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MECHANICAL TREATMENTS OF FLY ASHES. PART III: STUDIES ON STRENGTH DEVELOPMENT OF GROUND FLY ASHES (GFA) - CEMENT MORTARS

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ABSTRACT

Early and medium-term strength developments for mortars containing ground fly ashes (GFA) were studied and compared with the behaviour of mortars containing non-mechanically treated fly ash. Linear correlations between mechanical properties and the logarithm of curing time for mortars containing 15-60% fly ash replacing percentages were established. Compressive Strength Gain (SGi) and Pozzolanic Effectiveness Ratio (PER) were calculated, suggesting the increasing of pozzolanic activity with grinding of fly ash. A new mathematical model has been proposed for mechanical properties of mortars containing high replacing percentages and for a wide curing time range. Optimums for mechanical properties were calculated for mortars containing GFA. © *1997 Elsevier Science Ltd*

Introduction

The mechanical treatment by grinding of fly ashes alterates physical, mineralogical, morphological and chemical fly ash properties as have been recently reported (1,2). The properties of ground fly ash (GFA)/cement mixtures will be dependent on the degree of this mechanical treatment; so, GFA/cement mortars yield worse workability than non-ground fly ash/cement ones and good relationships between workability and specific surface area of GFA have been established (2). The properties of GFA will have an effect on hardened GFA/cement mixtures (3,4,5), due to, mainly, the increase of pozzolanic reaction rate and, probably, the alteration on the nature and distribution of colloidal and crystallized GFA/lime products.

On the other hand, values of fly ash/cement and water/binder ratio are essential on strength development of pastes, mortars and concretes containing fly ashes. Beneficiation of fly ashes by means several procedures (6,7,8) leads to obtain fly ash samples whose properties permits, in some cases, an optimization on above mentioned ratios.

In this paper, the influence of grinding time of original fly ash on GFA/cement mortars is studied: relationships between flexural and compressive strength and curing time, pozzolanic effectiveness ratio, strength gain and optimization of fly ash contents were determined.

Experimental

A low calcium fly ash (T0) was used for preparing GFA samples (T10, T40 and T60, corresponding to 10, 40 and 60 minutes of grinding time). Grinding procedure, physical properties and mineralogical and chemical compositions of fly ashes have already been reported (1,2).

Cement for preparing mortars 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 total cement by original (T0) of 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 hours; after demoulding, specimens were stored under water at 20 ± 1°C until test age (3, 7, 14, 28, 60, 90, 180 and 365 days). Flexural procedure was a center point load and then the portions were tested in compression.

Results and Discussion

Early and medium-term strength development. Compressive and flexural strength values were measured at early ages (3, 7, 14 and 28 days) and at medium-term ages (60, 90, 180 and 365 days. In order to establish good correlations between compressive strength and curing time several simple regression equations were tested; so, optimum linear relationships were calculated as follows:

$$R_c = a + b \log_{10} t$$

being R_c compressive strength, t curing time in days, $\log_{10}$ is base-10 logarithm and a and b constants for a given fly ash and replacing percentage. Linear regression data for used fly ashes and 15–60 replacing percentages are summarized in Table 1.

It can be observed, in general, that slopes (b parameter) of the linear relationships increase with fineness and greatest b values were found for T60 containing mortars; this fact indicates that the effect on strength development (lime fixation and hydration process) are more important for finest GFA. Moreover, differences among used fly ashes become more noticeable for 30 and 45% replacing percentages.

Figures 1 and 2 show linear correlations for T0 and T60 fly ash containing mortars and revealed that the slope of lines for 30 and 45% replacing percentages are greater than for 15 and 60% ones, specially for T60 containing mortars; these differences suggest that contribution to strength development depends strongly on fly ash/cement ratio. So, for low fly ash replacing percentages (15%), released lime from cement hydration process is high and reaction rate towards glassy fraction of fly ash particles is dependent of fly ash content; on the other hand, when mortars are performed with high fly ash replacing percentages, available lime becomes low for reacting towards glassy fly ash particles. Therefore, intermediate replacing percentages (30 and 45%) are the best for fly ash reactivity comparisons. Figure 3 shows correlations between slope and specific surface area of fly ashes; this figure confirms that dependence for 30 and 45% replacing percentages are much more perceptible, whereas 15% and 60% replacing percentages were almost independent of fly ash specific surface area.

