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Influence of elevated temperatures on the mechanical properties of blended cement concretes prepared with limestone and siliceous aggregates

A. Savva *, P. Manita, K.K. Sideris

Civil Engineering Department, Democritus University of Thrace, P.O. Box 252, Xanthi 67100, Greece

Abstract

The investigation performed was aimed at showing the influence of high temperatures on the mechanical properties and properties that affect the measurement by non-destructive methods (rebound hammer and pulse velocity) of concrete containing various levels (10% and 30%) of pozzolanic materials. Three types of Pozzolans, one natural pozzolan and two lignite fly ashes (one of low and the other of high lime content) were used for cement replacement. Two series of mixtures were prepared using limestone and siliceous aggregates. The W/b and the cementitius material content were maintained constant for all the mixtures. Concrete specimens were tested at 100, 300, 600 and 750 °C for 2 h without any imposed load, and under the same heating regime. At the age of 3 years, tests of compressive strength, modulus of elasticity, rebound hummer and pulse velocity were come out. Results indicate that the residual properties of concrete strongly depend on the aggregates' and the binder type. Relationships between strength of concrete as well as rebound and pulse velocity versus heating temperatures are established. The above results are evaluated to establish a direct relationship between non-destructive measurements and compressive strength of concrete exposed to fire.
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Keywords: Concrete compressive strength; High temperature; Modulus of elasticity; Limestone and siliceous aggregates; Rebound hammer; Pulse velocity; Fly ash; Natural pozzolan

1. Introduction

Concretes containing mineral admixtures are used extensively throughout the world for their good performance and for ecological and economic reason. The effect of high temperature on concrete containing fly ash or natural pozzolans has not been investigated in detail unlike fly ash concrete that has been under investigation since the 1960s. Researchers and investigators differ in their opinion regarding the changes in the properties of concretes, particularly in the range of 100–300 °C. Above 300 °C, there is a uniformity of opinion concerning a decrease in mechanical characteristics [1–5]. However, decreases in strength, reported in the literature reveal significant quantitative differences due to the variety of high temperature condition tested, and the variety of constituent materials of concrete used. It is recognized that the behavior of concrete subjected to high temperatures is a result of many factors [6,7]; such as heating rate, peak temperatures, dehydration of C–S–H gel, phase transformations, and thermal incompatibility between aggregates and cement paste. On the other hand, quality control of concrete, by means of non-destructive methods, in structures subjected to fire or not so high temperature exposure conditions, is not particularly easy to be carried out. The correlation already exists usually refers to the hydration age of 28 days.

The scope of this work is to provide experimental data on the residual mechanical properties of concretes subject to heat, containing pozzolanic materials, and to the properties that affect measurements by non-destructive methods (rebound hammer and pulse velocity). These properties are very important for the safe design of concrete and in the repair of concrete structures.

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* Corresponding author. Tel.: +30-2541079861; fax: +30-2541-0-29265.

E-mail address: asavva@civil.duth.gr (A. Savva).

2. Experimental details

Two series of concrete specimens were prepared. Specimens of the first series were made with crushed limestone while specimens of the second series were made with siliceous aggregates. The maximum nominal size and the grading curve were similar in both concretes 31.5 mm. The water to binder ratio ($W/b = 0.6$) and the binder (cement + pozzolanic material) content (300 kg/m^3) used were the same in both series. The binder was a normal Portland cement CEM I 42.5 N (OPC), as the control mix, and 6 blended cements prepared by the replacement (wt%) of OPC by three different pozzolans. One natural of volcanic origin, Milos earth (ME) and two Greek lignite fly ashes, Ptolemaida fly ash (PFA) and Megalopolis fly ash (MFA). MFA is a low calcium fly ash and has a significant pozzolanic activity, whereas PFA is a high calcium fly ash and has both pozzolanic and hydraulic activity [8]. MFA, PFA and ME contents of the mixes were either 10% or 30% by mass of binder. Physical and chemical properties of materials are given in Table 1 and the mineralogical composition of OPC, as calculated according to ASTM C150 [9], is presented in Table 2. The mix proportions of concretes are given in Table 3. No superplasticizer was used and slump values of all concretes were between 25 and 70 mm.

