaction but also the pozzolanic reaction.

3.2 Mechanical strength results

Mortars with cement substitution

The aim of this study is to test the effect of a low temperature curing process on the evolution of mechanical strength mortars with time. Mortars with 15% substitution of cement by MK or FCC were manufactured and cured at 5 and 20° C for this purpose. In this study on mechanical strength, limestone filler (CA) is also added in order to study the influence of particle effect in these experimental conditions. The dosage used for the control and replaced mortars is showed in Table 3.

Table 3. Dosage for control mortar and cement replaced mortars by FCC, MK and CA.

The compressive strength values (MPa) of control mineral and replaced mortars at 5 and 20° C are listed in Table 4.

Table 4. Compressive strength data (MPa) for the control and replacement mortars at 5 and 20° C.

With these data, a logarithmic adjustment (equation E5) of strength values (R_c) versus time (t) has been performed and the data of the equations obtained are summarized in Table 5 (also, the regression coefficient R^2 is given):

$$R_c = a + b \cdot \ln t \quad (E5)$$

Table 5. Coefficients of the logarithmic equations from mathematical ajustement according to equation E5, for control and replaced mortars cured at 5 and 20° C.

From the values of mechanical strength and the adjustment data for different mortars, we can conclude that:

- ✚ The strength data for mortars from early curing times and the lowest temperature are much lower than those obtained for mortars cured at 20° C. This behaviour occurs for all mixtures, the control mortar and for mortars with mineral admixtures.
- ✚ At 5° C, the compressive strength values of the control mortar and the FCC mortar are very similar, and consequently so are the values obtained from the settings of the logarithmic equation. The mortar with limestone filler presents a compressive strength lower than those found for the control one, a fact which is reflected in lower values of “b” in the logarithmic fit. Finally, the mortar with MK reaches mechanical strength higher than the control one in the 1-7 days period, which is reflected in a higher value of the parameter “a”, and slightly lower for the parameter “b”, in respect to the control mortar.
- ✚ At 20° C, as a result of a greater reaction rate in the cement hydration process, higher value of the parameter “a” is obtained for the control mortar (3.53 for 5 ° C experiences opposite to 18.93 for 20° C). But instead, the highest values of the parameter “b” are obtained for the FCC and MK mortars, representing this value the increase in compressive strengths with the curing time. The compressive strengths of mortars with

pozzolans are clearly higher to the control mortar at long curing times due to pozzolanic contribution.

- ✚ The values of R_c with CA mortar, are usually lower than control mortar, which corresponds to an inert material. Therefore, no pozzolanic activity for CA is related to “b” value for 20° C experience.

The increase of the strength values between control and replacement mortars for all ages of curing was represented in order to know the influence of replacing part of cement by FCC, CA and MK. These graphics are depicted in Figure 5. The control mortar strength data are multiplied by 0.85, because the pozzolan mortars contain 15% less of cement. The increase strength value ΔR is calculated as:

$$\Delta R = (R_C)_A - 0,85 * (R_C)_0 \quad (E6)$$

where $(R_C)_A$ is the strength value of the mortars with FCC, MK and CA, and $(R_C)_0$ is the value of the control mortar, for the same curing temperature and curing time.

Figure 5. Increase in compressive strength (ΔR) related to the replacement of cement by FCC, MK and CA, cured at 5 and 20° C.

From the study of the graphs of increase strength, we can conclude that:

- ✚ The mortar with MK has the best behaviour at 5° C for all curing times except 28 days. In the previous termogravimetric studies, it is assumed that the MK, due to their small particle size has a very important effect

that accelerates cement hydration. The matrix densified and therefore improves the strength obtained compared to the control mortar. The FCC also showed an improvement on strength values. In this case, this improvement can be due mainly to the pozzolanic reaction.

✚ At 20° C, the mortars containing MK always yield positive ΔR values, whereas for the FCC, mortar only was positive at 7 and 28 days. At 28 days the values obtained for ΔR for the FCC and MK mortars are higher than those obtained at 5° C, which could indicate that the pozzolanic reaction is more important and occurs to a greater extent at higher temperatures. This fact was confirmed by comparing the values of ΔR for both curing temperatures; at 20° C, ΔR values were achieved around 20 MPa for both the FCC and for the MK mortars, however, at 5° C ΔR values were about 8 MPa.

✚ The limestone filler (CA) at 5° C exhibits negative ΔR values, because the particle effect cannot exceed the dilution effect due to substitution of 15% of cement for an inert material. The replacement of cement for CA at 20° C can achieve low positive ΔR values at 28 days, confirming that it behaves as inert material.

