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Cement & Concrete Composites 29 (2007) 587–593
Cement & Concrete Composites
www.elsevier.com/locate/cemconcomp

Compressive strength of mortar containing natural pozzolan under various curing temperature

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Received 26 October 2005; received in revised form 13 February 2007; accepted 15 March 2007
Available online 24 March 2007

Abstract

A first set of mortar specimens was made with 0%, 10%, 20%, 30% and 40% of natural pozzolan replacement and cured under constant curing temperature of 20, 40 and 60 °C with saturated humidity. The second set incorporating only 20% of natural pozzolan was exposed to elevated temperatures for 1, 3 and 7 days and then cured in saturated environment under a temperature of 20 °C. This paper presents the experimental results of the mortar properties such as the ultimate strength, the half age of strength and the activation energy. The introduction of the pozzolan enhances the ultimate compressive strengths and increases the activation energy which indicates the slow reactivity. The effect of the temperature on the ultimate strengths is evaluated by a new model. By using the equivalent time method, we could estimate the strength at 28 and 90 days of the second set mortar. This method satisfactorily predicts the strength of concrete cured at any temperature history.

Keywords: Cement; Compressive strength; Equivalent time method; Mortar; Natural pozzolan; Temperature

1. Introduction

Concrete exposed to elevated temperature shows an accelerated hydration and nonuniform distribution of hydration products [1,2]. This generates a great porosity and an increase of the compressive strength at early age and a decrease at late age [3,4]. The presence of some mineral admixtures such as natural pozzolan, fly ash or granulated blastfurnace slag in the cement can modify the kinetic of hydration, reduce the heat evolution and produce additional CSH hydrates [5]. At an elevated temperature, they influence the cement hydration and minimise disorders caused by the temperature rise according to the characteristics of these admixtures and their replacement rates. These admixtures give to the concrete a noticeable performance in hot climates in which the negative effect of the temperature is partly reduced by the pozzolanic reaction, their weak hydration heat and their great activation energies.

Several investigators [1,3,5] reported that high temperature improves strengths at early age. At a later age, the important number of formed hydrates had no time to arrange suitably, and this engendered a loss of ultimate strengths; this behaviour had been called the crossover effect [6]. Bakharev et al. [7] reported that the more dense precipitates deposited at an elevated temperature may form a barrier for ion diffusion, thus causing an inhomogeneity in the microstructure and a significantly reduced strength. Escalante-Garcia and Sharp [8] observed in the microstructure of cement paste containing 22% of volcanic ash, cured at 60 °C, that CH was almost absent and the porosity was appreciably greater.

For Ordinary Portland cement, it appears that the ultimate strength decreases nearly linearly with curing temperature [9]. Videla et al. [10] studied the behaviour of

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doi:10.1016/j.cemconcomp.2007.03.002

Portland cement and blended cement under various curing temperatures (5, 20, 30 °C) and observed that blended cement strength shows ascending curves with the increase of temperature. Mirza et al. [11] found that blending OPC with pozzolanic materials such as powdered tuff, slag and silica fume helps to improve the performance of mortars and concrete strength on exposure to higher temperature than normal, particularly in the range of 35–50 °C.

Monzo et al. [12] examined the strength of fly ash blended cement under 20, 40, 60 and 80 °C; they found that 40 °C is an optimum temperature for pozzolanic reaction development in the experimental conditions studied. Other investigations conducted by Escalante and Sharp [1] concluded that 30 °C is the optimum temperature for strength development in the slag cement paste blend. Kim et al. [4] have reported that the crossover effect of type V cement concretes is not so obvious with regard to the type I cement due to the difference in the hydration rate with cement type. These results are in conformity with the Turkel and Alabas conclusions [13] that composite cements perform well under heat treatment and they do not exhibit a considerable loss in the compressive strength.

The natural pozzolan, used since the Roman era, is often used in the manufacture of cement, because of its economy and its performances brought to the concretes. In Algeria, this pozzolan is used largely in the west of the country in order to minimise the effect of the temperature rise in summer. This work is intended to study the behaviour of cement including different replacement level of the natural pozzolan and hardening under different curing temperatures. It is also expected to analyze the contribution of this pozzolan to the deduction of the ultimate strength of pozzolan cement under any temperature history. In addition, pozzolan cement, hardening under a temperature higher than 20 °C, is expected to use in summer period and in the southern part of the country (Sahara) in order to reduce the negative effects due to the hot climate.