TABLE 1
Analysis Regression Parameters§ for Linear Correlations $R_c = a + b \log_{10} t$

Replacing fly ash	Replacing percentage (%) 15	30	45	60
T0	a = 12.587	a = 4.810	a = −3.400	a = −5.747
	b = 19.142	b = 20.734	b = 19.774	b = 16.095
	R = 0.982	R = 0.994	R = 0.982	R = 0.0968
T10	a = 12.365	a = 5.374	a = −2.565	a = −4.891
	b = 20.616	b = 22.864	b = 23.162	b = 16.992
	R = 0.979	R = 0.994	R = 0.997	R = 0.985
T40	a = 14.357	a = 6 354	a = −1.641	a = −2.333
	b = 20.289	b = 23.940	b = 24.585	b = 17.260
	R = 0.978	R = 0.992	R = 0.998	R = 0.993
T60	a = 13.867	a = 6.265	a = −2.407	a = −2.443
	b = 20.350	b = 24.614	b = 25.457	b = 18.095
	R = 0.978	R = 0.996	R = 0.993	R = 0.993

§Linear regression data for control mortar: a = 22.471; b = 14.790; R = 0.988

Correlations between flexural strength values R_f and curing time also will be established, in the same way than before:

$$R_f = \alpha + \beta \log_{10} t$$

Again, slopes of the curves are greater for mortars containing 30 and 45% replacing percentages (see Table 2) although curves for 60% replacing percentage also show high slope

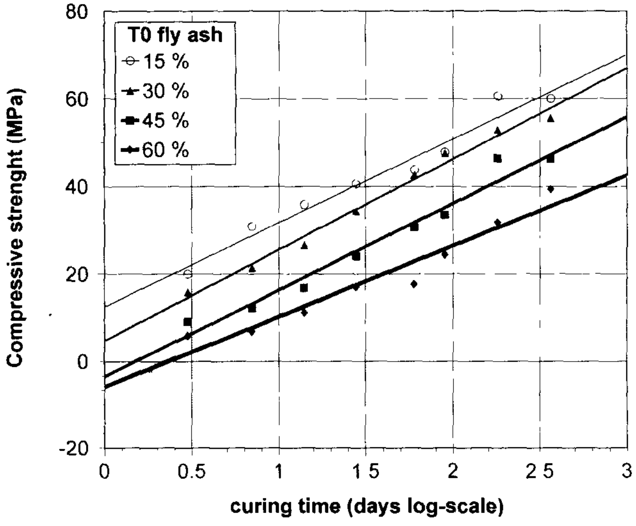

FIG. 1.
Linear Relationships Between Compressive Strength and Curing Time for T0 Fly Ash Containing Mortars.

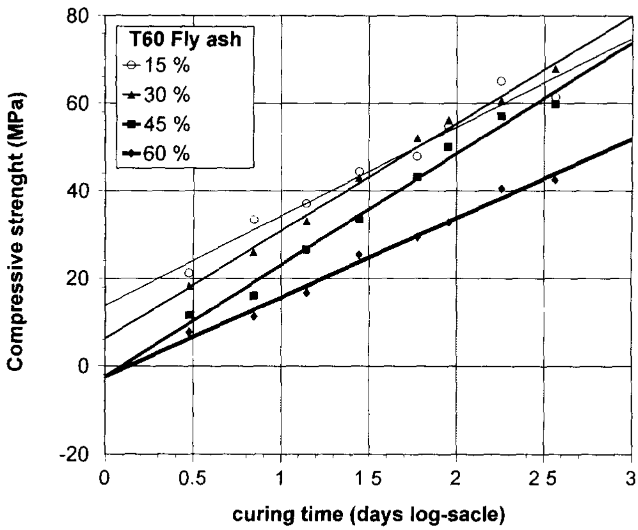

FIG. 2.
Linear Relationships Between Compressive Strength and Curing Time for T60 Fly Ash Containing Mortars.

values. Figures 4 and 5 show linear relationships for mortars containing T0 and T60 fly ashes. In this case no clear tendency with fineness was observed among fly ashes, and slopes are similar for a given replacing percentage (Figure 6).