Mixing was carried out in a pan mixer. Specimens were either $150 \times 150 \times 150$ mm concrete cubes or 150×300 mm concrete cylinders. The cubes were used for compressive strength testing, while static modulus of elasticity was performed on cylinders, before and after heating. After a period of 28 days of moist curing (20 ± 2 °C, RH $\geqslant 95\%$), the specimens were placed in a curing room (20 ± 2 °C and $60 \pm 5\%$ relative humidity), as far as required for testing. At ages of 3, 7, 14, 28, 60 and 1095 days, specimens were tested by means of the rebound hammer and pulse velocity and the compressive strength was determined. At each age, a set of 10 rebound

Table 2
Mineralogical composition of Portland cement (OPC), according to [9]

Phase	Percentage (%)
C_3S	57.0
C_2S	17.4
C_3A	11.5
C_4AF	5.9

numbers and a set of two determinations of pulse velocity were obtained from each of the three cubes. The compressive strength (a mean value of three cubes), as a function of hydration age, is given in Table 4. The average values of strength and in-place test results (rebound number and pulse velocity) at each strength level (age) were treated as one data pair. The data pairs were plotted using the in-placed test values as the independed value and the compressive strength as the depended value. Regression analysis was performed on the data pairs to obtain the best-fit estimate of the strength relationship. The relationship between compressive strength and rebound number ($f_{20} - R_{20}$) is given in Figs. 1 and 2, for limestone and siliceous concretes respectively, and between compressive strength and pulse velocity ($f_{20} - V_{20}$) is presented in Figs. 3 and 4, for limestone and siliceous concretes respectively.

After 3 years, the specimens were heated in an electric furnace. An electronic controller controlled the temperature and the temperatures were continuously recorded by thermocouples, incorporated in seven specimens, along their axis. The rate of heating was set at 2.5 °C per minute. Four temperature levels were examined for the compressive strength: 100, 300, 600 and 750 °C, and two, 300 and 750 °C, for the modulus of elasticity. Two cylinders and three cubes of each of the 14 mixtures were heated for each temperature level. The heating period was 2 h at maximum temperature and after heating was completed, the specimens were

Table 1
Chemical composition and physical properties of cementitious materials

	OPC	Ptolemaida fly ash (PFA)	Megalopoli fly ash (MFA)	Milos earth (ME)
Chemical composition (%)				
SiO_2	21.05	27.20	47.08	65.90
Al_2O_3	5.60	11.22	18.62	15.70
Fe_2O_3	1.95	4.14	6.72	3.95
CaO	63.70	38.43	17.50	4.00
MgO	2.40	3.50	3.81	1.40
SO_3	3.05	6.31	3.28	–
Na_2O	0.25	1.35	0.68	5.65
K_2O	1.05		1.52	
Free CaO	1.10	12.34	–	–
Loss on ignition	0.75	4.30	2.80	3.10
Physical properties				
Specific surface (m^2/kg)	330	475	503	555

Table 3
Mixture proportions

Mix	W/B	OPC	MFA	PFA	ME	Water	Fine aggregates	Coarse aggregates
		(kg/m^3)						
OPC-S[a]	0.60	300	–	–	–	180	807.6	1040.4
10 MFA-S	0.60	270	30	–	–	180	807.6	1040.4
30 MFA-S	0.60	210	90	–	–	180	807.6	1040.4
10 PFA-S	0.60	270	–	30	–	180	807.6	1040.4
30 PFA-S	0.60	210	–	90	–	180	807.6	1040.4
10 ME-S	0.60	270	–	–	30	180	807.6	1040.4
30 ME-S	0.60	210	–	–	90	180	807.6	1040.4
OPC-L[b]	0.60	300	300	–	–	180	794.7	1095.3
10 MFA-L	0.60	270	30	–	–	180	794.7	1095.3
30 MFA-L	0.60	210	90	–	–	180	794.7	1095.3
10 PFA-L	0.60	270	–	30	–	180	794.7	1095.3
30 PFA-L	0.60	210	–	90	–	180	794.7	1095.3
10 ME-L	0.60	270	–	–	30	180	794.7	1095.3
30 ME-L	0.60	210	–	–	90	180	794.7	1095.3

[a] S = siliceous aggregates, $d_{max} = 31.5$ mm.
[b] L = limestone aggregates, $d_{max} = 31.5$ mm.

Table 4
Compressive strength (MPa) of concretes at 20 °C

Mixture	Days					
	3	7	14	28	60	1095
OPC-L	28.3	33.4	35.3	40.9	42.8	48.9
10 MFA-L	27.3	34.1	37.2	42.4	46.1	51.6
30 MFA-L	21.4	27.2	33.4	37.5	43.5	46.1
10 PFA-L	28.7	35.5	36.6	41.7	41.8	40.7
30 PFA-L	21.7	29.5	35.2	40.2	40.9	44.5
10 ME-L	23.3	33.0	36.4	38.7	40.4	43.0
30 ME-L	18.1	22.8	25.8	29.9	33.7	42.9
OPC-S	20.1	29.1	33.2	34.7	36.4	53.3
10 MFA-S	17.6	27.1	31.0	38.0	41.6	54.7
30 MFA-S	15.0	21.9	27.1	34.6	42.3	42.3
10 PFA-S	21.8	30.3	36.0	41.2	41.5	45.8
30 PFA-S	18.3	25.9	33.0	39.1	42.3	47.6
10 ME-S	16.8	24.2	29.0	32.9	38.2	40.3
30 ME-S	11.8	17.9	21.9	26.2	31.3	38.7

remained until the furnace cooled down to room temperature. The cooling rate was about 0.4 °C/min. After the heating/cooling cycle, the specimens were tested for residual compressive strength, modulus of elasticity, rebound values and ultrasonic pulse velocity. In parallel, unheated specimens, selected at random, were tested and the compressive strength (mean value of three cubes) and the modulus of elasticity, (mean value of two cylinders), were taken as the strength f_{20} and the modulus of elasticity E_{20} of the mixtures at 20 °C.