2. Experimental procedure

As the concrete strengths depend on several factors and mainly mortar strength, we have preferred working with mortar. Mortar specimens were prepared by casting 40 mm cubes; the cement–sand–water weight ratio was 1–3–0.47. An amount of superplasticizer (1% by weight of the binder) has been added to obtain an acceptable workability. Table 1 gives characteristic physico-chemical of the materials used.

The first set of mortar specimens (named A) was prepared containing cement replacement by 0%, 10%, 20%, 30% and 40% of natural pozzolan ground to fineness similar to cement. These specimens were cured under constant temperatures of 20, 40 and 60 °C. The second set of mortars (named B) in which we have substituted the cement by 20% of natural pozzolan was exposed to various temperatures (Fig. 1).

All specimens were cured immediately after casting, then they were demoulded the next day and cured in room

Table 1
Physico chemical properties of natural pozzolan and ordinary portland cement

	Ordinary portland cement	Natural pozzolan
SiO_2	20.58	46.4
Al_2O_3	4.90	17.5
Fe_2O_3	4.70	10.5
CaO	62.8	10.5
MgO	0.53	3.8
SO_3	2.28	0.4
Residue	0.42	–
Free lime	2.17	–
Na_2O	–	3.4
K_2O	–	1.5
LOI	1.00	4.31
SSB (cm^2/g)	3100	3200
Glass content	–	>15%

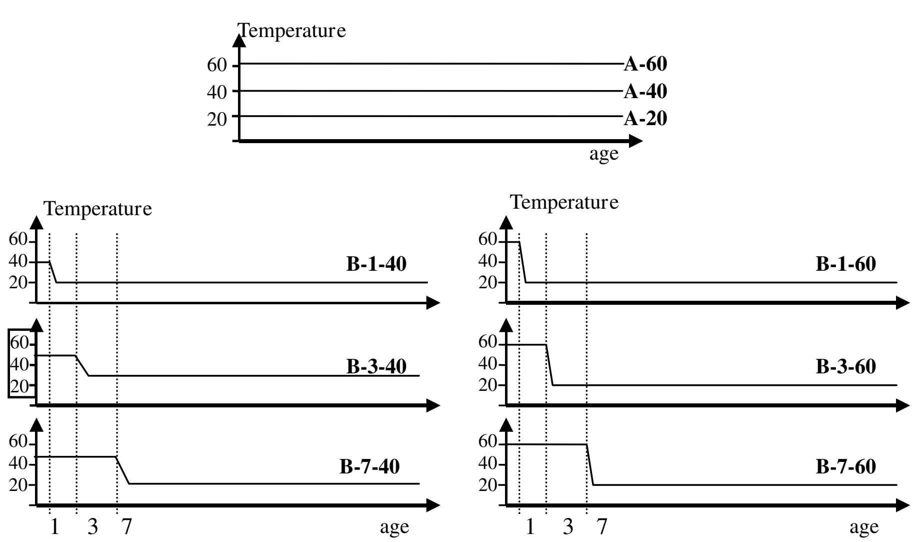

Fig. 1. Various type of temperature history used for curing.

Table 2
Compressive strength results of mortar for various curing temperature

Age in days	Set A														
	Temperature 20 °C					Temperature 40 °C					Temperature 60 °C				
	0	10	20	30	40	0	10	20	30	40	0	10	20	30	40
1	7.7	6.7	5.6	4.8	3.5	10.7	9.8	8.8	6.8	5.2	14.4	15.2	13.4	12.4	11.0
3	15.2	15.5	14.0	9.0	6.8	19.9	20.1	17.1	12.5	13.5	19.8	22.1	20.2	20.1	19.5
7	22.4	21.2	19.8	16.5	14.4	23.6	23.1	21.3	22.6	18.8	23.8	26.1	25.0	25.4	24.5
28	30.3	33.1	33.3	30.7	26.1	28.1	31.0	32.0	24.0	22.2	27.0	30.4	31.2	26.7	25.7
90	34.0	39.6	40.2	33.8	30.3	31.1	37.2	39.0	33.3	29.1	28.8	33.4	35.0	31.3	28.0
	Set B														
	B-1-40	B-3-40	B-7-40	B-1-60	B-3-60	B-7-60									
$P = 20\%$															
28	31.4	30.9	33.2	25.4	26.8	27.6									
90	31.6	31.9	34.2	29.4	28.8	28.4									

temperature until compressive testing at different ages of 1, 3, 7, 28 and 90 days for set A and at 28 and 90 days for set B. The compressive strength results are summarized in Table 2, in which each value represents the average of four tests with error deviation less than 8%.