<u>Pozzolanic activity: strength gain and pozzolanic effectiveness ratio</u>. With the aim of determining the contribution of fly ashes to strength development due to pozzolanic mechanisms (lime fixation and hydration processes), two parameters were calculated. The first of them is the compressive strength gain (SG_t) for a given replacing percentage and curing time, which can be calculated as follows (9):

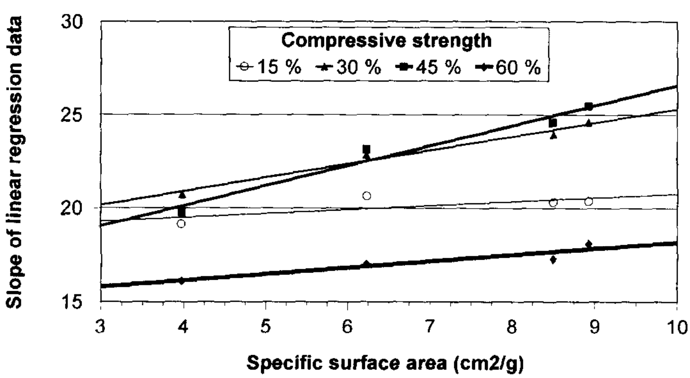

FIG. 3.
Dependence of Slope of Linear Regression Data for Compressive Strength and Specific Surface Area of Fly Ashes.

TABLE 2
Analysis Regression Parameters§§ for Linear Correlations $R_f = \alpha + \beta \log_{10} t$

	Replacing percentage (%)			
Replacing fly ash	15	30	45	60
T0	$\alpha = 3.925$	$\alpha = 1.448$	$\alpha = -0.483$	$\alpha = -0.922$
	$\beta = 2.073$	$\beta = 3.409$	$\beta = 4.090$	$\beta = 3.522$
	R = 0.929	R = 0.988	R = 0.990	R = 0.986
T10	$\alpha = 3.830$	$\alpha = 1.328$	$\alpha = 0.001$	$\alpha = -0.361$
	$\beta = 2.282$	$\beta = 3.934$	$\beta = 4.224$	$\beta = 3.354$
	R = 0.972	R = 0.991	R = 0.992	R = 0.965
T40	$\alpha = 4.081$	$\alpha = 1.218$	$\alpha = 0.289$	$\alpha = 0.180$
	$\beta = 2.138$	$\beta = 4.258$	$\beta = 4.184$	$\beta = 3.408$
	R = 0.925	R = 0.996	R = 0.985	R = 0.959
T60	$\alpha = 4.429$	$\alpha = 1.629$	$\alpha = -0.093$	$\alpha = 0.238$
	$\beta = 1.780$	$\beta = 3.991$	$\beta = 4.311$	$\beta = 3.344$
	R = 0.879	R = 0.987	R = 0.987	R = 0.963

§§Linear regression data for control mortar: $\alpha = 4.889$; $\beta = 1.489$; R = 0.939

$$SG_i = R_i - \left[R_0 \frac{w_c}{w_c + w_{fa}} \right]$$

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

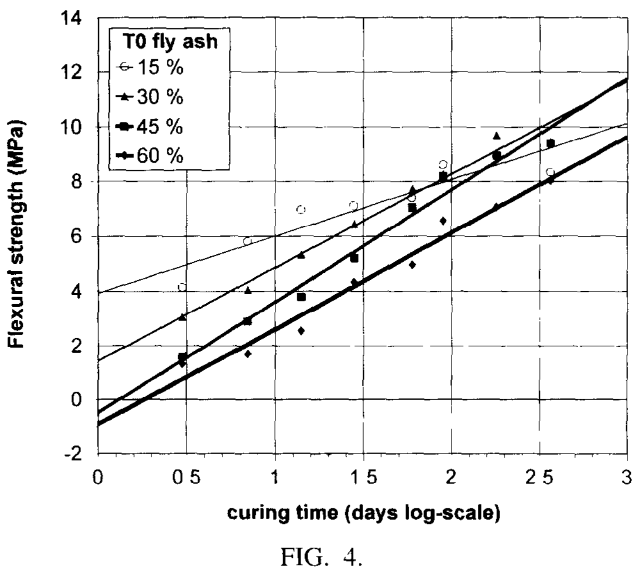

FIG. 4.
Linear Relationships Between Flexural Strength and Curing Time for T0 Fly Ash Containing Mortars.