Rebound number was measured by means of a rebound hammer (Schmidt type) according to ASTM C 805 [9] and pulse velocity (by means of a Pundit) according to ASTM C 597 [9]. The R number was calculated as the mean of 10 measurements, taken from four opposite faces of each of the three cubes and pulse velocity V, as the mean of two measurements, taken from the two opposite faces of each of three cubes.

3. Results and discussion

3.1. Compressive strength of concrete

The residual compressive strength after heating at different temperatures T was expressed as a ratio f_T/f_{20}, where f_T is the strength after heating at T °C and f_{20} is the initial strength of concrete at 20 °C. The strength ratio f_T/f_{20} as a function of the specimens' temperature T is shown in Figs. 5 and 6 for limestone and siliceous aggregates respectively, and versus the binder type for both aggregates, in Fig. 7.

From the viewpoint of strength loss, there were three temperature ranges: 20–100 °C, 100–300 °C and 300–750 °C. The strength loss was different between each of these temperature ranges.

Up to 100 °C, the initial strength was only slightly altered, depending on the binder type. The changes of
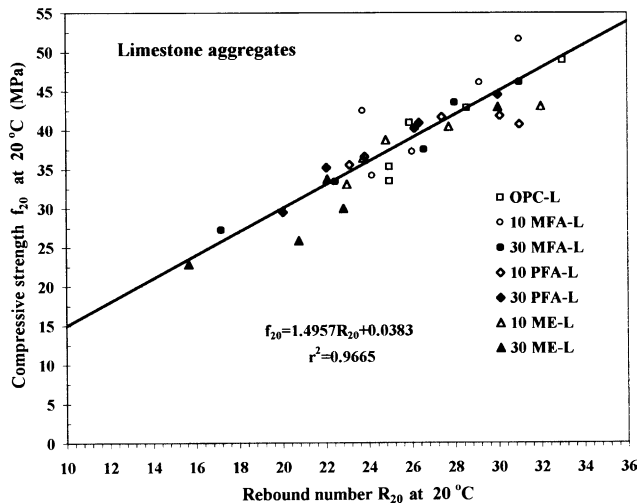
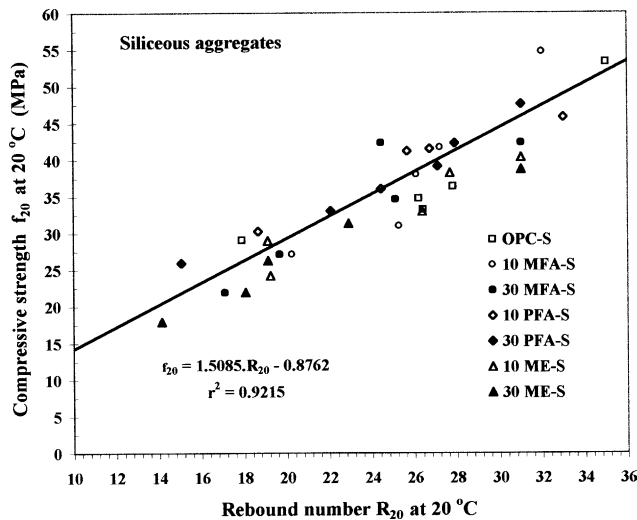Fig. 1. Compressive strength of limestone concretes versus rebound number at 20 °C.

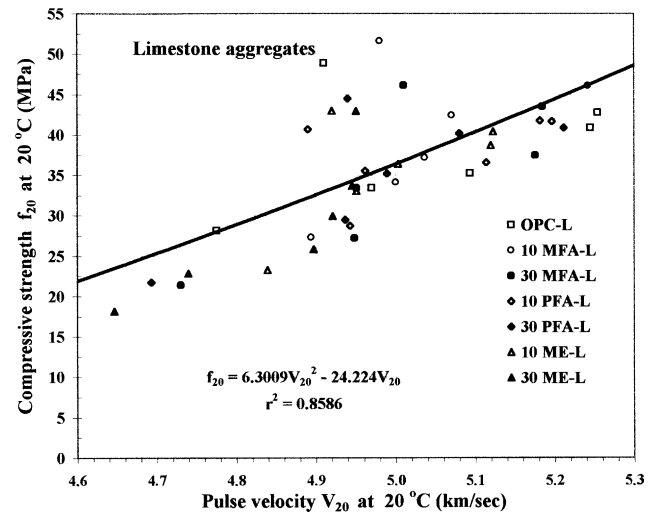Fig. 2. Compressive strength of siliceous concretes versus rebound number at 20 °C.

