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Effect of elevated temperature on the hydration heat and mechanical properties of blended cements mortars

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ABSTRACT

This paper presents an experimental investigation for testing the effect of elevated temperature on the hydration heat and mechanical properties of blended cement mortars. Two tests on mortars were made. The first set of mortars is tested of mechanical properties at various temperatures of 20, 35 and 50°C and the second test consists to determine the hydration heat by a semi-adiabatic calorimeter at isothermal temperature of 20, 35 and 50°C during seven days. The new empirical equation has been proposed to estimate the compressive strength depending on the hydration heat for blended cement preserved in constant temperature at early. The results founded from this relationship illustrate a good accuracy with the experimental ones and reflect the best choice to be used to predict the compressive strength depending on the heat of hydration at early age (7 days).

1 Introduction

The hydration process in cement paste produces the characteristics of mortar and mix proportions as well as the change in construction and environmental conditions. Cement hydration also consequently influences cement paste workability, setting time and mechanical properties. It is important to take into account the factor that the temperature and other climatic conditions will be applied that can cause undesirable effects on the structures in civil engineering.

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Several researches have been based in the influences of conditions of conservation such as temperature, humidity on the properties of mortar and the concrete. The cumulative hydration heat of the cement paste increased with the curing temperature, and the influence of the negative temperature on the cumulative hydration heat of the cement paste increased with the water-cement ratio [1]. In a study by the authors utilizing synchrotron X-ray diffraction, it was observed that the rate of conversion of ettringite to monosulfate increased with increasing temperature, and monosulfate became unstable when temperatures reached 85°C [2].

Korpa et al. [3] have shown that the pozzolanic activity is weak at 20 °C. On the other hand, at 90 °C the pozzolanic activity of both silica fume and crushed quartz is increased.

In the lower temperatures, the curves of hydration have been reduced intensively. On the other hand, when the cement paste subjected at higher temperature the density of hydration products is higher and the water that is found in the microstructure of the cement paste is slowed down [4, 5].

The loss of moisture due to high temperature is one of the main phenomena that should be avoided, because it causes plastic retraction and consequently, cracks. The best solution to such adversity is to keep the structure always-moist [6].

The mechanicals properties of the concrete take into account on the available and surface hydration will be interrupted at low humidity and also seriously deteriorate its mechanical and fracture properties[7-9].

The analysis of the hydration heat and compressive strength is a very effective way to examine the influence of mineral additives in cement.

The world consumes large quantities of cement every year. Large-scale cement production leads to a large amount of CO₂ emissions [10, 11]. For example, the substitution of 30% for cement clinkers can reduce CO₂ emissions and energy demand by 23% and 21%, respectively [11].

The presence of mineral additives in cement paste modifies the hydration kinetic and the resulting heat by reducing the reactive phase of cement and contributing to hydraulic and pozzolanic reaction.

The replacement of clinker by 20% of Limestone Powder (LP) gives similar strength development to Ordinary Portland Cement (OPC) up to 7 days. Fine limestone enhances cement hydration and offsets the dilution effect caused by the decrease of clinker amount. The accelerating effect of the limestone was explained by a higher degree of under saturation with respect to C3S [12]. On the other hand, that the dilution effect means that the increase considerably amount of LP reduces the amount of cement, but also tends to decrease matrix strength and durability [13].

Pane and Hansen [14] show that the cement with fly ash (FA) has less hydration heat compared to ordinary Portland cement (OPC). However, the addition of steel slag (SS) in cement increases the Ca concentration in the pore solution, reduces the supersaturation of the pore solution with respect to CH and inhibits the nucleation and growth of CSH [15].

The limestone powder (LP) has been used as an additive or replacing part in fabrication of the cement and allows improving the mechanical and physical properties of cement paste at early ages [16-18]. Ezziane et al. [19] have shown at 50°C of temperature, the compressive strength at early age of cement increased by about 30% compared by the decrease in cement exposed at 20°C. Hence, effective consumption of (LP) is essential not only for environmental protection but also for sustainable development of limestone industry [13].

The incorporation of mineral additives in the cement exposed to high temperature improves the mechanical properties and durability performance at later age [20-22].

Boubekeur et al. [23] have found that the heat of hydration of the Limestone Powder is almost similar to that of (OPC) with the rise of temperature and guard always its superiority from its positive effect, compared to other minerals additives that manifest tardily such as the natural pozzolana (PZ). Zhang et al.[24] studied the hydration process of sulphoaluminate cement-based dual liquid grouting material cured at high thermal conditions for example at 40°C of temperature, the early compressive strength of the cement is higher, but at later age the compressive strength increases slowly. Gallucci et al. [25] have shown that after three days of hydration, the compressive strength of concrete subjected at 20°C catches up with that of concrete at 40 and 60°C. After 28 days, it exceeds them, while the development of the compressive strength of a concrete at 60°C is 25% lower. The maximum development of resistance due to the pozzolanic reaction of fly ash has been observed for an optimal temperature of 40°C [26]. Other researchers Escalante-Garcia and Sharp [27] have concluded that 30°C is the optimal temperature for developing the resistance of slag cement.

The objective of this study is testing the effect of elevated temperature on the hydration heat and mechanical properties of blended cement mortars and predictive relationship between the compressive strength and hydration heat of blended cement cured at different temperature at early age. This new relationship in this paper will be checked based on experimental results on mortars based on different types of cement under various curing temperature historic.