3. Results and discussion

3.1. Constant temperature (Set A)

It is shown that the strength of the pozzolan mortar is considerably less than that of the OPC mortar at early stage of hydration. This has been due to the slow pozzolanic reactions. Fig. 2 shows the development of the compressive strengths of the OPC and set A mortar containing 20% of natural pozzolan against the curing temperature changes. One-day strength of the OPC mortar increases linearly with the curing temperature followed by that of the pozzolan mortar. At 3 days, the compressive strength of the OPC mortar increases with the temperature up to 40 °C, beyond this value, the temperature has only a marginal effect; whereas the strength of pozzolan mortar increases up to 60 °C. At 7 days, the effect of the temperature on the compressive strength of the OPC mortar is insignificant while that of the pozzolan mortar strength increases linearly with temperature and exceeds the OPC mortar strength at a temperature of 60 °C. At 28 days, the compressive strength of the pozzolan mortar exceeds that of the OPC mortar but decreases linearly with the curing temperature. At 90 days, the compressive strength of the pozzolan mortar distinctly exceeds that of the OPC mortar and decreases with the curing temperature according to a parabolic shape contrary to that of the OPC mortar which decreases linearly. Topçu and Toprak [14] observed similar results with the decrease in compressive strength for the OPC concrete subjected to curing at different temperature at the 28 days. On the other hand, Monso et al. [12] found parabolic curves of strength change with fly ash addition. Mortars with other replacement rates take the same evolution of the compressive strength as the 20% pozzolan mortar.

3.2. Variable temperature (Set B)

Fig. 3 shows the variation of the compressive strength development of set B mortar with curing time and temperature higher than 20 °C. The loss of strength at 28 and 90 days is very noticeable for the mortar specimens under

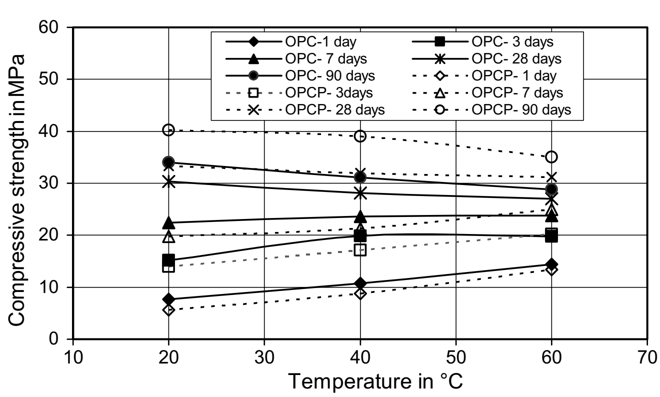

Fig. 2. Compressive strength evolution according to the curing temperature for OPC mortar and OPCP mortar containing 20% of natural pozzolan.

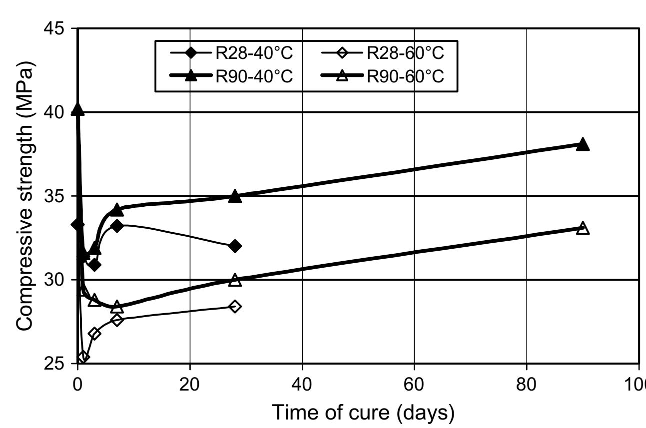

Fig. 3. Variation of compressive strength at 28 and 90 days according to the time under high temperature.

elevated temperature at early age. However, the presence of the pozzolan in the mixtures reduces the negative effect of the temperature rise, and the longer the spresemens are exposed the lesser the effect would be. For one day of curing at higher temperature, the pozzolanic mortar undergoes a great loss of its ultimate strength. More than one day of curing, the pozzolanic reaction reduces the decrease of the compressive strength expressed by raised slices. Thus, the ultimate strength of pozzolanic mortar increases, but it still remains lower than that cured at ambient temperature. These results are confirmed by Kim et al. [15]. They reported that the increase of curing temperature before 7 days has great effect on strength development at later age.