Fig. 3. Compressive strength of limestone concretes versus pulse velocity at 20 °C.

Fig. 4. Compressive strength of siliceous concretes versus pulse velocity at 20 °C.

the initial strength ranged from −9% to +8% for limestone concretes and from −3% to +24% for siliceous ones. Limestone concretes with OPC or 10 MFA had a reduction in strength of the order of 1% and 9% respectively, while the respective reduction for the siliceous concretes was 11% and 5%. Thirty MFA with limestone aggregates and 30 PFA with siliceous aggregates also had a reduction in strength (5% and 3% respectively). The others mixtures had an increase in strength, which was higher for concretes made with siliceous aggregates. The concrete includes capillary water, physically absorbed water and interlayer and chemically bound water in C–S–H and $Ca(OH)_2$. During heating the cement paste dried and water was free to be driven out. From the ambience temperature to 100 °C, the water that escapes is the free water. The mixtures
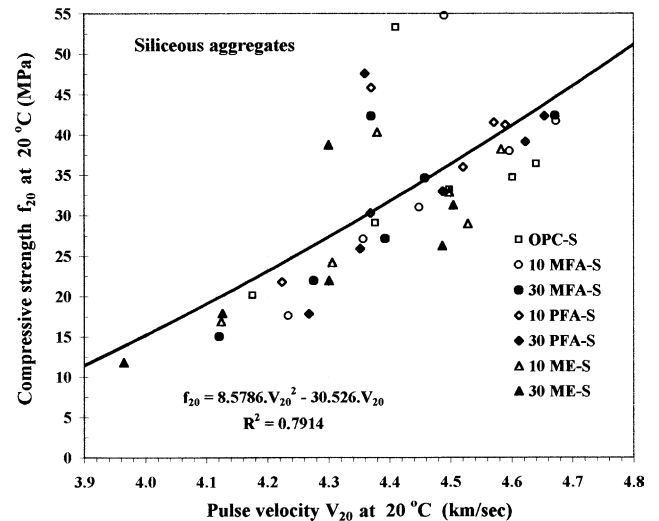
were 3 years old, so there was not much free water and no significant differences between the mixtures were observed.

At 300 °C, the change of strength ranged from about −8% to +14% of the initial strength for limestone and from 5% to 39% of the initial strength for siliceous concretes, depending on the binder type. The concretes made with OPC or 10% MFA had the same changes in strength: a slight reduction of 7–8% for limestone aggregates and a slight increase of 5–6% for siliceous aggregates. The initial strength of the other mixtures was increased from 16 to 39% for siliceous and only from 6 to 14% for limestone concretes. Therefore, up to 300 °C, the best performance was given by pozzolanic concretes and concretes made with siliceous aggregates.
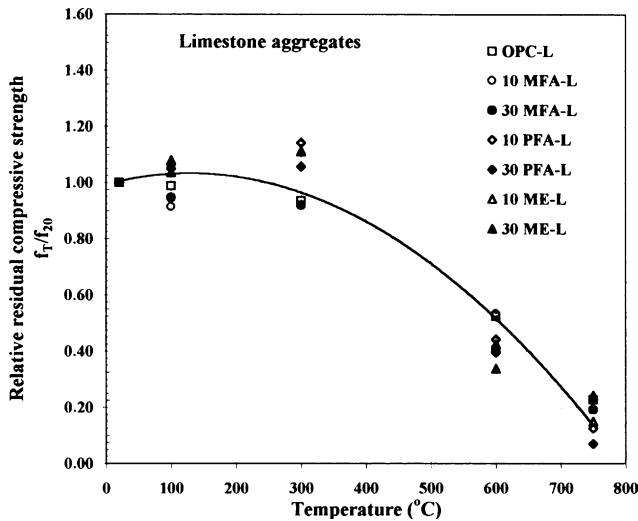
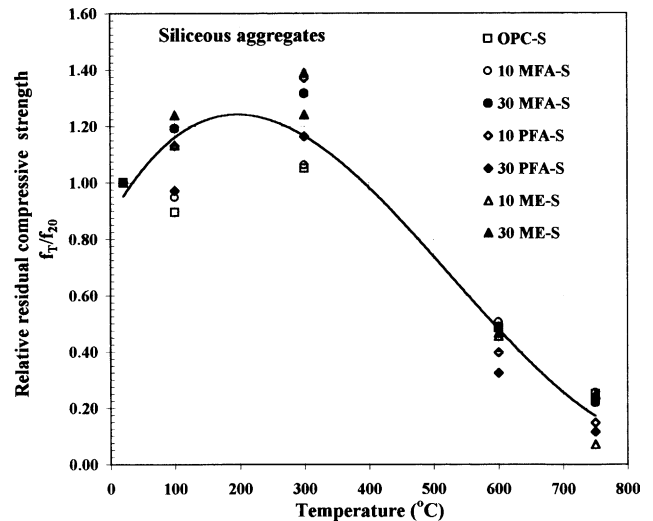Fig. 5. Relative residual compressive strength of limestone concretes.