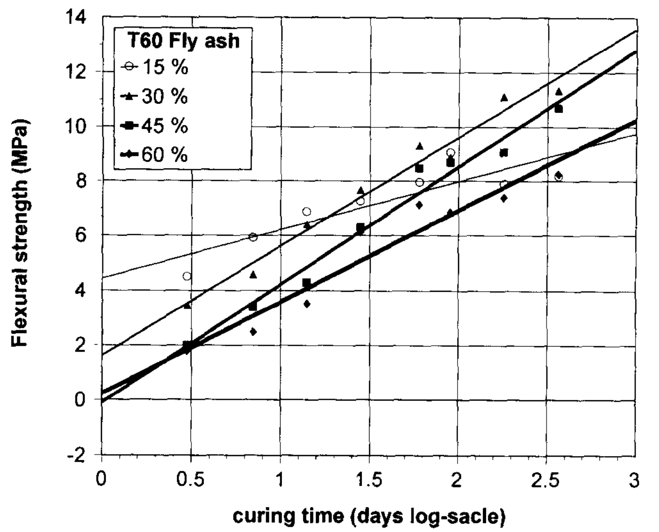

FIG. 5.
Linear Relationships Between Flexural Strength and Curing Time for T60 Fly Ash Containing Mortars.

Contour graphical representation of SG_t values with replacing percentages and curing time dependence are showed in Figure 7. These contour graphics allow to see that, generally, for early ages (≤14 days) lowest percentage yields highest SG_t values, except for T60 containing mortars (Figure 3d), where maximum value at 14 days curing time is near to 30% replacing percentage. For longer curing times, a shift to higher percentages for optimum SG_t values is noticed, in such a way that these optimum values appear between 30 and 45% replacing percentages.

The second parameter calculated in order to estimate pozzolanic activity of GFA is the pozzolanic effectiveness ratio (PER). Based on Feret's law (10) which establish a relation-

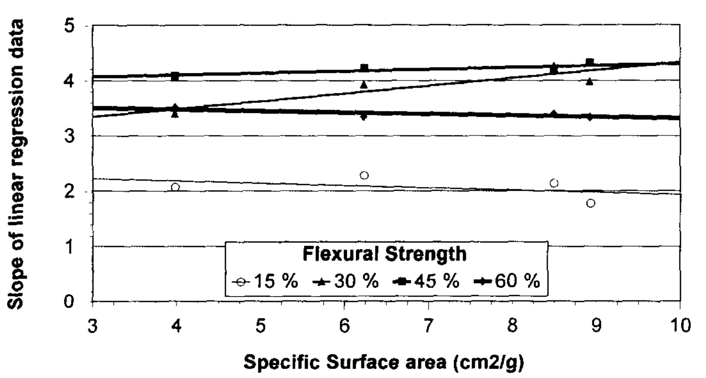

FIG. 6.
Dependence of Slope of Linear Regression Data for Flexural Strength and Specific Surface Area of Fly Ashes.