4. Analysis of the results

An approach suggested by Knudsen (1982) and adopted by the ASTM Standards [16] to estimate the concrete strength gives that the kinetic of the hydration can be predicted by the following equation

$$S = S_u \frac{t - t_0}{(t - t_0) + t_{50}} \quad (1)$$

with S being the property of the cement (hydration heat, shrinkage, strength,.. etc.); S_u the ultimate value; t_0 the age when strength development is assumed to begin (can be assimilated by the setting time in days); and t_{50} the necessary time to reach 50% of the ultimate value in days (half age of strength).

This equation can be written as

$$\frac{1}{S} = \frac{1}{S_u} + \frac{t_{50}}{S_u}\left(\frac{1}{t - t_0}\right) \quad (2)$$

The interrelationship of the results gives linear curves ($Y = b + aX$), from which we can deduce the half age of strength t_{50}. The obtained results are represented in Table 3 for different temperatures and the different replacement rates of the pozzolan. For the calculation, strengths at 90 days are taken as ultimate strength.

4.1. Half age of strength

The half age of strength represents the corresponding age for which the strength reaches the half of its ultimate value. The experimental results perfectly agree with the previous studies which show slower hydration of the cement with increases of pozzolanic admixtures, these differences decrease with the temperature rise. Fig. 4 shows linear variations, from which we can write

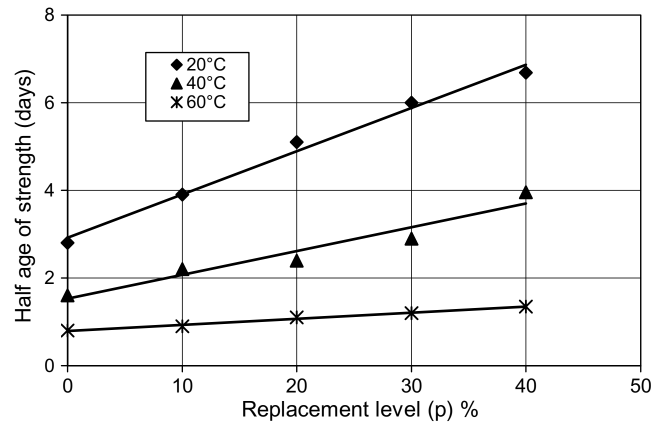

Fig. 4. Half age of strength according to the replacement level.

$$t_{50}(p) = t_{50}(0\%) + k_p p \quad (3)$$

where p is the replacement level to the cement content and k_p is a coefficient given according to the curing temperature.

4.2. Activation energy

The activation energy defines the temperature sensitivity of a mixture. It is used in the equivalent age maturity method to convert concrete properties that are determined at the reference temperature to any other curing temperature. It is very interesting to use this concept to show the difference between the activation of the pozzolan and that of OPC. Considering the hydration reaction as a simple reaction, we can evaluate its kinetic by the Arrhenius relation [16]

$$k = A \exp\left(-\frac{E_a}{RT}\right) \quad (4)$$

with k being the kinetic constant; E_a the activation energy; R the constant of the perfect gases ($R = 8.314$ J/mole K°); T the temperature in Kelvin degree; and A the constant.

For two temperatures T_1 and T_2, the same degree of evolution of the hydration must be achieved at the times t_1 and t_2 from which we can write [16–18]

$$\exp\left[\frac{E_a}{R}\left(\frac{1}{T_2} - \frac{1}{T_1}\right)\right] = \frac{t_2}{t_1} \quad (5)$$

Table 3
Half age of strength, ultimate strengths and activation energy results for various curing temperature and replacement level

Replacement level (p) (%)	Ultimate strength S_u (MPa)			Half age of strength t_{50} (days)			Activation energy (kJ/mole)
	20 °C	40 °C	60 °C	20 °C	40 °C	60 °C	
0	34.0	31.1	28.8	2.8	1.6	0.8	24.5
10	39.6	37.2	33.4	3.9	2.2	0.9	26.5
20	40.2	39.0	35.0	5.1	2.4	1.1	28.5
30	33.8	33.3	31.3	6.0	2.9	1.2	30.6
40	30.3	29.1	28.0	6.9	3.9	1.3	32.6

