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Mechanical treatment of fly ashes
Part IV. Strength development of ground fly ash-cement mortars cured at different temperatures

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Abstract

The influence of fly ash grinding on the strength development of fly ash/cement mortars cured at different temperatures was studied. A significant increase of compressive strength (R_c) for fly ash mortars was found at early age (3 days) when curing temperature is raised. However, the highest R_c values at 28 days' curing time were obtained for fly ash mortars cured at 40°C. The higher reactivity of fly ashes was found for ground fly ashes (GFA) compared to unground fly ash (T0). When R_c vs. curing time (in logarithmic scale) is represented, the slope values from regression analysis data obtained for the 20°C curing temperature series and origin ordinate from regression analysis data obtained for the 40°C or 60°C curing temperature series become good parameters for evaluating pozzolanic activity. An equivalence among R_c values obtained at curing temperatures of 20°C and 40°C and different curing times was proposed from strength gain data for mortars containing 15%, 30%, 45% and 60% replacing percentages. Finally, the compressive (R_c) and flexural (R_f) strength values for early ages and different curing temperatures were measured. A mathematical model has been proposed for the mechanical properties at early age of mortars containing fly ashes in 15–60% replacement range and cured in 20–80°C range.

Keywords: Curing; Grinding; Thermal treatment; Compressive strength; Fly ash

1. Introduction

It is well-known that fly ash concrete shows important strength gain as a consequence of increasing the curing temperature, whereas cement Portland concrete shows an increase in strength at early ages but a significant decrease is observed for mature concrete [1,2]. On the other hand, several upgrading procedures for fly ashes have been reported [3–12]. However, very few studies about the influence of processed fly ashes on the mechanical strength of mortar or concrete cured at different temperatures have been carried out [12,13].

Obviously, the pozzolanic reaction rate of fly ashes towards hydrated lime increases with temperature [14] and kinetics of this reaction is influenced by curing temperature [15]. The mechanical properties of fly ash concrete depend on several interesting parameters, for example, the microstructure of the hydrated products [16] and the nature of colloidal and crystalline products formed in various conditions (curing time, relative humidity, curing temperature, etc.). Moreover, the chemical, physical, morphological and mineralogical parameters of fly ashes have a decisive influence on pozzolanic reaction, microstructure and nature of the hydrated products.

The scope of this investigation was to conduct an in-depth analysis of the influence of fly ash grinding on the strength of fly ash-cement mortars cured at different temperatures, as part of a complete study on the use of ground fly ashes (GFA) in concrete [17–19].

2. Experimental

A low-calcium fly ash (T0) was used for preparing GFA samples (T10, T40 and T60, corresponding to 10, 40 and 60 min of grinding time). The grinding procedure, physical

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Table 1
Compressive strength values (R_c in MPa) for 30:70 fly ash/cement mortars cured at 20°C, 40°C, 60°C and 80°C for 3, 7, 14 and 28 days

T (°C)	Binder	3 Days	7 Days	14 Days	28 Days	*T* (°C)	Binder	3 Days	7 Days	14 Days	28 Days
20	C	28.09	35.36	38.57	46.25	40	C	31.56	37.69	43.05	46.17
20	C/T0	15.91	21.42	26.49	34.35	40	C/T0	24.50	37.65	46.24	47.32
20	C/T10	17.31	23.69	29.17	38.33	40	C/T10	26.95	39.52	51.63	49.50
20	C/T40	18.19	25.21	32.18	41.55	40	C/T40	30.30	41.16	52.81	51.48
20	C/T60	18.20	25.94	33.06	42.98	40	C/T60	31.15	41.65	54.54	51.17
60	C	33.81	39.79	41.75	48.71	80	C	34.53	41.67	43.54	48.13
60	C/T0	28.91	34.79	36.26	37.24	80	C/T0	32.16	33.03	34.57	31.90
60	C/T10	30.13	38.12	36.95	36.84	80	C/T10	33.06	34.14	35.02	36.41
60	C/T40	34.59	36.95	41.06	38.02	80	C/T40	35.58	38.25	38.65	37.69
60	C/T60	35.36	37.14	40.87	39.00	80	C/T60	35.65	37.74	38.25	37.92

properties and mineralogical and chemical compositions of fly ashes have already been reported [17,18]. The cement used for preparing mortars [12] was commercial Spanish cement II-F/35A, which consists of ASTM Type I Portland cement blended with finely ground limestone (13% by mass).