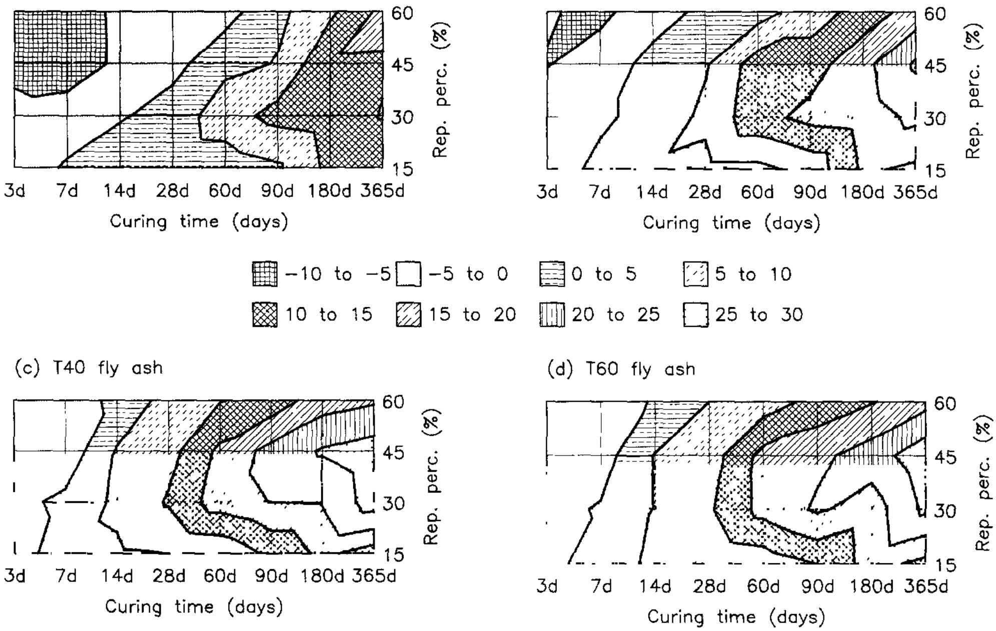

FIG. 7.

Contour Graphics of Replacing Percentage and Curing Time Dependence of SG_t Values: a) T0; b) T10; c) T40; d) T60.

ship between strength (f) and absolute volumes of water (w) cement (c) and air (a), Berry et al. (7,11) proposed the PER parameter as the value of S_f/F:

$$f = K\left[\frac{c}{c + w + a}\right]^2 \text{ (Feret's Law)}$$

S_f is the experimental strength ratio s_t/s_o, where s_t is the compressive strength at curing time t for fly ash containing mortar and s_o is the compressive strength at the same curing time for control mortar; and F is the Feret ratio (air content negligible):

$$F = \frac{f_1}{f_0} = \left[\frac{c_0 + w_0}{c_1 + w_1} * \frac{c_1}{c_0}\right]^2$$

that is to say the calculated strength ratio based on mixture composition.

Figure 8 shows PER values for mortar mixtures at 28, 90 and 365 days curing times. Four facts could be noticed. Firstly, for all cases, PER values are greater than unit, and, consequently, important pozzolanic activity of fly ashes can be assumed. In second place, PER increase with curing time, indicating that pozzolanic reaction is being extended. In third place, PER increase with fly ash replacing percentage; Berry et al. (7) and Ramyar and Erdogan (12) also found this behaviour. However, when volume of fly ash approaches that of the cement volume PER was not assumed as a good measurement of fly ash contribution to strength (12) because increasing of water demand. In our case, mortars have been prepared with the same water/cementicious material ratio, and, even, prepared mortars with original

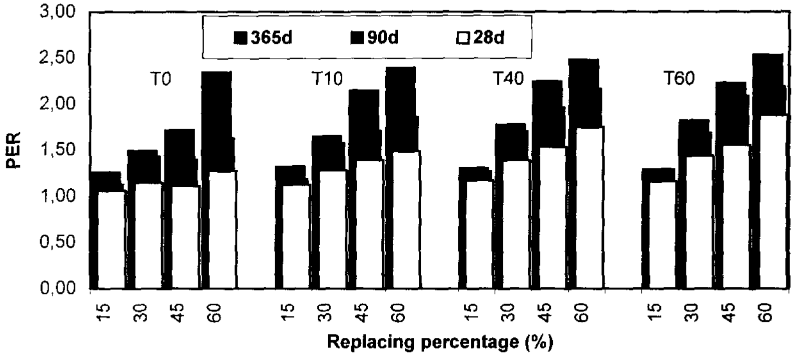

FIG. 8.
PER Values of Mortars at 28, 90 and 365 Days Curing Times.

T0 fly ash and GFA yielded better workability than control mortar (2). Therefore, at least, PER values could be considered as a qualitative indicator of pozzolanic activity. Finally, in fourth place, PER values increase with fineness of fly ashes, although 15% replacing percentage mortars show little differences. This behaviour is in good agreement with the increase of fly ash reactivity with fineness when chemical and mineralogical parameters are similar.

Optimization of fly ash quantity in GFA/cement systems. Mathematical modeling and optimizing fly ash quantities in fly ash/cement systems also have been carried out. Very recently, a mathematical model for mechanical characteristics was proposed by Fisang et al. (13); this mathematical model takes into account linear and higher order terms and interactions terms between fly ash quantity and curing time. The final model which was selected by these authors was the following equation:

$$R = b_1 + b_2\Phi + b_3t + b_4\Phi t + b_5\Phi^2 + b_6t^2 + b_7\Phi^2t + b_8\Phi t^2 \quad (1)$$

where R is the mechanical property (flexural or compressive strength) b_1–b_8 are the coefficients of the polynomial equation, Φ is the replacing percentage and t the curing time.