Table 4
Activation energy values for several binders

Reference	OPC Cement (kJ/mol)	Blended cement (kJ/mol)	Observations
Roy et Idorn [2]	44.3	49.1	Cement with 50% of slag
Shindler [18]	45.2	49.9	Type I and type III cement
Ma et al. [5]	39.0	49.3	Cement with 65% of slag
Ma et al. [5]	39.0	30.4	Cement with 7.5% of silica fume
This paper	24.4	32.6	Cement with 40% of natural pozzolan

The values of the activation energy E_a can be calculated by using the time corresponding to 50% of the degree of hydration (t_{50}) for the two given temperatures. Table 3 presents the energy found for the different mixtures of the cement. It is obvious that the presence of the pozzolan attributes additional energy (around 33%) to the mixtures. Because of this additional energy, the kinetic of hydration becomes slow and improves the uniformity in the distribution of the hydrate products.

If we make a comparison of the activation energy found with those given in the literature for the different mineral admixtures used in the world, see Table 4, we can conclude that this pozzolan gives a great improvement than those of the other admixtures. This energy gives to the cement a desirable property allowing it to be considered among the supplementary cementitious materials used in Algeria.

4.3. Effect of the replacement level

The presence of the natural pozzolan in the composition of the binder modifies the ultimate strength considerably. This has led toward an optimal use of this product in order to acquire best performances with respect to the type of suitable cure. The variation of the ultimate strength obtained for different curing temperatures shows parabolic curves giving an optimal replacement rate correspondent to the maximum strength. This optimum pozzolan content is estimated as 15% and 20% for 20 °C and 60 °C curing temperature, respectively. This natural pozzolan has an acceptable optimal replacement rate than that in the world. Pekmezci and Akyuz [19] found that the optimum pozzolan/cement ratio to obtain maximum strength is approximately 0.28. Shannag and Yeginobali [20] consider that the mix with 25% natural pozzolan was the optimum.

Fig. 5 illustrates the strength variation at different temperatures. This variation decreases with the increase of pozzolan replacement level. This encourages their uses in hot climate without considerable risk of loss of the ultimate strength. According to the results the evolution of the ultimate strength follows a parabolic law described by the following equation with a and b coefficients of the polynomial given according to the curing temperature.

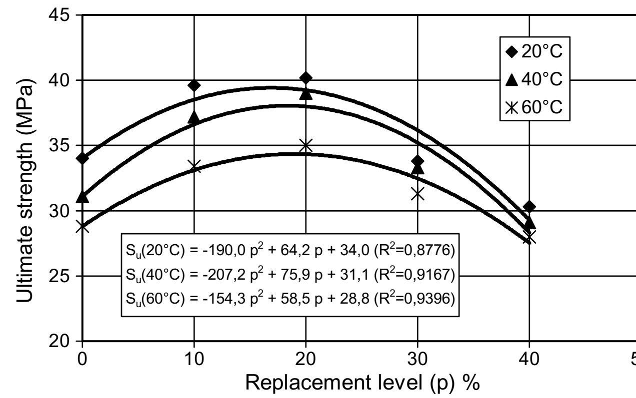

Fig. 5. Ultimate strength according to the replacement level.

$$S_u(p) = S_u(OPC)(ap^2 + bp + 1) \tag{6}$$

4.4. Ultimate strength

In the context of a very simple approach, and considering the influence of the temperature on the development of the concrete strength, we may assume that the ultimate strength is very close to the strength at 90 days. Generally, these strengths can be represented by a linear decreasing function with temperature rise according to the following equation [17]

$$S_u(T) = S_u(20\,^\circ\mathrm{C})[1 - k_0(T - 20)] \tag{7}$$

where k_0 is the slope of the normalized model that corresponds to the average of the values obtained from the literature equal to 10.2×10^{-3}.