2 Materials and methods

2.1 Materials used

The cement type selected in this research is ordinary Portland cement CEMI 42.5 (C1), limestone cement (CEMII-A-L-42.5) containing 15% of limestone powder (C2) and pozzolanic cement (CEMII-A-P-42.5) containing 18% of natural pozzolana (C3). The specific surface for cement and the addition was measured with Blaine method. The oxide composition crystalline phases and physical properties of the cementitious materials are summarized in Tables 1 and 2. All mortars are prepared according to the ASTM C305-06 standard [28] The mixtures proportion for all mortars are presented in Table 3.

Table 1– Oxide composition and crystalline phases of the cementitious materials.

	C1	C2	C3
SiO₂	20.58	18.25	23.44
Al₂O₃	4.90	4.48	5.47
Fe₂O₃	4.70	3.28	2.83
CaO	62.8	62.3	60.49
MgO	0.53	0.94	0.39
SO₃	2.28	2.00	2.46
C₂S	60.9	18.68	11.97
C₃S	17	58.2	67.2
C₃A	8.54	7.55	8.13
C₄AF	10.9	11.43	9.3

Table 2– Physical properties of cementitious materials.

Property	Unit	C1	C2	C3
Fineness	cm ² /g	3000	3400	3600
Density	g/cm ³	3.1	2.7	2.9
Activation energy	kJ/mole	29191	28335	36947

Table 3– Mix proportion for all mortars studied.

Mix design	Cement (g)	Sand (g)	Water (g)
Mortar of hydration heat			
C1	360	1080	180
C2	360	1080	180
C3	360	1080	180
Mortar of mechanical strength			
C1	450	1350	225
C2	450	1350	225
C3	450	1350	225

2.2 Test method

2.2.1 Heat of Hydration

The heat of hydration test was carried out according to NF 15-436 (1988) [29] standard. The method is based on the Langavant Calorimeter. This method consists in quantifying the heat generated during cement hydration using a thermally isolated bottle, reserved in the temperature of 20, 35 and 50°C during seven days. The hydration heat of the mortar is calculated by the Eq. (1).

$$q(t) = \frac{C}{m_c} \Delta\theta + \frac{1}{m_c} \int_0^t \alpha \Delta\theta dt \quad (1)$$

where C presented the thermal capacity (J/°C), m_c is the weight of cement (g), $\Delta\theta$ is the mortar heating difference to the ambient temperature (°C), and α is the total calorimeter thermal loss coefficient (J/h/°C).

2.2.2 Mechanical strength of mortar

The flexural and compressive strength of all mortars were determined in accordance with the European standard EN 196-1 [30]. The mortar is placed in 4×4×16 cm prismatic steel molds. After 24 hours, the mortar specimens are removed from the molds and was immersed after molding at temperature of 20, 35 and 50°C, details of curing treatment is presented in Table 4. Where they will be removed an hour before the flexural and compressive strength crushed at 1, 3, 7 and 28 days. The flexural strength of mortars has been determined by three-point bending tests carried out on 4×4×16 cm prismatic specimens, with a loading speed of 50 N/s. The two parts of each 4×4×16 cm mortar subjected to flexural crushing are reused for the compression crushing.

3 Results and discussion

3.1 Apparent activation energy

It is very interesting to use the concept of the apparent activation energy in hydration reaction to visualize the difference between the activation of mineral additions and that of reference cement (cement without additions). It can be evaluated its kinetics by the following Arrhenius relation [31, 32].

$$k = A \exp\left(-\frac{E_a}{RT}\right) \quad (2)$$

where k is the kinetics constant, A is the constant of proportionality, R is the perfect gas constant, 8.314 J/molK; and E_a is the apparent activation energy (kJ/mole).

For two temperatures T_1 and T_2 , the same degree of advancement of hydration must be completed at times t_1 and t_2 from which we can write:

$$\frac{k_1}{k_2} = \exp\left[\frac{E_a}{R} \left(\frac{1}{T_2} - \frac{1}{T_1}\right)\right] = \frac{t_2}{t_1} \quad (3)$$

Table 2 shows the energy found for each type of cement and for the different temperatures. The (C3) has a higher activation energy than (C2), it is considered one of the most active and recommended additions in hot climates. However, the (C2) gives lower activation energies than that of other additions. This makes him more active at early age and less susceptible to rising temperatures.

3.2 Hardening properties

An approach recommended by ASTM Standard C107422 [33], to analyze propriety-age data under isothermal curing was used. It was assumed that the kinetics of the propriety development at a constant temperature can be represented by the following hyperbolic equation.

$$\frac{1}{S} = \frac{1}{S_u} + \frac{t_{50}}{S_u} \left(\frac{1}{t-t_0} \right) \tag{4}$$

where: S: is the concrete property at t age; S_u is the ultimate value property; t₀: is the age when the concrete property development is assumed to start and t₅₀: is the time beyond t₀ where the concrete reaches 50% of its ultimate value property

For the calculation, we take as ultimate strength those corresponding to 90 days. These results obtained are shows in Table 4, for the different temperatures.

Table 4 – Values of ultimate strength and half strength age.