Fisang et al. (13) applied this model to mortar containing three different types of fly ashes and 15–30% replacing percentage range for early-age mortars (3–28 days), and good results were obtained. This model has been applied to our experimental data from mortars using GFA ashes, in the range 15–60% replacing percentages and also 3–28 days curing time period.

The coefficients on eq. 1 for both compressive and flexural strengths were calculated by regression analysis and Table 3 summarizes obtained data. Standard deviation, σ, for each regression analysis was determined as:

$$\sigma = \sqrt{\frac{\Sigma(R_i - R)^2}{N - n}} \quad (2)$$

where $(R_i - R)$ is the difference between measured and calculated sample characteristic, N is the number of different experiments designed and n is the number of coefficients of the proposed polynomial.

TABLE 3
Coefficients of the Eq. 1 for 3–28 Days Curing Time Period.

	Compressive Regression Data				Flexural Regression Data			
Coeff.	T0	T10	T40	T60	T0	T10	T40	T60
b_1	23.4077	20.53	22.65	22.57	4.745	4.557	4.418	5.068
b_2	−0.5355	−0.320	−0.412	−0.391	−0.122	−0.082	−0 0894	−0.1167
b_3	2.5151	2.532	2.425	2.229	0.482	0.394	0.482	0.408
b_4	−2.93E-2	1.71E-2	−3.80E-3	1.99E-3	−3.59E-3	−1.43E-3	−2.02E-3	−5.05E-4
b_5	3.77E-3	8.23E-4	1.79E-3	1.34E-3	9.63E-4	3.11E-4	5.39E-4	8.82E-4
b_6	−5.68E-2	−5.91E-2	−5.79E-2	−5.03E-2	−1.32E-2	−9.94E-3	−1.36E-2	−2.00E-2
b_7	−1.21E-4	−2.46E-4	−3.29E-4	−3.45E-4	−4.22E-5	−3.57E-5	−3.93E-5	−6.08E-5
b_8	1.02E-3	9.49E-4	7.65E-4	6.69E-4	2.24E-4	1.54E-4	2 02E-4	2.05E-4
σ	1.55	1.40	1.56	1.93	0.20	0.30	0 41	0.38

Regression analysis data summarized in Table 3 were obtained from N = 16 experiments and standard deviation σ (eq. 2) are similar or lower than obtained by Fisang et al. (13), but some coefficients (zero order and first order mainly) are very different (value and sign) than Fisang's ones. This fact can be attributed to Portland cement type used, the number of the experiments, the water/cementicious material ratio and the differences in the reactivity of fly ashes.

When model proposed in eq. 1 is applied to experimental data in 3–365 days period, no convergence in the regression analysis succeed.

So, an alternative mathematical model must be proposed. Assuming linear dependence of mechanical properties with the logarithm ($\log_{10} t$) of curing time (see first part of results and discussion of present paper), the following mathematical model will be studied:

$$R = \beta_1 + \beta_2\Phi + \beta_3(\log_{10} t) + \beta_4\Phi(\log_{10} t) + \beta_5\Phi^2 + \beta_6(\log_{10} t)^2 + \beta_7\Phi^2(\log_{10} t) + \beta_8\Phi(\log_{10} t)^2 \quad (3)$$

This proposed model (eq. 3) was tested for 3–28 days curing time period and coefficients for this polynomial equation are summarized in Table 4; standard deviations for compressive

TABLE 4
Coefficients of the Eq. 3 for 3–28 Days Curing Time Period.