Mortars were prepared by mixing 450 g of this cement, 1350 g of fine aggregate (natural sand, 2.93 modulus fineness) and 200 ml of water. Mortars containing fly ashes were prepared replacing in weight 15%, 30%, 45 and 60% of cement by original (T0) or GFA (T10, T40 or T60) fly ashes. Mortar mixtures were put in a mold for obtaining three $16 \times 4 \times 4$ cm^3 specimens, which were stored in a moisture room (20±1°C) for 24 h; after demoulding, specimens were stored under water at experience temperature until test age. Flexural procedure was a center point load and then the two portions were tested in compression.

The cement for preparing pastes was an ASTM type I Portland cement, which presented the following chemical composition: 20.21% SiO_2, 4.94% Al_2O_3, 2.85% Fe_2O_3, 62.87% CaO, 1.05% MgO, 3.54% SO_3, 0.95% K_2O, 0.10% Na_2O, 3.02% loss on ignition and 0.95% insoluble residue. The original fly ash (T0) presented the following chemical and physical parameters: 41.4% SiO_2, 26.2% Al_2O_3, 16.0% Fe_2O_3, 6.1% CaO, 1.1% MgO, 0. 5% K_2O, 0.1% Na_2O, 2.44% loss on ignition, 2810 cm^2/g Blaine fineness and 2.44 specific gravity.

3. Results and discussion

A first batch of specimens were prepared as follows: mortars were composed of sand, water and binder in a 3:0.5:1 ratio, being binder cement or a mixture of cement and fly ash. Prismatic specimens ($160 \times 40 \times 40$ mm) were cured for the first 24 h at 20°C and, after demoulding, immersed in water at a given temperature. The selected curing temperatures were 20°C, 40°C, 60°C and 80°C, in order to study the different hydration kinetics and their consequence on strength development. Mortars containing a 30:70 fly ash/cement ratio were cured at 20°C, 40°C, 60°C and 80°C and compressive and flexural strength were tested at 3, 7, 14 and 28 days age. A series of specimens were prepared for each fly ash type: original fly ash (T0) and GFA (T10, T40 and T60). Table 1 summarizes the compressive strength data for this series. As expected, in general, the compressive strength values of control mortar and fly ash mortars increase progressively with curing time. In order to establish good correlation between compressive strength and curing time

Table 2
Analysis regression parameters for linear correlations according to Eq. (1) for fly ash/cement mortars with 30% replacement percentage

T (°C)	Binder	%	*a*	*b*	*R*	*T* (°C)	Binder	%	*a*	*b*	*R*
20	C	–	19.486	17.961	0.990	40	C	–	24.575	15.367	0.994
20	C/T0	30	6.235	18.703	0.990	40	C/T0	30	15.136	24.305	0.956
20	C/T10	30	6.344	21.344	0.989	40	C/T10	30	17.720	25.162	0.926
20	C/T40	30	5.914	23.873	0.993	40	C/T40	30	20.777	23.661	0.937
20	C/T60	30	5.324	25.254	0.994	40	C/T60	30	22.125	22.988	0.907
60	C	–	26.802	14.520	0.981	80	C	–	28.862	13.388	0.983
60	C/T0	30	26.074	8.403	0.934	80	C/T0	30	32.602	0.319	0.110
60	C/T10	30	29.423	6.218	0.710	80	C/T10	30	31.339	3.390	0.993
60	C/T40	30	33.162	4.590	0.710	80	C/T40	30	35.755	2.214	0.672
60	C/T60	30	33.570	4.620	0.807	80	C/T60	30	35.083	2.357	0.830

for the curing period of 3–28 days, the linear relationships were calculated [19] as follows:

$$R_c = a + b\ \log_{10} t \quad (1)$$

where R_c is the compressive strength in MPa, t is curing time in days, and a and b are constants for a given fly ash type and curing temperature. The linear regression data for prepared series of specimens are given in Table 2.

Fig. 1a and b plots the compressive strength/curing time linear regressions for T0 and T60 fly ash mortars, respectively, with a 30% replacing percentage. In these figures, a very important increase of compressive strength with curing time for 20°C and 40°C can be observed, whereas the increase is less important when curing temperature is raised to 60°C and 80°C. In this manner, the compressive strength of fly ash mortars at an early age (3 days) increases with curing temperature, whereas the highest compressive strength values were found for 40°C at the age of 7, 14 and 28 days. When the R_c values of mortars containing fly ashes are compared (see Fig. 1a and b), it may be noted that mortars made with GFA showed higher R_c values than mortars that contained original fly ash (T0). Moreover, greater R_c values were obtained when fly ash grinding time was increased, although differences between mortars containing T40 and T60 fly ashes were not much significant.