Fig. 6. Relative residual compressive strength of siliceous concretes.

The water (in the form of steam) is eliminated most intensively [10], and affects the surrounding phases of cement paste. Mainly due to flow resistance and high temperature, steam creates a high pressure in the paste. In consequence, the so-called condition for internal autoclaving [6] appears in the cement paste and the result is an additional hydration of anhydrated cement grains [11]. For the pozzolanic material an additional tobermorite gel was formed as a result of the pozzolanic reaction of $Ca(OH)_2$ in OPC, with reactive silica in pozzolanic material. Lea and Straddling [12] also indicate a strength increase for temperatures up to 300 °C. The fact that higher strength was gained of concrete made with siliceous aggregates perhaps is due to the bond strength (cement- aggregate bond) that is higher in the case of concrete made with siliceous aggregates [13,14].

With further increase in temperature between 300 and 600 °C the loss of strength became more significant. Concrete had a reduction in strength ranging from 66%
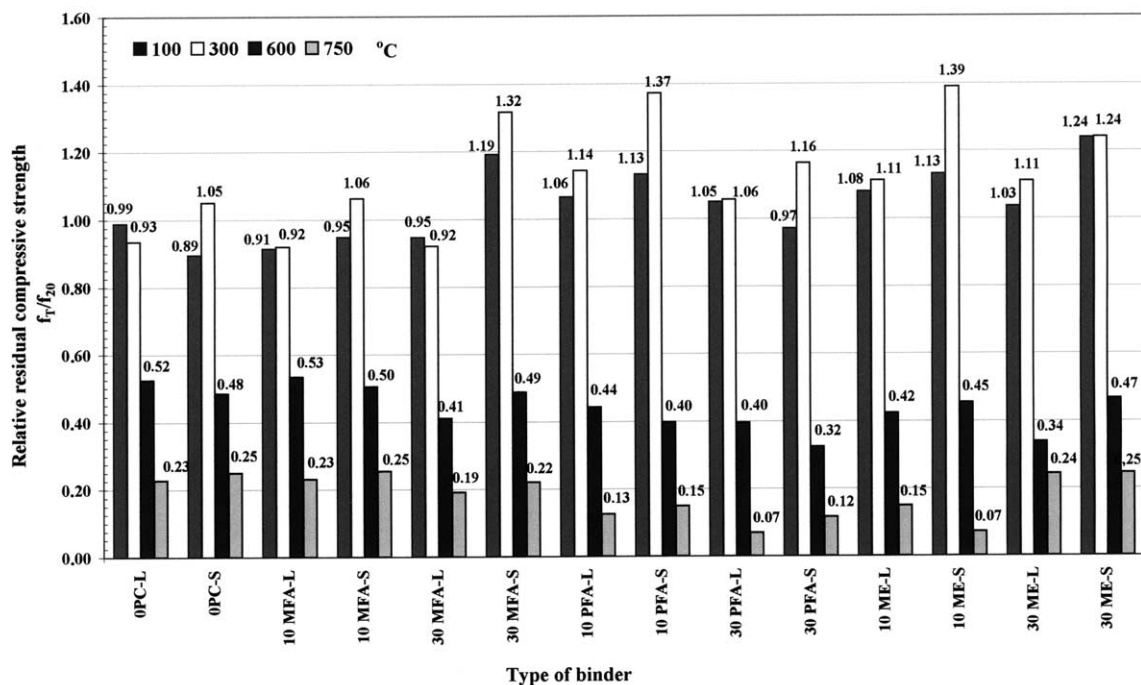

Fig. 7. Relative residual compressive strength as a function of the binder type.

to 47% for limestone and from 68% to 51% for siliceous mixtures. The temperature region above 300 °C is characterized by the decomposition of $Ca(OH)_2$ (above 400 °C). At about 600 °C all the mixtures were almost completely dehydrated. OPC and 10% MFA had the same behavior for both aggregates. Concretes made with natural pozzolans (ME) and limestone aggregates and the PFA mixtures for siliceous aggregates had the highest loss of the initial strength (58–68%). At these temperatures OPC showed better results for both aggregates and the greater the percentage of the replacement of OPC became, the greater the reduction in initial strength tended to be.

At a temperature over 600 °C, all tested concretes suffered deterioration and only a small part of the initial strength was left, ranging from 7% to 25% for all mixtures. In this case, the binder, that is the main source of concrete strength, is bound to undergo dehydration of the C–S–H gel and lose its cementing ability [14–16]. At this temperature, transformation of quartz (570 °C) and decarbonation of limestone also occurs. Concretes with limestone aggregates had a slightly higher reduction of strength. Therefore, the temperature range between 300 and 750 °C may be regarded as critical to the strength loss of concrete.