Mortars with aggregate substitution

Once analyzed the behaviour of the different mineral additions in the replacement of cement in mortars, the study with aggregate replacement mortars (addition mortar) was performed in order to analyze that kind of behaviour: additions in these mortars when the amount of cement remains constant. As indicated in the experimental section, in this type of mortar the amount of cement is kept constant and 10% of aggregate is replaced by FCC, MK and CA, so all of them have the same water/cement ratio (0.5). In this type of mortar, it was necessary to add a plasticizer to maintain the similar workability obtained by the control mortar ($145 \pm 10\text{mm}$). Table 6 shows the dosage used in the manufacture of addition mortars.

Table 6. Dosage of control mortar and aggregate replaced mortar by FCC, MK and CA.

In Table 7, the values of compressive strength (MPa) of control and addition mortars cured at 5 and 20° C are shown. As in the mortar substitution section, a logarithmic adjustment with strength values versus curing time has been made and, in Table 8, the coefficients of the equations obtained are shown.

Table 7. Compressive strength data (MPa) of control and addition mortars cured at 5 and 20° C.

Table 8. Coefficients for adjusted logarithmic equations of control and addition mortars cured at 5 and 20° C

From the values obtained from compressive strength and the adjustment data of different mortars, we can conclude that:

- ✚ For all ages in both curing temperatures, compressive strengths of mortars with FCC, MK and CA are higher than those obtained by the control mortar. This fact is reflected in higher parameters “a” and “b” in the logarithmic equation.
- ✚ At 5° C and 20° C, the FCC and MK mortars have the highest values of coefficients “a” and “b”, thereby demonstrating that the pozzolanic effect for these mixtures is very important for any age and temperature.

In this section, the increase strength (ΔR) was calculated, but in this case no correction to the data of control mortar strength was applied since all mortars contain the same amount of cement. The equation used was:

$$\Delta R = (R_C)_A - (R_C)_0 \quad (E7)$$

Where $(R_C)_A$ is the compressive strength for the addition mortars with FCC, MK y CA; and $(R_C)_0$ is the compressive strength value for the control mortar. In Figure 6, the results obtained are shown

Figure 6. Increase strength values (ΔR) in addition mortars with FCC, MK y CA, cured at 5 y 20° C.

From the study of the graphs of increase strength we can conclude that:

- ✚ The mortars containing FCC and MK presented $\Delta R'$ values always equal or higher than 3 MPa in all curing ages and for both temperatures studied.
- ✚ The values of both pozzolan mortars are very similar, reaching values higher than $\Delta R'=20\text{MPa}$ even for the mortars cured at 5°C and higher than 25 Mpa for the mortars cured at 20°C .
- ✚ The CA mortars also obtained positive ΔR values, being these much lower than those obtained by the FCC and the MK. This fact shows that both at 5°C and 20°C , the increase in the amount of fine materials has an important role in the strength gain at all curing times. This fact was not observed in mortars with cement replacement, where even its presence had a negative effect ($\Delta R' < 0$), increasing the amount of fines, the matrix becomes more dense and this will be reflected in improved mechanical strength.
- ✚ The highest strengths obtained by the FCC and MK are the result of the addition of a filler effect and the contribution of the pozzolanic reaction, for both 5°C and 20°C temperatures.

CONCLUSIONS

The following conclusions have been obtained from the study of the temperature effect on pastes and mortars with FCC, MK, and CA:

- ✚ Through a thermogravimetric study in cement pastes with 15% replacement of cement by MK and FCC carried out, it can be concluded that the FCC pastes present fixed lime percentages always positive for all curing times and all selected curing temperatures, that is, the pozzolanic reaction also occurs at low temperatures. Instead, the MK paste, in the early curing times and for temperatures below 20° C, has negative fixed lime percentages; in this case the MK behaves as a filler accelerating the cement hydration process, due to its higher fineness.
- ✚ The replacement of cement for MK and FCC produced an increase in strength in respect to control mortar; this improvement is mainly related to the strength gain due to the pozzolanic reaction. In addition, in the case of MK, at low temperatures, there seems to be an acceleration contribution of cement hydration. The compressive strength increase was higher at 20° C. Mortars with CA showed lower strength than the control mortar, despite having a particle effect, due to the lower cement content of these mortars
- ✚ The addition mortars (replacement of aggregate by MK, FCC and CA) obtained in all cases positive $\Delta R'$ values. For both temperatures studied (5° C and 20° C) the behaviour of the FCC and MK were similar and higher than the mortar with CA,

proving once again that the pozzolanic reaction contributes greatly to the strength gain.