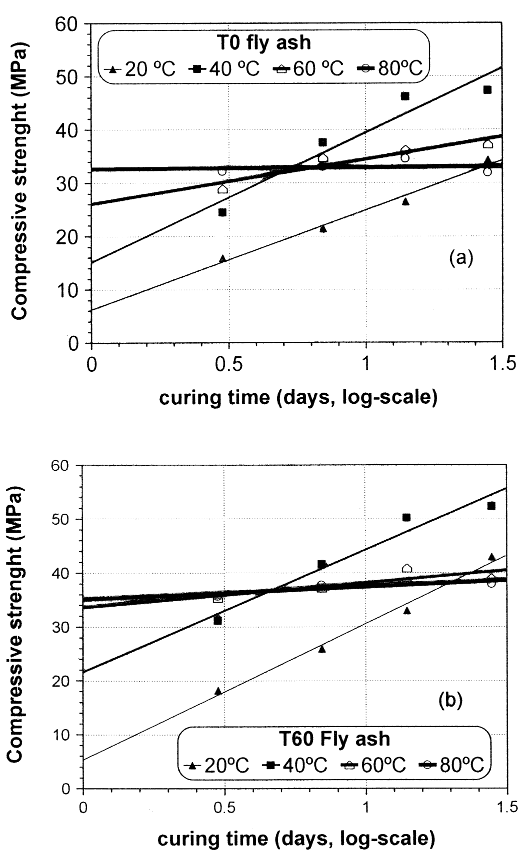

Fig. 1. Compressive strength/curing time linear regressions for fly ash/cement mortars with 30% replacement for different curing temperatures: (a) T0 fly ash; (b) T60 fly ash.

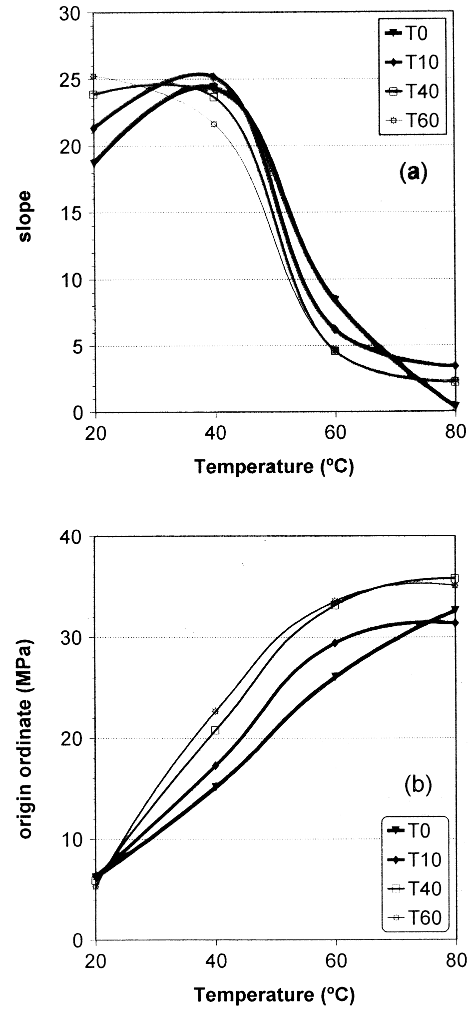

Fig. 2. Correlation between regression data from Eq. (1) and curing temperature for cement/fly ash mortars: (a) slope of regression line; (b) origin ordinate of regression line.

From the analysis regression data for 30% fly ash replacement mortars, two interesting observations can be made. First, when the slope of these regression lines are plotted vs. curing temperature (Fig. 2a), a good correlation between fly ash reactivity and the value of the slope at 20°C was found: T60 GFA presented the highest compressive strength and T0 the lowest one. So, the slope of the linear regression for fly ash mortars cured at 20°C becomes a good parameter for evaluating pozzolanic activity. For the 40°C and 60°C data, values tend to change over: this observation suggests that the increase of curing temperature promotes the pozzolanic activity of fly ashes and high compressive strength values are obtained at very early age, and, consequently, increase in strength is limited, specially for highest reactive fly ashes. The slope values for 80°C are very