Cement type	Ultimate strength (MPa)			Half strength age (t ₅₀ , days)		
	20 °C	35°C	50°C	20 °C	35°C	50°C
Cement (C1)	39	44	49	3,6	3.0	2,1
Cement (C2)	38	43	48	3,1	2,3	1,8
Cement(C3)	40	45	50	4,6	3,3	2,2

3.2.1 Half strength age

The half strength age represents the time it takes for mortar to reach half of the ultimate strength. Figure 1 shows the variations in the half strength age obtained as a function of the curing temperature, for the different types of mortars studied. On the other hand, the natural pozzolana is considered as active additions, their presence in the composition of the cement delays the development of strength at early age and increases the half strength age. On the other hand, the temperature rise decrease in the half strength age, as shown in Figure 1. The addition of limestone is considered inert, but it contributes to improving the compressive strength at early age, by modifying the kinetics of hydration. This results in a decrease in the half strength age, which remains slightly lower than that of ordinary cement. It can be noted that the rise in temperature leads to a reconciliation of the half-age values regardless of the addition used; thus, it appears that the effect of the nature of the addition disappears with the rise in the curing temperature.

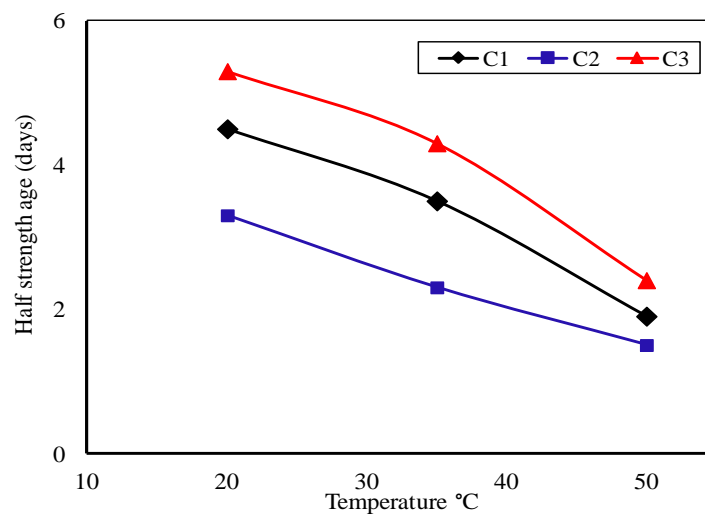


Fig. 1– Variation de half strength age depending the temperature rise

3.2.2 Ultimate strengths

One can assume that the ultimate strength is very close to compressive strength at 90 days. Generally, these strengths can be represented by an affine and decreasing function of the increase in the cure temperature, according to the following equation described by the equation of Chanvillard and D'Aloia[31].

$$S_u(T) = S_u(20^\circ\text{C})[1 - k(T - 20)] \quad (5)$$

where k is the slope of the normalized model which corresponds to the average of the values obtained in the bibliography equal to $10.2 \cdot 10^{-3}$.

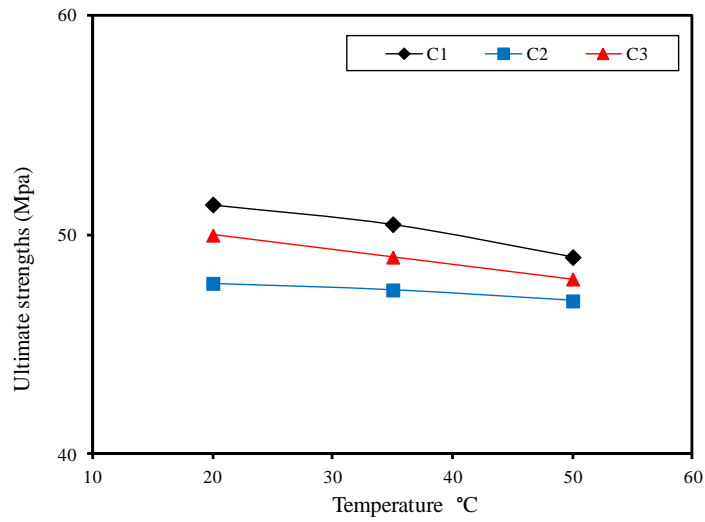


Fig.2– Variation de ultimate strength depending the temperature rise.

The presence of mineral additions such as limestone and pozzolana in the composition of the cement modifies considerably the ultimate strengths. From Figure 2 we notice decreasing strengths values with the temperature rise for all the mixtures studied and represented by affine lines. This has led research into the use of this product in order to acquire better performance under appropriate storage methods, especially under severe atmospheric temperature conditions.

3.3 Heat of Hydration

The effect of chemical composition can be identified by evaluating the rate of hydration of the individual compound and its percentage in the cement. As it has been discovered in the early researches that in the middle period of hydration after 4 hours, the dissolving clinker grains are completely covered by cracked shells of C-S-H [34, 35].

Figs. 3, 4 and 5 show the evolution of the heat of hydration to 168 hours cured under constant temperatures 20, 35 and 50°C for several types of mortars.

The temperature of cure has a positive effect on the heat of hydration under a temperature of 50°C; the mortar (C1) has the higher values of heat of hydration compared than other mortars. This explains that at very early age the increasing in temperature accelerates the dissolution of the anhydrous clinker in cement paste. In addition, when the cement paste subjected at higher temperature the density of hydration products is higher and the water that is found in the microstructure of the cement paste is slowed down [4, 5].

Okemute and Leon [36] observed that increasing the thermal condition results in greater heat evolution for cement paste. Conversely, at 20 °C initial heat evolution was lower and will increase over time.