3.2. Modulus of elasticity

The residual modulus of elasticity after heating at different temperatures T was expressed as a ratio E_T/E_{20}, where E_T is the modulus of elasticity after heating at T °C and E_{20} is the initial modulus of elasticity of concrete at 20 °C. In Figs. 8 and 9 the tests results for E_T/E_{20} can be seen as a function of the temperature and in Fig. 10 as a function of the binder type.
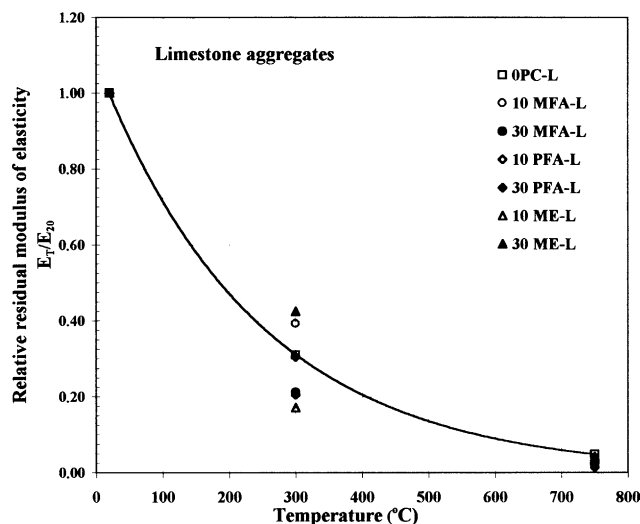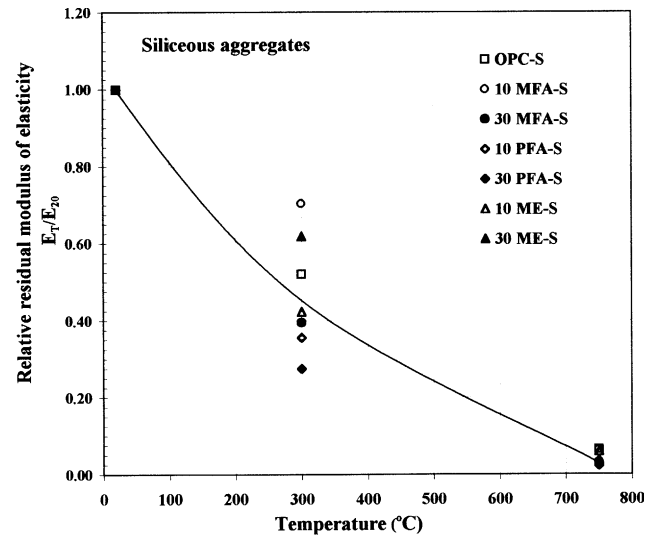

Fig. 9. Relative residual modulus of elasticity of siliceous concretes.

A continuous drop in the modulus was noticed at all the temperatures.

At the temperature of 300 °C, the modulus of elasticity of limestone concrete with OPC had lost about 69% of its initial value, while the respective decrease for concrete with pozzolanic materials ranged from 57% to 83%. The corresponding decrease for the siliceous concretes with OPC was 48% and it ranged from 30% to 73% for the pozzolanic concretes. It is also notable that at 300 °C, the relative residual modulus of elasticity of siliceous concretes was higher than those of limestone mixtures. It seems that at in the region near 300 °C, the stiffness of limestone concretes was more affected than that of siliceous concretes. The residual modulus of elasticity was higher for 10 MFA and 30 ME concretes, followed by pure OPC (both aggregates).

At 750 °C, the reduction in the modulus of elasticity ranged from 94% to 99%, no matter which type of binder or of aggregate was used.

Differences among concretes with various pozzolanic materials were not very clear. But it was clearly noticeable during the compression tests that, the specimens' failure was less brittle and more gradual when increasing the temperature test.