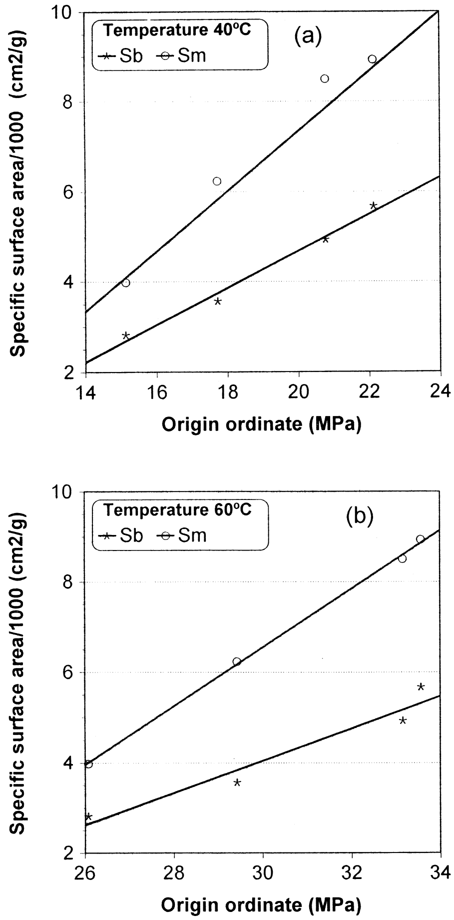

Fig. 3. Correlations between specific surface area values (S_b, Blaine method; S_m, obtained from granulometric data) and origin ordinate values from the regression data for fly ash/cement mortars cured at: (a) 40°C; (b) 60°C.

low due to the rapid development of strength and no conclusion can be established.

In second place, when the origin ordinate of these regression lines are plotted vs. curing temperature (Fig. 2b), additional information about the reactivity of fly ashes may be obtained. Origin ordinate should be assumed as the extrapolated compressive strength value for 1 day, and, obviously, this value increases with temperature. At 20°C, the origin ordinate values were similar for all tested fly ashes, indicating that the addition acts as an inert material. However, at 40°C and 60°C — where fly ash pozzolanity is achieved — a good correlation between origin ordinate and type of fly ash can be proposed: T0 fly ash shows lowest pozzolanic activity, and reactivity increases with grinding. Obviously, these differences on the reactivity of fly ashes are related mainly with fineness, a fact that can be seen in Fig. 3. A linear relationship has been obtained when specific surface area [19] was plotted vs. the ordinate origin values for experiences carried out at 40°C (Fig. 3a) and 60°C (Fig. 3b). These correlations have been established for specific surface area values obtained from granulometric data (S_m) and for Blaine method (S_b).

A second batch of mortar specimens, prepared with 15:85, 45:55 and 60:40 fly ash/cement ratios were cured at 20°C and 40°C, in order to obtain information about the behaviour of mortars cured at different temperatures and containing different fly ash/cement ratios (see Table 3 for compressive strength data). Compressive strength values fit to Eq. (1) linear relationship. Linear regression data for this second batch of specimens are listed in Table 4.

It can be denoted that the increase in curing temperature yielded a pronounced increase in compressive strength for all replacing percentages. The influence of the fly ash replacement level would be better studied by means of calculating the strength gain (SG_i), according to the already reported Eq. (2) [13,19]:

$$SG_i = R_i - \left[R_o \frac{w_c}{w_c + w_{fa}}\right] \quad (2)$$

where R_i is the compressive strength for a given replacing percentage and curing time, R_o the compressive strength for control mortar at the same age, and w_c and w_{fa} are the weight of cement and the weight of fly ash, respectively.

Fig. 4 shows the SG values for T0 and T60 fly ash containing mortars cured at 20°C and 40°C. The behaviour of specimens cured at 20°C was already reported [19]. In

Table 3
Compressive strength values (R_c in MPa) for 15:85, 45:55 and 60:40 fly ash/cement mortars cured at 20°C and 40°C for 3, 7, 14 and 28 days

T (°C)	Fly ash (%)	3 Days	7 Days	14 Days	28 Days	T (°C)	Fly ash (%)	3 Days	7 Days	14 Days	28 Days
20	T0 (15)	20.00	30.84	35.83	40.53	40	T0 (15)	29.18	40.19	45.04	46.29
20	T0 (45)	9.30	12.30	16.90	24.00	40	T0 (45)	21.72	34.25	41.96	45.55
20	T0 (60)	6.01	7.02	11.21	17.12	40	T0 (60)	9.33	19.35	22.22	27.48
20	T10 (15)	20.58	31.59	37.30	42.98	40	T10 (15)	31.69	40.63	46.12	50.20
20	T10 (45)	10.30	16.00	23.31	30.02	40	T10 (45)	24.09	36.90	42.49	48.16
20	T10 (60)	6.54	8.37	12.21	19.99	40	T10 (60)	11.50	21.99	27.78	30.75
20	T40 (15)	21.53	33.48	38.42	44.74	40	T40 (15)	30.61	41.65	46.98	50.20
20	T40 (45)	11.24	17.40	26.41	33.02	40	T40 (45)	29.02	41.01	47.65	49.03
20	T40 (60)	7.94	10.60	15.97	23.56	40	T40 (60)	15.00	27.48	31.56	34.48
20	T60 (15)	21.15	33.20	37.08	44.30	40	T60 (15)	31.85	43.41	47.22	51.59
20	T60 (45)	11.60	16.10	26.61	33.52	40	T60 (45)	30.89	42.29	48.00	50.68
20	T60 (60)	7.66	11.20	16.70	25.36	40	T60 (60)	16.70	27.39	31.38	37.85