According to the results in Fig. 3, it can be noted that the heat of hydration at seven days of the (C1) cured at 20°C is higher compared to other cement; it has about 330 J/g followed by the cement (C2) and cement (C3) that is 280 J/g and 235 respectively. The dilution effect created by (C2) additive is quickly compensated by their physical activity. Contrary to

(C3) that react slowly, because the pozzolanicity of this addition which triggers slowly. The pozzolanic additives are known to decrease the hydration heat processes of cement [37].

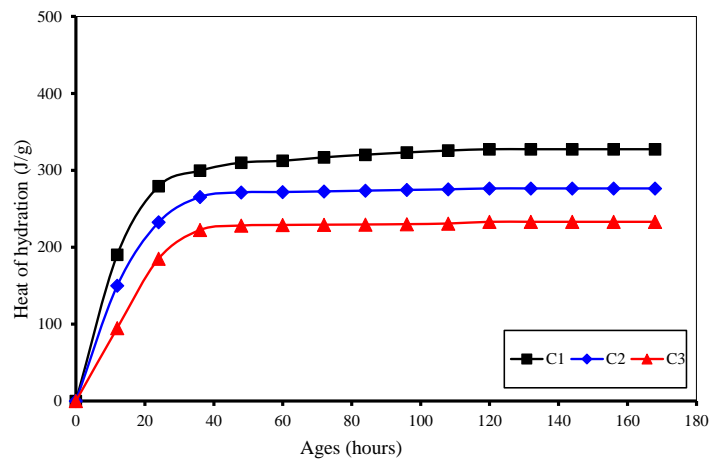


Fig.3–Evolution of the hydration heat for different cement types at 20°C.

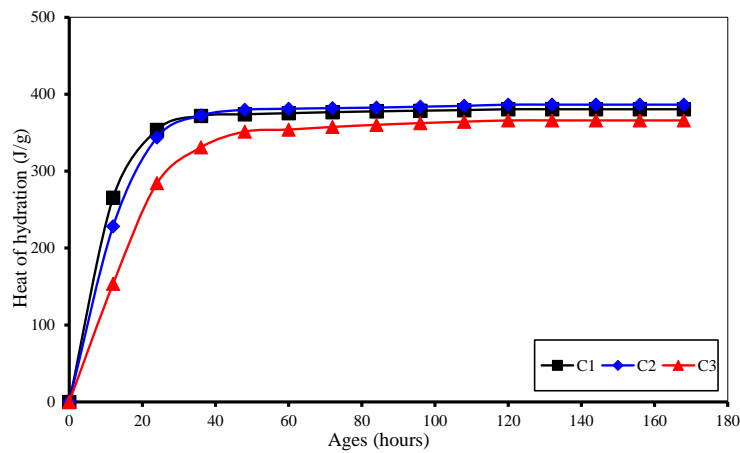


Fig.4–Evolution of the hydration heat for different cement types at 35°C.

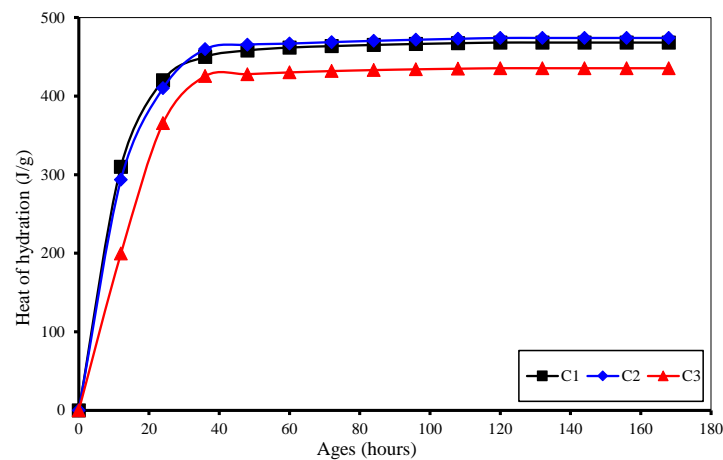


Fig.5– Evolution of the hydration heat for different cement types at 50°C.

Fig. 5 shows an acceleration of the heat of hydration when the temperature reaches to 50 ° C, for all mortars. The heat of hydration of (C2) is almost similar to that of (C1) that reach 470 J/g at seven days. This result is consistent with the work of several researches[4, 38, 39] where the degree of heat hydration of cements, increases with temperature rise.

It is also worth to note that the highest heat hydration, around 475 J/g at 50°C, is released from the mixture made with cement C2, due to the presence of limestone in this cement.

3.4 Flexural and compressive strength

The results of the flexural strength for the different types of all mortars subjected to temperatures of 20, 35 and 50 °C are presented in Fig. 5. At very early age, temperature rise has a positive result on the evolution of flexural strengths, where mortar containing (C2) is similar by that of ordinary cement and has the highest strengths followed by (C3) (Figs. 4a and 4b).