Ghosh and Nasser [17] found that a decrease in elastic modulus was due to a gradual deterioration of the binding matrix with a rise in temperature. In the same work, the transformation of the matrix seemed to be purely physical at lower temperatures up to 71 °C, wherein small lumps of CSH gel coalesced together to form a larger deforming gel mass. However, at further higher temperatures, chemical transformation of the gel was noticed where the fairly dense lumps of CSH gel underwent changes to form a loose white matrix that brought about loss of elasticity of concrete.
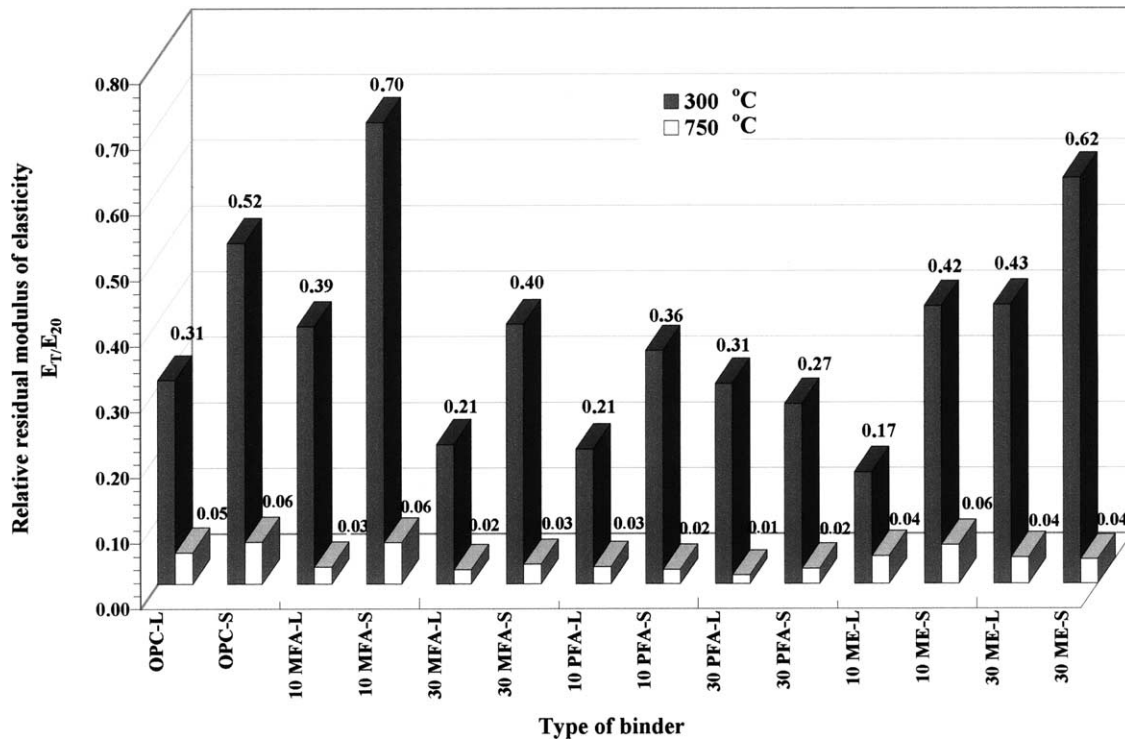

Fig. 8. Relative residual modulus of elasticity of limestone concretes.

Fig. 10. Relative residual modulus of elasticity as a function of the binder type.

3.3. Rebound number

The residual rebound number after heating at different temperatures T was expressed as a ratio R_T/R_{20}, where R_T is the rebound number after heating at T °C and R_{20} is the initial rebound number of concrete at 20 °C. In Figs. 11 and 12, the ratio R_T/R_{20} versus the specimen temperature T is presented, for limestone and siliceous concretes respectively.
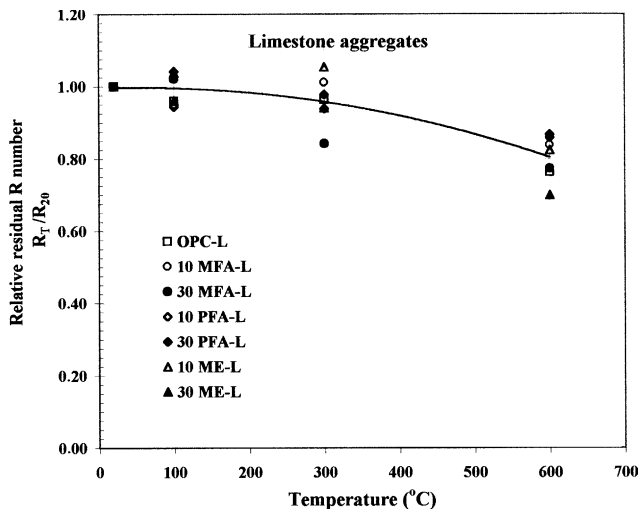

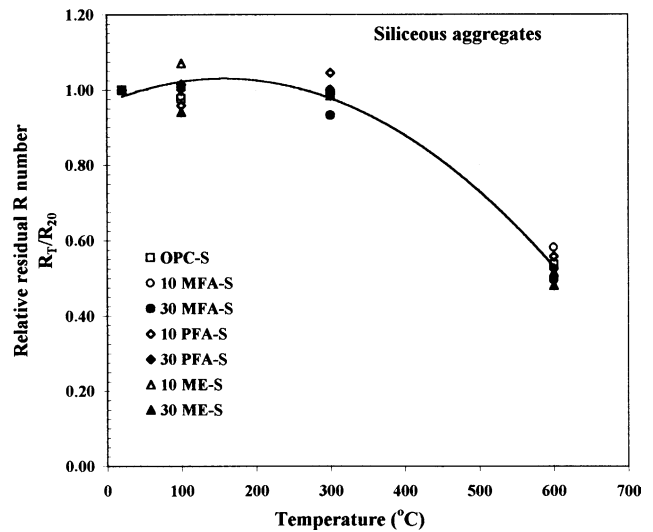Fig. 12. Relative residual rebound number of siliceous concretes.