- ✚ The incorporation of silicoaluminous pozzolans has a positive effect on the mortars cured at 5° C and 20° C. Their use would be recommended in cases where temperatures of mixing, setting and curing are low, especially in the partial replacement of aggregate.

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| | SiO₂ | Al₂O₃ | Fe₂O₃ | CaO | MgO | SO₃ | Na₂O+K₂O | LOI[*] | IR[#] |
|------------|------------------------|------------------------------------|------------------------------------|------------|------------|-----------------------|---------------------------------------|------------------------|-----------------------|
| CEM | 19,90 | 5,38 | 3,62 | 63,69 | 2,14 | 3,66 | 1,27 | 2,02 | 0,90 |
| FCC | 46,04 | 47,47 | 0,58 | 0,11 | 0,17 | 0,02 | 0,32 | 0,49 | n.d |
| MK | 51,60 | 41,30 | 4,64 | 0,09 | 0,16 | n.d | 0,63 | n.d | n.d |

n.d. not determined

^{*}LOI: Loss on ignition [#] Insoluble Residue

Table 1. Chemical Composition of cement, FCC y MK (%)

| | 5° C | | | | | | 10° C | | | | | |
|-----------|-----------------|----------------|-----------------|----------------|-----------------|----------------|-----------------|----------------|-----------------|----------------|-----------------|----------------|
| | CON | | FCC | | MK | | CON | | FCC | | MK | |
| | CH _P | P _H | CH _P | P _H | CH _P | P _H | CH _P | P _H | CH _P | P _H | CH _P | P _H |
| 3 | 7.92 | 11.56 | 5.97 | 11.43 | 7.91 | 11.14 | 9.84 | 13.41 | 6.90 | 13.28 | 8.48 | 12.73 |
| 7 | 11.30 | 14.31 | 9.03 | 13.98 | 10.63 | 13.30 | 11.97 | 13.85 | 9.10 | 13.78 | 10.57 | 14.31 |
| 14 | 14.29 | 15.78 | 9.49 | 15.72 | 10.16 | 15.32 | 12.75 | 15.10 | 7.47 | 16.50 | 9.34 | 18.88 |
| 28 | 14.23 | 16.72 | 9.52 | 17.81 | 10.17 | 18.71 | 14.86 | 16.48 | 9.49 | 18.75 | 8.23 | 18.30 |
| | 15° C | | | | | | 20° C | | | | | |
| | CON | | FCC | | MK | | CON | | FCC | | MK | |
| | CH _P | P _H | CH _P | P _H | CH _P | P _H | CH _P | P _H | CH _P | P _H | CH _P | P _H |
| 3 | 9.58 | 13.25 | 7.17 | 13.50 | 8.92 | 13.18 | 12.28 | 13.67 | 8.60 | 14.16 | 9.09 | 13.81 |
| 7 | 12.64 | 15.41 | 7.51 | 14.64 | 10.17 | 14.14 | 13.30 | 16.07 | 9.12 | 16.08 | 7.89 | 16.61 |
| 14 | 13.94 | 15.50 | 10.03 | 17.21 | 8.54 | 16.85 | 13.65 | 16.92 | 8.87 | 18.55 | 6.74 | 19.67 |
| 28 | 14.19 | 16.88 | 8.47 | 17.69 | 4.80 | 17.97 | 13.17 | 17.69 | 8.23 | 22.66 | 6.51 | 20.36 |

Table 2. Portlandite percentages, CH_P and combined water in hydrates P_H for the control, FCC and MK pastes.

| | Cement(g) | Addition (g) | Aggregate (g) | Water(g) |
|------------|------------------|---------------------|----------------------|-----------------|
| CON | 450.0 | -- | 1350.0 | 225.0 |
| FCC | 382.5 | 67.5 | 1350.0 | 225.0 |
| MK | 382.5 | 67.5 | 1350.0 | 225.0 |
| CA | 382.5 | 67.5 | 1350.0 | 225.0 |

Table 3. Dosage for control mortar and cement replaced mortars by FCC, MK and CA.