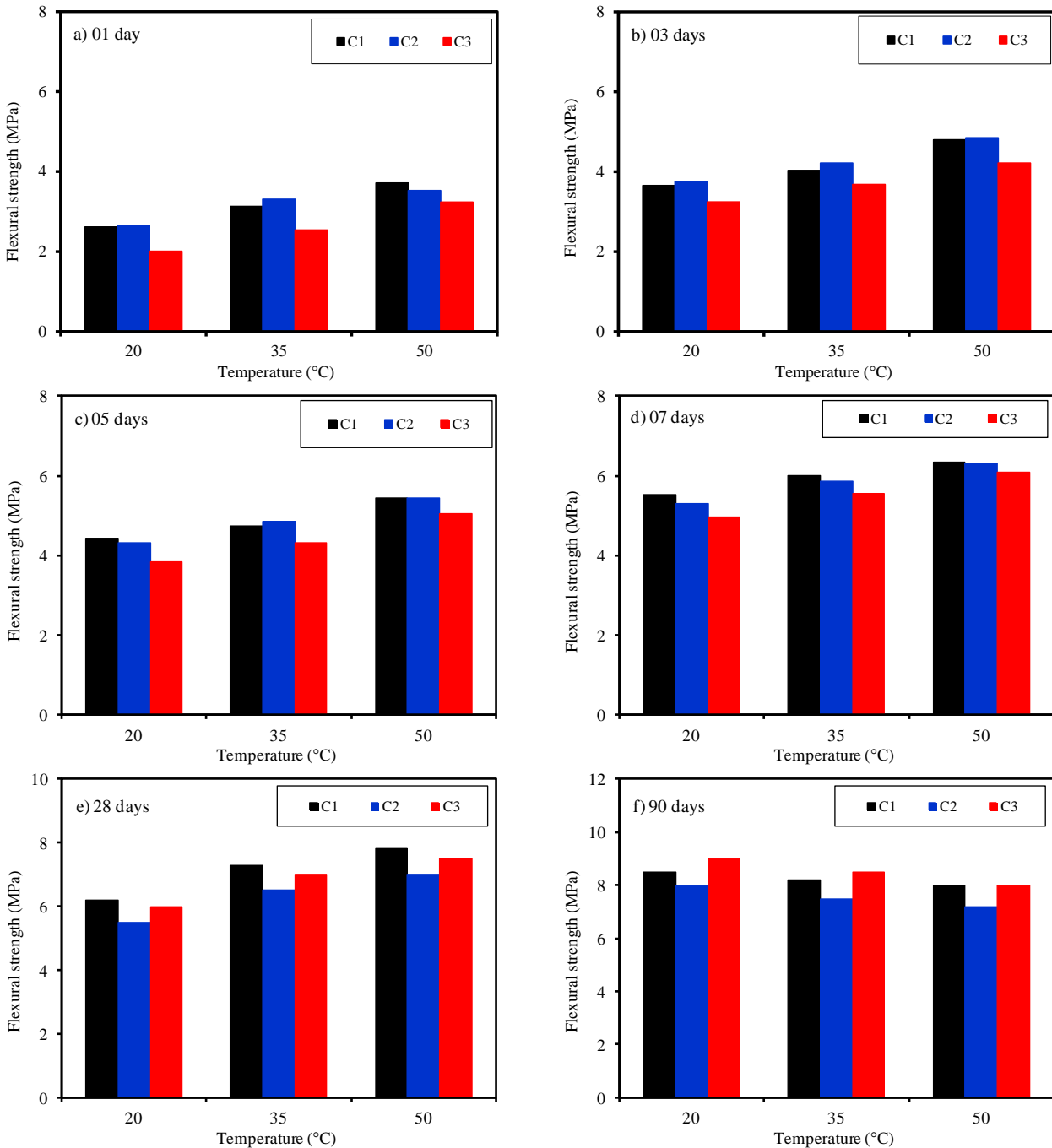


Fig.6– Flexural strength of different cement mortars according to curing temperature.

At 7 days, the increase of flexural strength is clear when a mineral additive replaces the cement, where the increase in flexural strength between 20 and 50°C is 12, 16 and 18% for a mortar (C1), (C2) and 18% (C3) respectively (Figs. 6c and

6d). The influence of temperature upon the flexural strength is insignificant at early age of curing, but it affects greatly the compressive strength. At 28 and 90 days, the cement (C3) is activated and cause an increase in flexural strength of mortars, which exceed that of mortar (C1) (Figs. 6e and 6f).