Table 4
Analysis regression parameters for linear correlations according to Eq. (1) for fly ash/cement mortars with 15%, 45% and 60% replacement percentages

T (°C)	Binder	%	a	b	R	T (°C)	Binder	%	a	b	R
20	C/T0	15	11.326	20.916	0.986	40	C/T0	15	22.768	17.783	0.948
20	C/T0	45	0.942	15.000	0.973	40	C/T0	45	11.501	24.896	0.980
20	C/T0	60	−0.903	11.486	0.944	40	C/T0	60	1.955	18.020	0.981
20	C/T10	15	10.742	22.854	0.992	40	C/T10	15	23.439	19.124	0.991
20	C/T10	45	−0.243	20.578	0.995	40	C/T10	45	13.982	24.445	0.985
20	C/T10	60	−1.482	13.546	0.943	40	C/T10	60	3.435	19.992	0.977
20	C/T40	15	11.638	23.400	0.988	40	C/T40	15	22.600	20.187	0.975
20	C/T40	45	−0.527	23.024	0.994	40	C/T40	45	21.069	21.053	0.956
20	C/T40	60	−1.203	16.061	0.967	40	C/T40	60	7.772	19.776	0.957
20	C/T60	15	11.410	23.009	0.986	40	C/T60	15	24.072	19.865	0.974
20	C/T60	45	−1.081	23.529	0.983	40	C/T60	45	22.884	20.315	0.970
20	C/T60	60	−2.432	18.044	0.971	40	C/T60	60	7.633	21.144	0.989

general, compressive strength values obtained for the 40°C experience presented approximately the following agreement with the corresponding compressive strength values for 20°C experience:

3 days at 40°C ≡ 14–90 days at 20°C,
7 days at 40°C ≡ 60–180 days at 20°C,
14 days at 40°C ≡ 90–180 days at 20°C,
28 days at 40°C ≡ 180–365 days at 20°C.

Again, as reported [19], mortars containing 30% and 45% replacing percentages yielded optimum SG values, and T60 became clearly much more reactive than T0 fly ash for all replacements studied.

Finally, a third batch of specimens was performed in order to study the early-age compressive and flexural strength developments of cement mortars containing fly ashes, the influence of fly ash grinding and the influence of curing temperature. In this manner, prismatic specimens were prepared for several replacing percentages (15%, 30%, 45% and 60%) and, after storing at 20°C for 24 h, they were submerged in water at different temperatures (20°C, 40°C, 60°C and 80°C) for 48 h. The compressive and flexural strengths at this age were

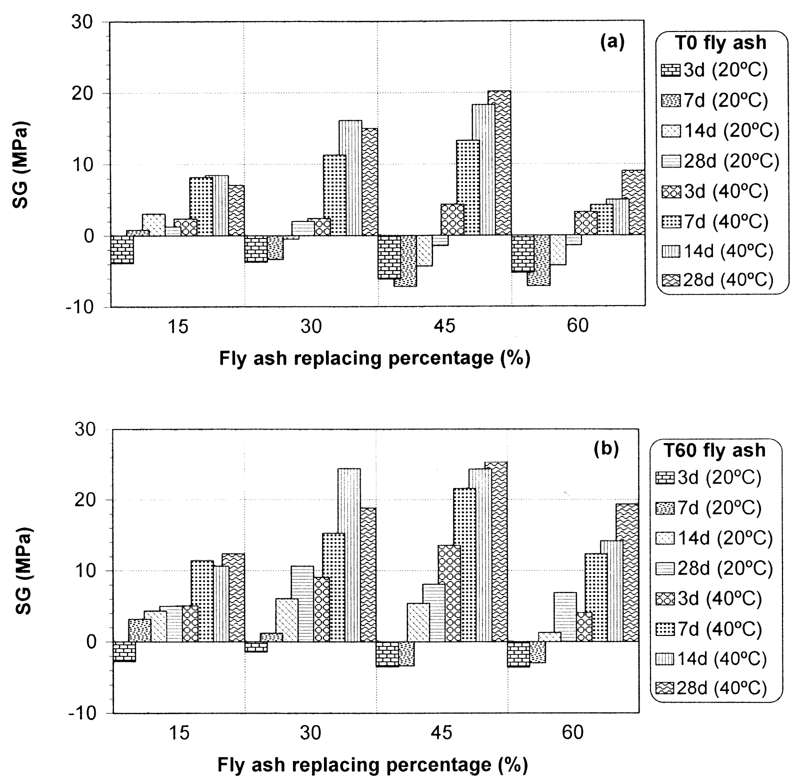

Fig. 4. Compressive strength gain (SG_i) for fly ash mortars cured at 20°C and 40°C and different curing times: (a) T0 fly ash; (b) T60 fly ash.