	Compressive Regression Data				Flexural Regression Data			
Coeff.	T0	T10	T40	T60	T0	T10	T40	T60
β_1	18.1578	8.8120	26.7357	28.1592	4.2797	8.3470	2 6846	7.1096
β_2	−0.3157	−0.3975	−0.8259	−0.7828	−0.1831	−0.2231	−0 1461	−0.2425
β_3	49.8079	43.7397	42.0698	38.7827	10.8197	5.8148	12.4997	8.6636
β_4	−1.1094	−0.6609	−0.4118	−0.3761	−0.1819	−7.18E-2	−0 1808	−0.1280
β_5	8.30E-3	8.08E-3	1.291E-2	1 211E-2	3.34E-3	2 74E-3	3.24E-3	4.06E-3
β_6	−5.4065	−5.3936	−5.4519	−4.9466	−1.9412	−0.8880	−2.4997	−1.7183
β_7	1.44E-3	6.01E-3	8.26E-3	−8.59E-4	−1.06E-3	−1.37E-3	−1.14E-3	−1.71E-3
β_8	0.22758	0.2087	0.1993	0.2061	5.875E-2	4.256E-2	7.07E-2	6 41E-2
σ	1.17	0.95	1.09	1.55	0.19	0.29	0.33	0.39

TABLE 5
Coefficients of the Eq. 3 for 3–365 Days Curing Time Period.

	Compressive Regression Data				Flexural Regression Data			
Coeff.	T0	T10	T40	T60	T0	T10	T40	T60
β_1	42.7312	47.0682	49.1444	51.2546	12.9099	15.2972	15.2951	16.5763
β_2	−1.6791	−1.7252	−2.0040	−2.0897	−0.5587	−0.6103	−0.6319	−0.7093
β_3	25.4037	19.5531	20.5582	19.1448	2.8751	0.2027	0.2987	0.1851
β_4	0.1241	0.4452	0.6317	0.7791	0.1292	0.2358	0.2482	0.2641
β_5	1.795E-2	1.702E-2	1.947E-2	1.889E-2	5.46E-3	5.18E-3	5.43E-3	6.48E-3
β_6	−2.0227	−1.2466	−1.4571	−1.0101	−0.4438	−4.77E-2	−3.43E-2	−0.1274
β_7	−8.61E-3	−1.13E-2	−1.24E-2	−1.13E-2	−2.21E-3	−2.69E-3	−2.60E-3	−2.94E-3
β_8	6.748E-2	4.48E-2	3.277E-2	1.174E-2	9.45E-3	−2.33E-3	5.32E-3	−2.93E-3
σ	3.27	2.07	2.13	2.33	0.41	0.50	0.68	0.74

data are lower than obtained by means Fisang's model ones, and, for flexural data, are similar than obtained on eq. 1.

When eq. 3 is tested as a model for regression analysis in 3–365 days curing time period, convergence occurs, although standard deviations are greater than for early-age period. Coefficients for regression analysis are given in Table 5.

From model proposed in eq. 3, optimal quantity of fly ash and curing time age can be calculated. Unlike results reported in Fisang's paper (13), in our case for all fly ashes tested, optimal mechanical characteristics were found for longest curing time (365 days). Table 6 summarizes optimum values of fly ash replacing percentage for the following curing times: 28, 60, 90, 180 and 365 days, and Figures 9 and 10 show comparisons between mechanical strength response curves for T0 and T60 fly ash containing mortars at several curing times.

Globally, optimum replacing percentages at early-age curing times are 15%, indicating that fly ash contribution to strength development is not important. However, for longer curing times, fly ash replacing quantities must be increased in order to obtain the best yield of fly ash properties. Moreover, these optimum values revealed that grinding procedure beneficiates clearly the activity of fly ashes, at least, in the age range studied.

TABLE 6
Optimums for Mechanical Characteristics for Mortars at Different Ages.

	Optimums for Compressive Strength				Optimums for Flexural Strength			
Curing time (days)	T0	T10	T40	T60	T0	T10	T40	T60
28	15	15	15	15	15	20	21	21
60	15	15	18	21	18	27	28	29
90	15	18	21	23	24	29	30	31
180	15	23	24	26	30	31	32	33
365	21	26	27	28	35	33	33	34