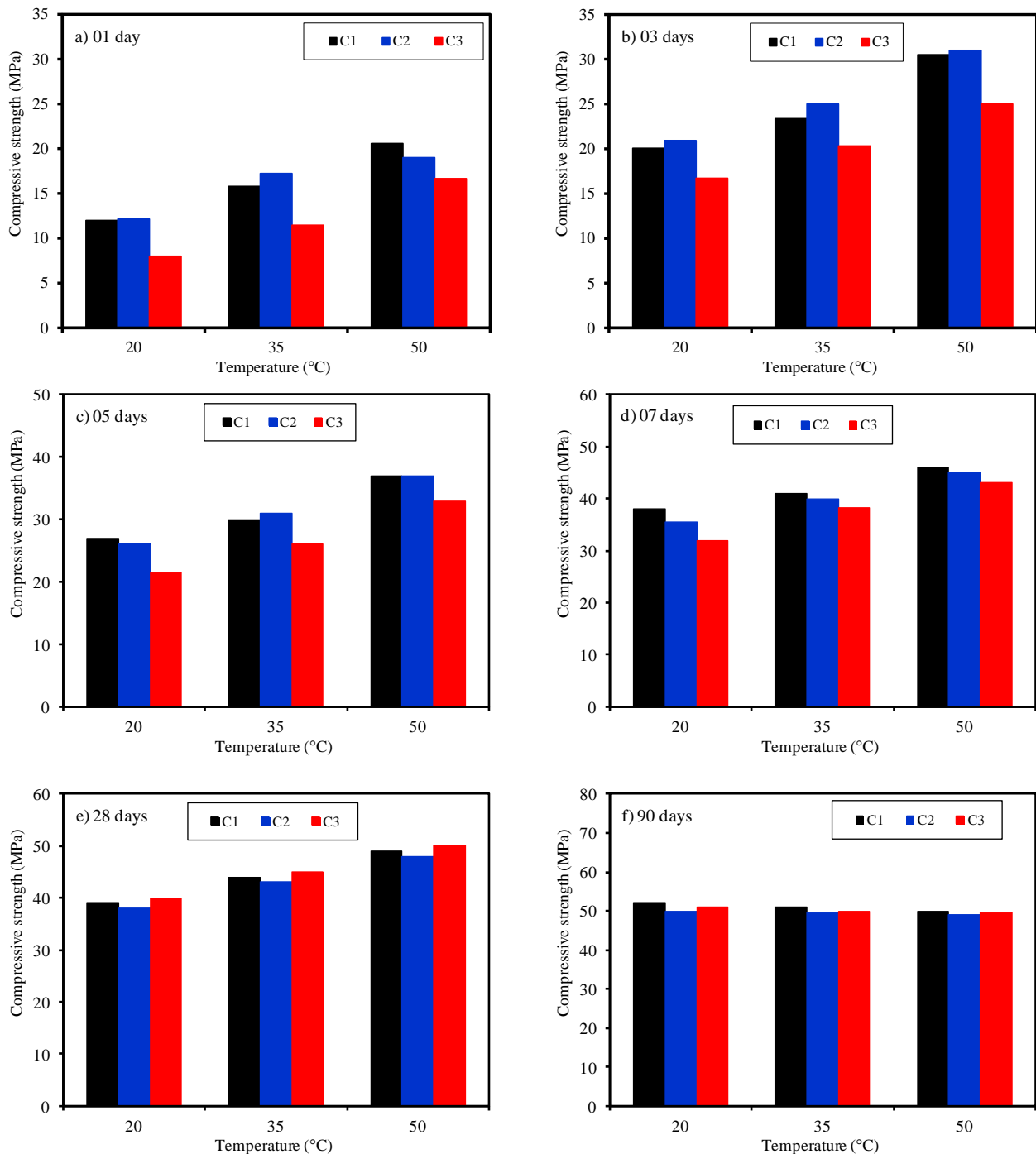


Fig.7–Compressive strength of different cement mortars according to curing temperature.

Fig. 7 shows the compressive strength according to temperature rise of 20, 35 and 50°C, at very early ages for different cement types. The results indicate that the compressive strengths for all mortars increase with the temperature rise. However, the mortar (C1) has the highest compressive strength values followed by that (C2) and (C3) respectively. At 1-day the raising of the temperature from 20 °C to 50 °C, will increase the compressive strength for (C1), (C2) and (C3) by 18, 14 and 19% respectively (Figs. 7a and 7b).

The (C2) cement present a positive effect for increasing the compressive strength at early age; it is due to its high activity at very early age and small particles. At 7-day under temperature of 50°C, the compressive strength of (C2) is 46.5 MPa close to that of the control cement (C1) which gives 47 MPa (Figs. 5c and 5d). However, a retardation of increase in the compressive strength is observed for cement (C3) when compared with the (C1) and (C2) cement. This negative effect (C3) on the early-age strength can be explained by the pozzolanic activity, which can be alleviated by prolonging the hardening. At 28 days, the pozzolanicity of natural pozzolana additives is activated and cause an increase in compressive strength mortars which exceed that of cement (C2) and (C1). The increase of compressive strength is noticeable when minerals additive changes the cement, where the increase in compressive strength between 20 and 50°C is 19, 20 and 21% for a mortar (C1), (C2) and (C3) respectively. At 90 days, for mortar cured one day at 50°C, a decrease in compressive strength is 3 and 2% for (C1) and (C2) compared with that cured at 20°C. On the other hand, this reduction of compressive strength is only 3 % for mortar containing (C3) as shown in Fig.7

It can be remarked that the compressive strength of the mortar exposed at higher temperature will exceed that subjected at lower temperature in less time. On the other hand, at later age the compressive strength increases slowly. These results are consistent with those reported by Zhang. Yaohui et al.[24] where are found that the temperature is both 40 °C, the early strength of the mortars is higher, but the later strength increases slowly. However, when the curing temperature is both 0 °C, the early strength is lower, but the later strength increases more rapidly and the final strength is higher than that of the samples at 40 °C.

The temperature rises during early stage of hydration process to the formation of products of larger pores in cement paste, which decrease the compressive strength of concrete[13, 40, 41]. The increase of compressive strength is noticeable when minerals additive changes the cement, where the increase in compressive strength between 20 and 50°C is 19, 23 and 27% for a mortar (C1), (C2) and (C3) respectively.

The studies of Shoukry et al. [9] point out that the increase in concrete temperature at 80°C results in a loss of compressive strength and tensile strength at 38% and 26%, respectively.

The correlation between the flexural and compressive strength is presented in Fig. 8 and can be expressed as follows:

$$S_f = a(S_c)^b \quad (6)$$

where S is the flexural strength and a and b are coefficients specific to the mortar mixture.