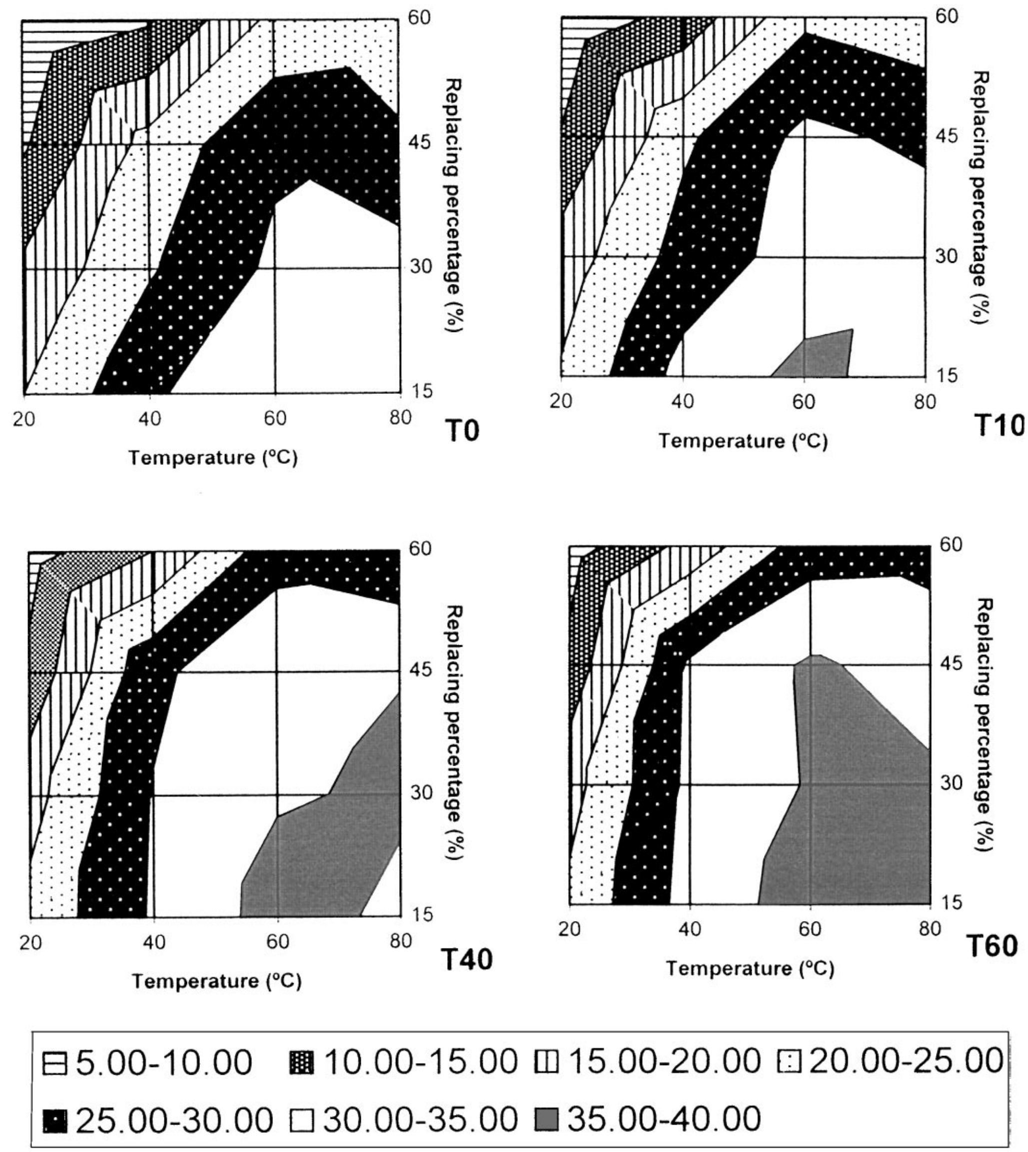

Fig. 5. Contour graphical plots for compressive strength of fly ash mortars cured at 20°C for 24 h and submerged in water at different temperatures (20°C to 80°C) for 48 h.

measured for mortars prepared using T0 and T10, T40 and T60 GFAs.

The contour graphical plotting of compressive strength values with fly ash replacing percentage and curing temperature are showed in Fig. 5 for all studied fly ashes. It can be observed that, in general, the highest compressive strength values are found for higher curing temperatures (60–80°C) and lower replacing percentages (15–30%). Apparently, 60°C curing temperature showed the best compressive strength values for all replacing percentages, while T40 and T60 fly ash containing mortars yielded the highest rates. Thus, nearly all of T40 and T60 fly ash mortars cured between 40°C and 80°C and containing 15–45% replacing percentage yielded compressive strength greater than 30 MPa, whereas an important part of T0 fly ash mortars for the same curing temperature range and replacing percentage range yielded compressive strength values of less than 30 MPa.