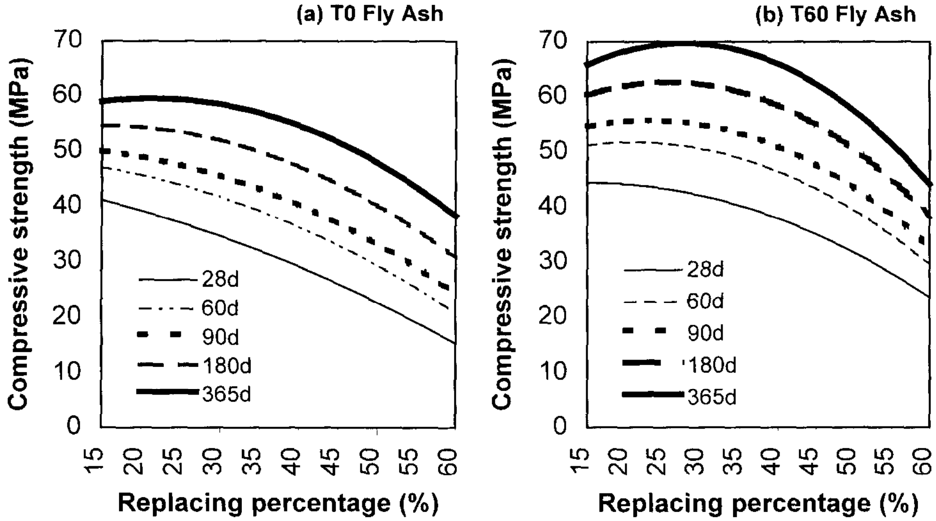

FIG. 9.
Compressive Strength Response Curves for T0 (a) and T60 (b) Fly Ash Containing Mortars.

Conclusions

1. Good linear correlations have been established between compressive or flexural strength and the logarithm of curing time for 3–365 days curing time period, and slopes of these linear relationships increased with fineness of GFA used. Intermediate replacing percentages (30 and 45%) in the studied range (15–60%) were the best for reactivity comparisons.
2. Compressive Strength Gain (SGi) and Pozzolanic Effectiveness Ratio (PER) were calculated from experimental data; SGi values showed that early mechanical strength

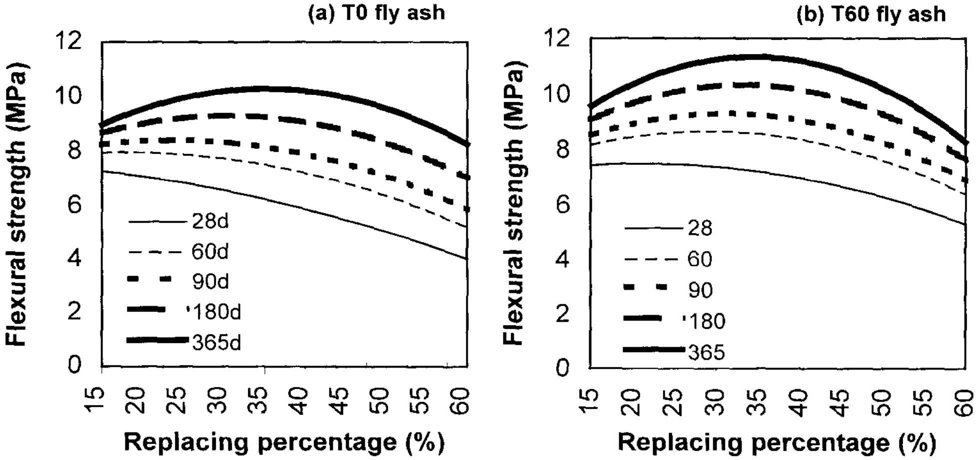

FIG. 10.
Flexural Strength Response Curves for T0 and T60 Fly Ash Containing Mortars.

development was optimum for lowest replacing percentage (15%), although this optimum replacing percentage shifted to higher values (30–45%). PER values for mortars containing GFA were greater than for mortars containing non-mechanically treated fly ash (T0), suggesting the activation of the fly ash with grinding.

3. Model proposed by Fisang et al (eq. 1) yielded good results for mechanical properties of mortars prepared with GFA and short curing times (3–28 days). This model did not show convergence when medium-term data were included for calculation.
4. A new model (eq. 3), assuming linear dependence of mechanical properties with the logarithm of curing time, has been proposed and studied. For this model, convergence using experimental data succeed, and coefficients for this polynomial equation were calculated for GFA used.
5. Replacing percentage optimums for compressive and flexural strengths for mortars at different ages were calculated from the new model. Finest GFA permitted to obtain mortars containing the highest optimum replacing percentage for mechanical properties, specially for compressive strength.

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