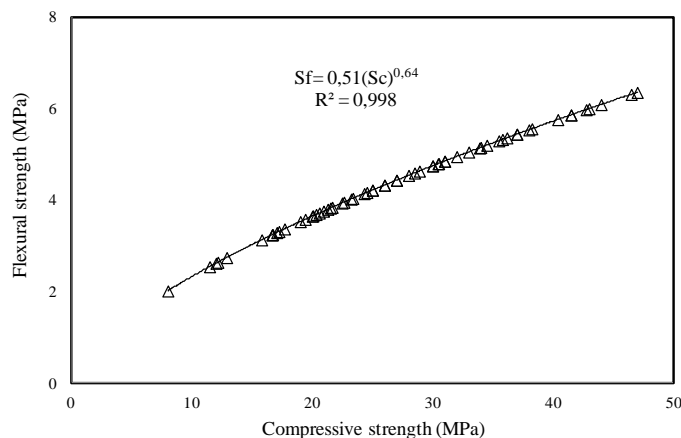


Fig. 8–Correlation between flexural and compressive strength for different types of cement.

4 Predicting the compressive strength

Little researches indicate that it exists a relationship between the mechanical strength and heat of hydration of cements, taking into account the parameter of the temperature [42]. After many adjustments of the experimental results found in this research, an exponential shape equation have been founded to estimate the compressive strength between the heat of hydration of mortars containing blended cement taking into account the type of cement, the temperature and the age

of mortar. The error deviation Eq. (7) is calculated for all points as the difference between the calculated compressive strength and the measured compressive strength.

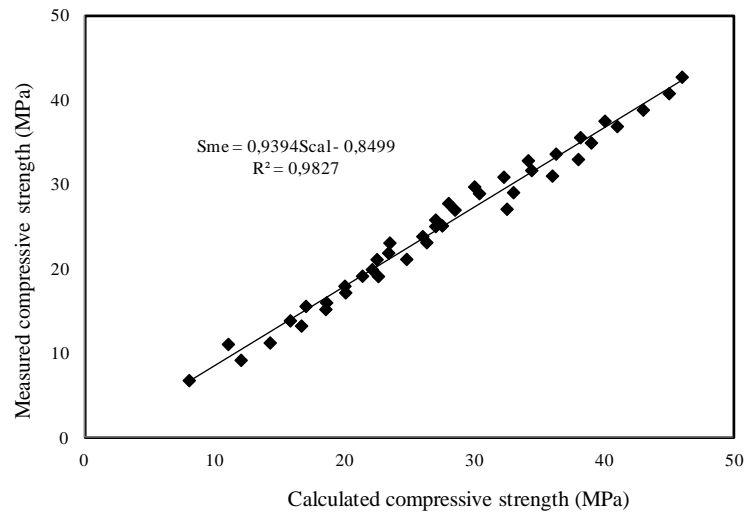


Fig.9– Comparison of measured and calculated compressive strength by Eq. (8) of C1

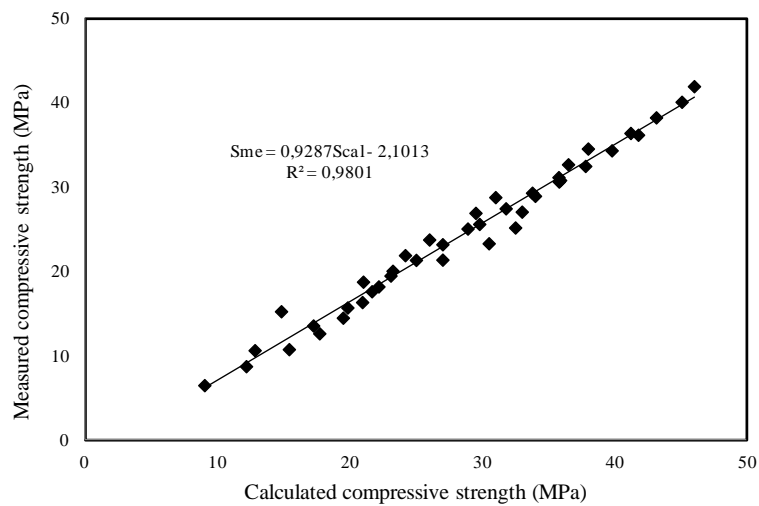


Fig.10– Comparison of measured and calculated compressive strength by Eq. (8) of C2

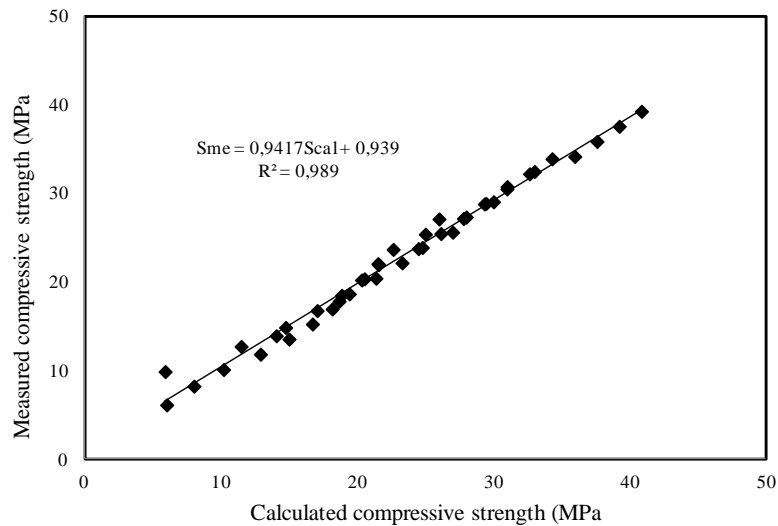


Fig.11– Comparison of measured and calculated compressive strength by Eq. (8) of C3

$$Error = \frac{\sqrt{(S_{calculated} - S_{measured})^2}}{n} \tag{7}$$

The relationships between the compressive strength and the heat of hydration proposed in this study can be written as follows:

$$S_c = 0.2T e^{q(t)10^{-3}} + k_c t \tag{8}$$

where S_c (MPa) is the compressive strength at age t , $q(t)$ is the hydration heat, k_c coefficient depending on the type of cement used which is determined by the minimization of error deviation value (Table 5), t (days) is the age of mortar which must not exceed 7 days and T is the temperature ($^{\circ}C$).