Since the study concentrates on the dependence of compressive strength with curing temperature of fly ash mortars at early ages, a few alternative equations

Table 5
Coefficients (*a*, *b*, *c*, *d* and *e*) and standard deviation (σ) of Eq. (3) for all tested fly ashes

Fly ash	*a*	*b*	*c*	*d*	*e*	σ
T0	39.55	−0.403	−0.00567	68.41	0.00169	2.3
T10	40.80	−0.419	−0.00727	62.87	0.00219	2.5
T40	40.98	−0.396	−0.00768	62.46	0.00302	3.0
T60	42.07	−0.383	−0.00866	61.85	0.00278	2.7

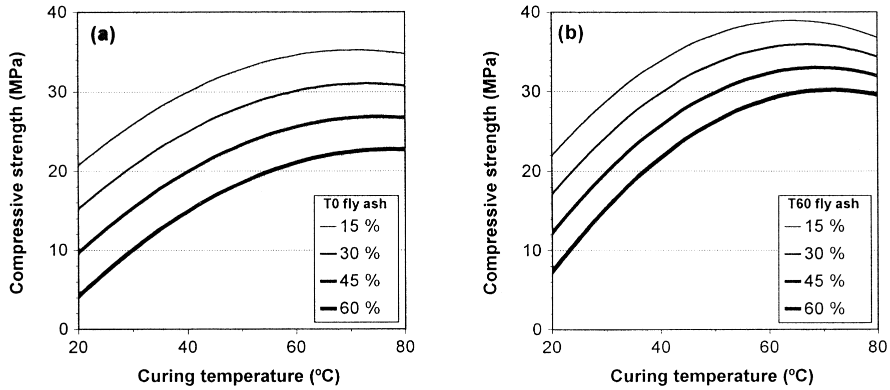

Fig. 6. Compressive strength values from Eq. (3) for: (a) T0 fly ash; (b) T60 fly ash mortars.

were tested for regression analysis. Finally, the following mathematical model with the minimal values of σ was selected:

$$R_c = a + b\varphi + c(T - d)^2 + e\varphi T \quad (3)$$

where R_c is the compressive strength (in MPa), φ is replacing percentage (15–60% range), T is curing temperature (20–80°C range), and a, b, c, d and e are the coefficients obtained by regression analysis from experimental data (see Table 5).

From Eq. (3) and coefficient values for T0 and T60 fly ash mortars, the compressive strength dependence with curing temperatures has been obtained for all studied replacing percentages, as can be seen in Fig. 6a