| t (days) | 5° C | | | | 20° C | | | |
|-----------------|---------------|----------------|----------------|----------------|----------------|----------------|----------------|----------------|
| | 1 | 2 | 7 | 28 | 1 | 2 | 7 | 28 |
| CON | 3.53 ±0.16 | 12.55 ±0.38 | 22.98 ±0.42 | 43.46 ±0.94 | 18.93 ±0.21 | 28.54 ±0.36 | 39.02 ±0.27 | 51.96 ±0.91 |
| FCC | 3.61 ±0.08 | 12.89 ±0.11 | 25.02 ±0.38 | 44.12 ±1.29 | 14.12 ±0.09 | 23.09 ±0.17 | 36.16 ±0.34 | 64.52 ±0.96 |
| MK | 5.20 ±0.09 | 17.34 ±0.32 | 30.29 ±1.18 | 40.75 ±0.68 | 19.38 ±0.15 | 24.86 ±0.28 | 39.28 ±0.81 | 62.60 ±0.70 |
| CA | 3.00 ±0.09 | 10.17 ±0.18 | 18.36 ±0.70 | 37.16 ±0.86 | 16.04 ±0.12 | 22.96 ±0.25 | 32.46 ±1.01 | 49.43 ±0.94 |

Table 4. Compressive strength data (MPa) for the control and replacement mortars at 5 and 20° C.

| | 5° C | | | 20° C | | |
|------------|-------|--------|----------------|--------|--------|----------------|
| | a | b | R ² | a | b | R ² |
| CON | 3.279 | 11.623 | 0.986 | 20.238 | 9.6293 | 0.993 |
| FCC | 3.655 | 11.894 | 0.995 | 12.388 | 14.794 | 0.975 |
| MK | 7.893 | 10.385 | 0.972 | 17.078 | 13.030 | 0.981 |
| CA | 2.362 | 9.921 | 0.976 | 15.582 | 9.807 | 0.989 |

Table 5. Coefficients of the logarithmic equations from mathematical adjustment according to equation E5, for control and replaced mortars cured at 5 and 20° C.

| | Cement (g) | Addition (g) | Aggregate (g) | Agua (g) | Plasticizer (g) |
|------------|-------------------|---------------------|----------------------|-----------------|------------------------|
| CON | 450.0 | 0 | 1350.0 | 225.0 | -- |
| FCC | 450.0 | 135.0 | 1215.0 | 225.0 | 3.0 |
| MK | 450.0 | 135.0 | 1215.0 | 225.0 | 3.0 |
| CA | 450.0 | 135.0 | 1215.0 | 225.0 | -- |

Table 6. Dosage of control mortar and aggregate replaced mortar by FCC, MK and CA.

| t (days) | 5° C | | | | 20° C | | | |
|-----------------|---------------|----------------|----------------|----------------|----------------|----------------|----------------|----------------|
| | 1 | 2 | 7 | 28 | 1 | 2 | 7 | 28 |
| CON | 3.53 ±0.16 | 12.55 ±0.38 | 22.98 ±0.42 | 43.46 ±0.94 | 18.93 ±0.27 | 28.54 ±0.63 | 39.02 ±0.41 | 51.96 ±0.28 |
| FCC | 8.57 ±0.40 | 21.39 ±0.55 | 44.51 ±0.63 | 53.85 ±0.46 | 22.15 ±0.35 | 37.67 ±0.51 | 60.48 ±0.75 | 78.35 ±0.78 |
| MK | 8.47 ±0.32 | 25.25 ±0.58 | 46.98 ±0.37 | 57.92 ±0.74 | 27.21 ±0.52 | 35.81 ±0.43 | 56.58 ±0.82 | 80.52 ±1.02 |
| CA | 5.11 ±0.08 | 16.58 ±0.39 | 37.59 ±0.65 | 51.50 ±0.80 | 24.49 ±0.07 | 32.89 ±0.28 | 41.73 ±0.31 | 60.07 ±0.96 |

Table 7. Compressive strength data (MPa) of control and addition mortars cured at 5 and 20° C.

| | 5° C | | | 20° C | | |
|------------|--------|--------|----------------|--------|--------|----------------|
| | a | b | R ² | a | b | R ² |
| CON | 3.279 | 11.623 | 0.986 | 20.238 | 9.6293 | 0.993 |
| FCC | 11.429 | 13.834 | 0.953 | 24.616 | 16.778 | 0.987 |
| MK | 12.693 | 14.712 | 0.952 | 25.872 | 16.183 | 0.997 |
| CA | 6.668 | 14.074 | 0.985 | 24.411 | 10.306 | 0.984 |

Table 8. Coefficients for adjusted logarithmic equations of control and addition mortars

cured at 5 and 20° C