Table 5–Correlation results between measured and calculated compressive strength for different cement types

Cement type	Values of k_c	Coefficient of correlation R^2	Mean squared error (%)
C1	3.90	0.9827	3.2
C2	3.70	0.9801	3.3
C3	3.40	0.9890	4.2

Figs. 9, 10 and 11 show the comparison between the measured and calculated results using the Eq. (8) for all mortars studied, in which a perfect position of the values with an acceptable correlation coefficient. Just as shown in Table 5 the correlation coefficients is very near to the unit demonstrating that the equation Eq. (8) found is able to accurately estimate the compressive strength at early age depending on to the hydration heat for different types of mortar cured under constant temperature. The obtained correlations between strength and heat of hydration in linear form allow calculation with a very low error the compressive strength of cement. The validation showed that the equation predicts the compressive strength with an error deviation of $\pm 4\%$ (Table 5).

5 Validation of the equation proposed

In order to check this relationship founded, four experimental results published in the literature used [5, 40-42]. The first study realized by Kadri *et al.* [43] on mortar containing (OPC) kept at temperatures of $20^{\circ}C$ with w/c ratio equal to 0.425. Fig. 12 show good results between the measured and calculated values by Eq.(8).

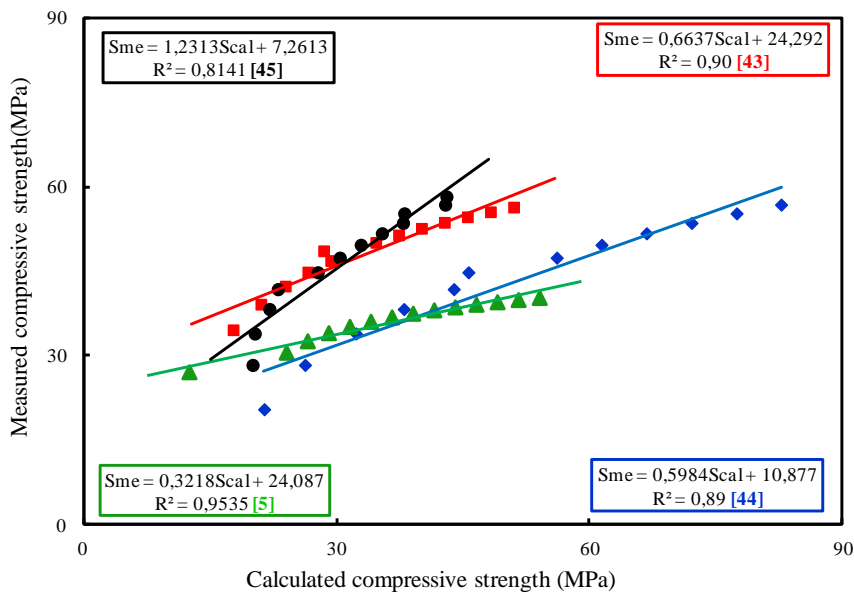


Fig.12– Comparison of measured and calculated compressive strength by Eq. (8) for four sets of data.

The second study is presented by Dale *et al.* [44] on mortar with a 10% cement replacement by the (LP) cured at 20°C and w/c ratio equal to 0.4. The results measured [45] and those calculated by Eq. (8), are illustrated in Fig. 12. Again, there is also a good agreement between the measured and calculated results. The third study is conducted by Ľubomír *et al.* [42] on mortar (OPC) and cured at temperatures of 10, 20 and 30°C testing at 1, 2 and 7 days. After the comparison between the measured and these determined by Eq. (8) as indicated in Fig.12 the obtained results show good agreement between the calculations of the compressive strength of blended cement cured under constant temperature where we have the heat of hydration values at early age. The fourth research is presented by [45] on mortar containing ternary cement (15% PZ +15% GBFS) and Blaine values of 480 m²/kg. The correlation coefficient is low compared to others researches such as Binici *et al.* [45]. This may be due to the ternary blended cement used (PZ+GGBFS). We can conclude that the results obtained are very satisfying.

6 Conclusion

According to the results, an experimental study lead the influence of the high temperature on compressive strength and heat of hydration of mortar containing blended cement the following conclusions can be drawn.

In this research, the effect of thermal loading on the heat of hydration and the compressive strength of cement containing several minerals additives are studied.

At a very early age, the heat of hydration of all the cements studied increases with the high temperature.

The mortar with (C2) is almost similar to that of (C1) and has the highest heat of hydration compared to (C3).

All cements display increase in the compressive strength with temperature rise.

The relationships obtained by the correlations of the experimental results express the variation of compressive strength depending on heat of hydration can only be used for mortar containing blended cement cured at constant temperature and not for other types such as ternary blended cement.

The empirical equation found is applicable to estimate compressive strength at early age (≤ 7 days) and does not suffice at later age.

The obtained results are encouraging and testifying to the reliability of the relationship proposed to estimate the compressive strength depending on the heat of hydration of blended cement at early age.

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