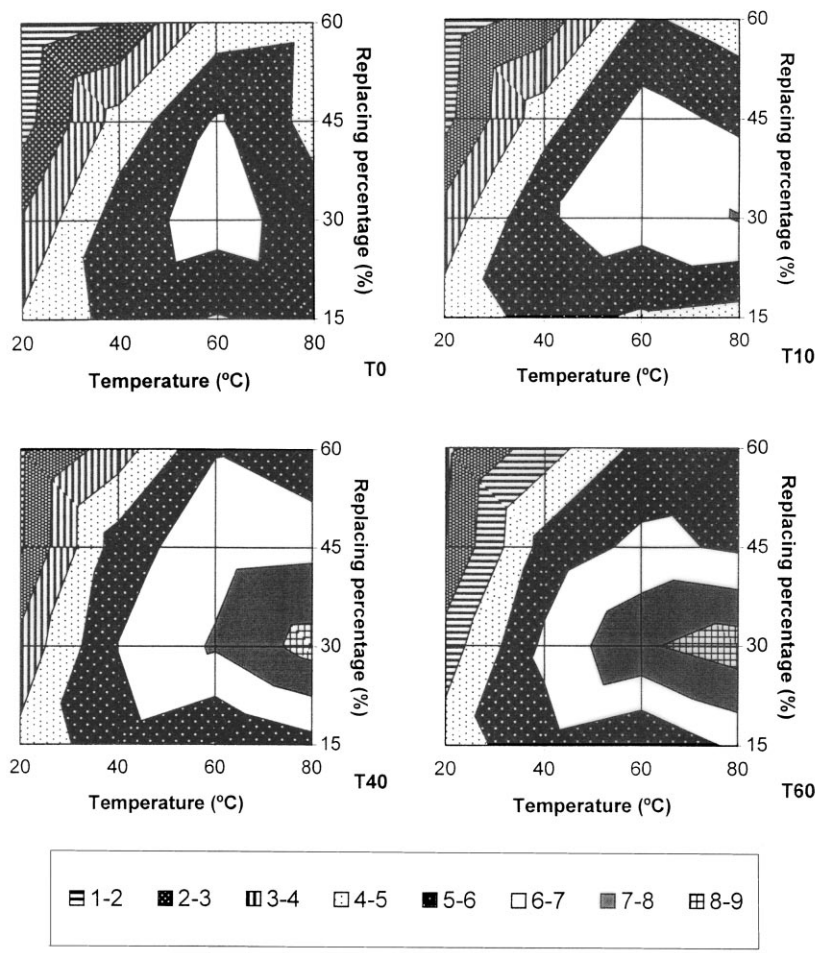

Fig. 7. Contour graphical plots for flexural strength of fly ash mortars cured at 20°C for 24 h and submerged in water at different temperatures (20°C to 80°C) for 48 h.

Table 6
Coefficients (α, β, γ, δ, ε and η) and standard deviation (σ) of Eq. (4) for all tested fly ashes

Fly ash	α	β	γ	δ	η	ε	σ
T0	5.114	−0.00138	−0.00142	51.78	3.560	0.00103	0.66
T10	4.579	−0.00238	−0.00126	49.63	16.60	0.00128	0.75
T40	5.475	−0.00242	−0.00125	60.58	22.79	0.000884	0.85
T60	5.912	−0.00226	−0.00115	64.29	21.72	0.000682	0.92

and b. It can be noted that optimum compressive strength values are dependent on type of fly ash and on replacing percentage. In this manner, the optimum R_c values for T60 fly ash mortars (Fig. 6b) are reached at slightly lower temperatures than T0 fly ash ones (Fig. 6a). On the other hand, higher curing temperature for yielding optimum R_c is required when fly ash replacing percentage increases. Even so, in all cases, 95% of the value of optimum R_c is reached by curing the specimens in the range 50–60°C, suggesting that the energy cost in extreme curing conditions (60–80°C) does not supply an important strength gain.

In the same way, the flexural strength values R_f for this third batch of specimens were also measured, and contour graphical plots are showed in Fig. 7. In this case, an evident increase in flexural strength is observed when fly ash is ground.

Two aspects may be noted: first, the highest R_f values for T0 fly ash mortars were obtained at a curing temperature of about 60°C, whereas for GFA fly ashes, they are obtained at higher temperatures (70–80°C). Second, optimum values were obtained for 30% replacing percentage. After testing a few alternative equations, the mathematical model proposed for flexural strength calculation takes into account this second point, and a parabolic term "$\beta(\varphi-\eta)^2$" substitutes the linear term "$\beta\varphi$," which appeared in Eq. (3). Thus, Eq. (4) was selected:

$$R_f = \alpha + \beta(\varphi - \eta)^2 + \gamma(T - \delta)^2 + \varepsilon\varphi T \quad (4)$$

The coefficients from regression analysis for fly ash mortars cured at different temperatures and with different replacing percentages are summarized in Table 6.

4. Conclusions

1. For 20°C and 40°C curing temperatures, an important increase of compressive strength (R_c) values was observed in the 3–28 days curing period. However, this increase was lower for 60°C curing temperature. No significant increase was obtained for the highest tested temperature (80°C) due to the rapid development of R_c in these conditions.

2. Good linear relationships were obtained between R_c values and the logarithm of curing time for fly ash/cement mortars cured at different temperatures.

3. The slope and ordinate origin of these regression lines (R_c vs. log curing time) became appropriate parameters for evaluating pozzolanic activity of GFAs and T0s. The highest slope value was found for mortars containing T60 fly ash cured at 20°C, whereas the highest values of ordinate origin for the same type of mortars were found for series cured at 40°C and 60°C, suggesting the greater pozzolanic activity of T60 compared to other fly ash samples.

4. The curing temperature increase yielded an important increase in compressive strength for fly ash replacing percentages from 15% to 60%.

5. By means of calculating the compressive strength gain (SG), an equivalence of the curing time for mortars cured at 20°C and 40°C has been established.

6. The dependence of flexural and compressive strength with curing time at early age for mortars containing different replacing percentages of fly ash has been studied. Two mathematical models have been proposed and the coefficients of the equations have been calculated for all tested fly ashes.

7. The calculated optimum compressive strength values for T60 fly ash mortars have been obtained for lower curing temperatures that T0 fly ash mortars, indicating, again, the higher reactivity of GFA from pozzolanic activity point of view.

8. Optimum flexural strength (R_f) values were obtained for 30% fly ash replacing percentage, finding highest R_f values for GFA mortars cured at 80°C; whereas T0 mortars showed highest R_f values cured at 60°C.

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