Both of the mineral admixture and the curing temperature have an enormous effect on the ultimate strength. For an OPC mortar, the ultimate strengths depending on the curing temperature decrease linearly (Fig. 6). In contrast, for the pozzolan mortar these curves decrease in a form of a parabola. The value of the k_0 coefficient in Eq. (7) needs a correction which takes into account the presence of the natural pozzolan. This coefficient is not anymore a constant and possesses a parabolic variation according to the replacement rate used, from which therefore Eq. (7) can be written as

$$S_u(T) = S_u(20\,^\circ\mathrm{C})[1 - (k_0 + k_1)(T - 20) - k_2(T - 20)^2] \tag{8}$$

with

$$k_0 = 3.9 \times 10^{-3} \quad R^2 = 0.99$$
$$k_1 = (70.8p^2 - 36.6p) \times 10^{-3} \quad R^2 = 0.91$$
$$k_2 = (-2p^2 + 0.8p) \times 10^{-3} \quad R^2 = 0.92$$

k_0 characterizes the OPC mortar, on the other hand k_1 and k_2 quantify the pozzolan effect according to the replacement level. The interrelationship coefficients R^2 are near to the unit which justifies the choice of the model.

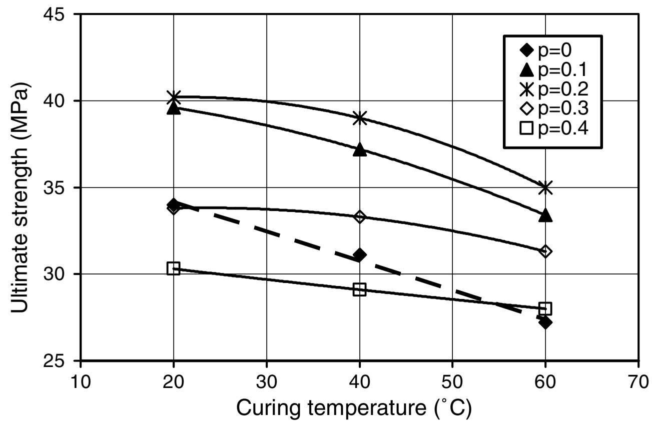

Fig. 6. Ultimate strength according to the curing temperature.

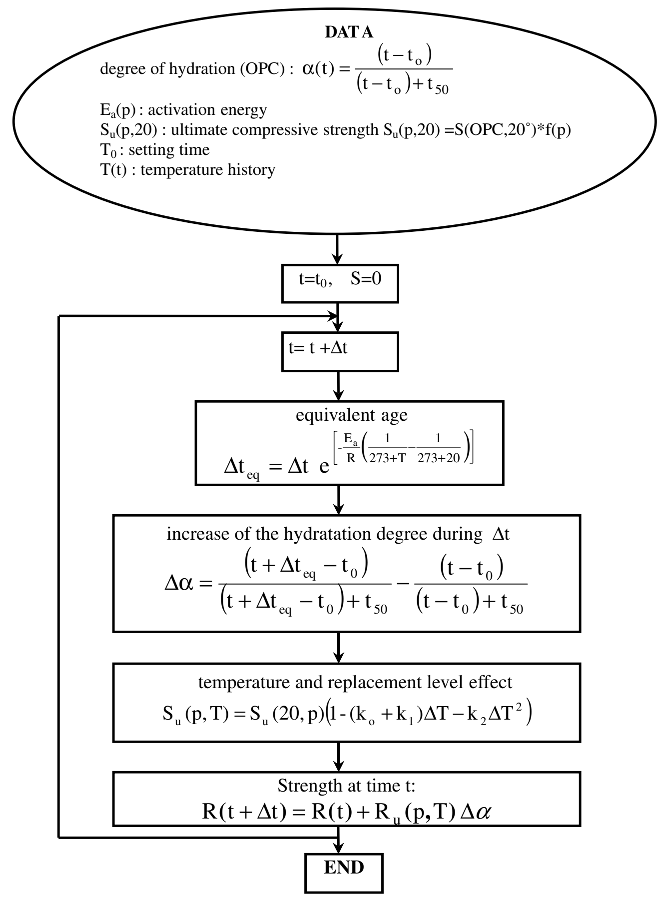

Fig. 7. Diagram chart for prediction of compressive strength under temperature history.

5. Prediction of the strength

5.1. Degree of hydration

The degree of hydration of the cement can be represented as the ratio of the strength at a given age on the ultimate strength. From Eq. (1), we deduct Eq. (9) to predict the evolution of the degree of hydration α with the time.

$$\alpha(t) = \frac{S(t)}{S_u} = \frac{(t - t_0)}{(t - t_0) + t_{50}} \tag{9}$$

5.2. Equivalent age method

The equivalent age method is used to compute the time at the reference temperature T_r in which the mixture reaches the same degree of hydration as under any other temperature T [16–18]. The ratio between the equivalent time (for T_r) and the real time (for T) is the function of the two temperatures and its law is issued from the general law of acceleration of the chemical reactions established by Arrhenius

$$t_{eq} = t \ \exp\left[-\frac{E}{R}\left(\frac{1}{T} - \frac{1}{T_r}\right)\right] \tag{10}$$

This method will be used to predict the compressive strength of the mortar cured at a variable temperature. Since, we know the isotherm evolution of the hydration degree α (strength) under a reference temperature (20 °C) and the activation energy; it is possible then to deduce the evolution of the compressive strength for mortar exposed to any temperature history.

5.3. Diagram chart of prediction

The prediction of the compressive strengths of the mortar specimens or concrete containing some mineral additions exposed to the variable temperature can be made by the equivalent age method. For this, we need the evolution model of the hydration degree according to the time under a reference temperature (Eq. (9)), the modified ultimate strengths taking into account the presence of natural pozzolan (Eq. (6)), the curing temperature (Eq. (8)) and the activation energy (Table 3). Thus, to predict the strength, the flowchart, illustrated in Fig. 7, is followed step by step for any replacement rate of the natural pozzolan and temperature history.

5.4. Calculated results

In order to validate this method, we use the B set results of the pozzolan mortar hardening under a variable temperature. We have the data of the OPC mortar hardening under a reference temperature 20 °C. In using the diagram chart (see Fig. 7), we convert the real interval of time Δt in equivalent time according to the curing temperature then we calculate the increase of the strength during this interval. The calculated results are presented in Table 5, in which we notice that the results are acceptable and agree with the experimental results with small error deviation around 5%. The equivalent time method including the correction of the ultimate strength according to the curing temperature and the presence of the mineral admixture is a very reliable mean to predict the compressive strength under any temperature history.

Table 5
Experimental and predicted strength by the equivalent age method

Age	Type of cure	S_{exp} (measured) (MPa)	S_{pre} (predicted) (MPa)	$100\frac{S_{pre}-S_{exp}}{S_{exp}}\%$
28 days	B-0-40	33.3	34.0	2.0
	B-1-40	33.1	33.8	1.9
	B-3-40	32.5	33.8	3.8
	B-7-40	34.7	34.1	−1.7
	B-28-40	32.0	35.7	11.4
90 days	B-0-40	40.2	38.0	−5.4
	B-1-40	37.3	37.8	1.5
	B-3-40	35.9	37.7	4.8
	B-7-40	35.4	37.6	6.2
	B-90-40	39.0	38.4	−1.7
28 days	B-0-60	33.3	34.0	2.0
	B-1-60	30.1	32.7	8.7
	B-3-60	31.9	32.4	1.6
	B-7-60	32.8	32.9	0.4
	B-28-60	31.0	34.4	11.1
90 days	B-0-60	40.2	38.0	−5.4
	B-1-60	34.4	36.3	5.5
	B-3-60	35.9	35.3	−1.8
	B-7-60	34.1	34.9	2.4
	B-90-60	35.0	35.5	1.4

6. Conclusion

This study has allowed to valorise the natural pozzolan and to analyse its behaviour under elevated temperatures. As all cementitious materials, we notice that the pozzolan cement is characterized by a weak strength at early age followed by an increase exceeding especially that of the OPC after 28 days. The optimal replacement rate is about 15% for a normal temperature and till 20% for an elevated temperature.

When a part of the cement is substituted by the natural pozzolan, the experimental results show an increase of the activation energy. This explains the slowness of its hydration and its beneficial effect in hot climate.

Under elevated temperatures, the fast formed hydrates cause a great porosity and a loss of ultimate strength as reported by several authors. This phenomenon improves when we substitute part of cement by the natural pozzolan. The ultimate strength depending on the curing temperature is expressed by a polynomial model contrary to the linear model in the case of the OPC.

When the pozzolan cement is exposed to elevated temperatures at early age and the curing time is less than 1 day, we notice substantial decrease of the ultimate strength. After 1 day, the natural pozzolan starts to contribute to improvement due to its pozzolanic reaction and great activation energy. This benefits their uses in hot climate without considerable risk of loss of the ultimate strength.

The equivalent age method is an efficient mean to predict the compressive strength evolution of the cement hardening under any temperature history. The results showed a perfect satisfaction between the calculation and the experimental data with error deviation less than 5%.

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