Fig. 11. Relative residual rebound number of limestone concretes.

The rebound measurements present no significant changes in the range up to 300 °C. They even show a small increase. The rebound values are influenced mainly by the condition of the surface of concrete to depth not exceeding 3 cm approximately. Since temperature increase up to 300 °C causes drying and hardening of the surface layer, rebound measurements present a small increase.

For higher temperature, intensive internal cracking and chemical decomposition (carbonation or transformation of quartz) of the surface layer become more significant and R numbers show a higher decrease. At 600 °C, the rebound number R decrease was higher for siliceous concretes.

At the temperature of 750 °C it was impossible for measurements to be taken, due to the deterioration of the surface of the specimens.

3.4. Pulse velocity

The residual pulse velocity after heating at different temperatures T was expressed as a ratio V_T/V_{20}, where V_T is the pulse velocity after heating at T °C and V_{20} is the initial pulse velocity of concrete at 20 °C. In Figs. 13 and 14, the ratio V_T/V_{20} versus the specimen temperature T is presented, for limestone and siliceous concretes respectively.

Pulse velocity has a continuous drop as the temperature was increased.

Up to 300 °C, the percentage replacement of OPC and the binder or aggregate type proved to have small influence on the results. At higher temperatures, in mixtures with siliceous aggregates a significant higher reduction of pulse velocity was noticed. It is obvious that the transmission of pulse waves through a concrete mass is highly influenced by the microcracking of concrete. Thus, the decrease in pulse velocity with increasing temperature is a sensitive measure of the progress of cracking in the material.

3.5. Strength determination by non-destructive methods

In principle the above-mentioned experimental results permit the estimation of the residual strength of concrete
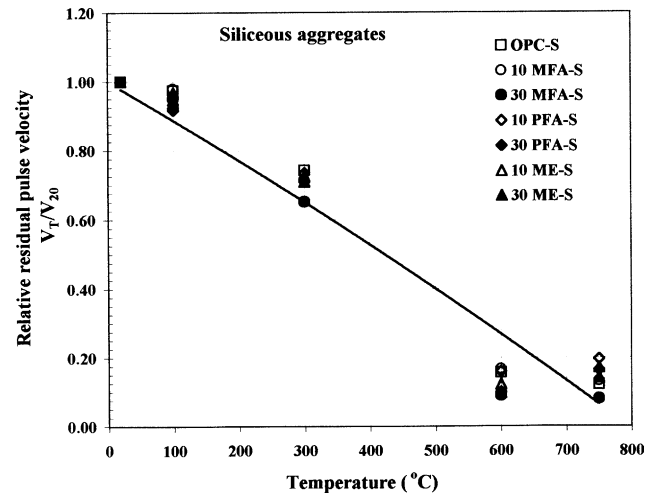

Fig. 14. Relative residual pulse velocity of siliceous concretes.

after heating at a given temperature T °C, by means of non-destructive methods.

The value R_T (or V_T), obtained after heating concrete at temperature T °C, corresponds to an initial value R_{20} (or V_{20}) before heating, given in Figs. 11 and 12 (or in Figs. 13 and 14). On the basis of this estimated initial value R_{20} (or V_{20}), one could find the corresponding initial strength f_{20} of concrete at 20 °C (before heating), by means of existing best fit of the strength relationship in Figs. 1 and 2 (or in Figs. 3 and 4). Nevertheless under the influence of heating, the strength will be decreased to the values f_T given in Figs. 5 and 6 for limestone and siliceous aggregates respectively, or in Fig. 7 for both aggregates. This process is the basis for drawing the lines in Figs. 15 and 16 (rebound number) and in Figs. 17 and 18 (pulse velocity), relating directly the strength f_T to the measured R_T (or V_T).
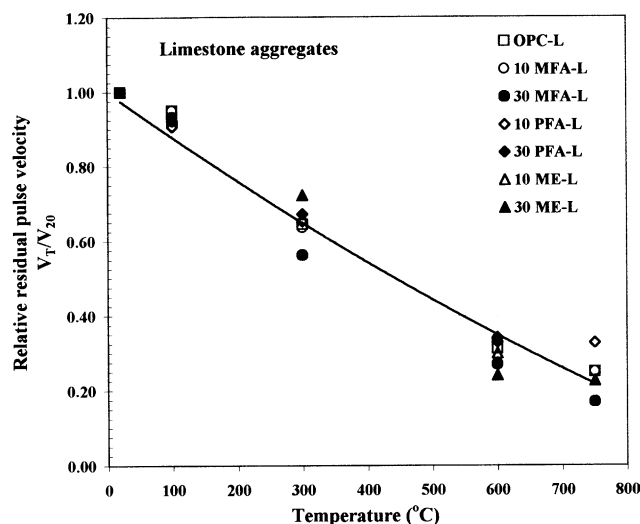

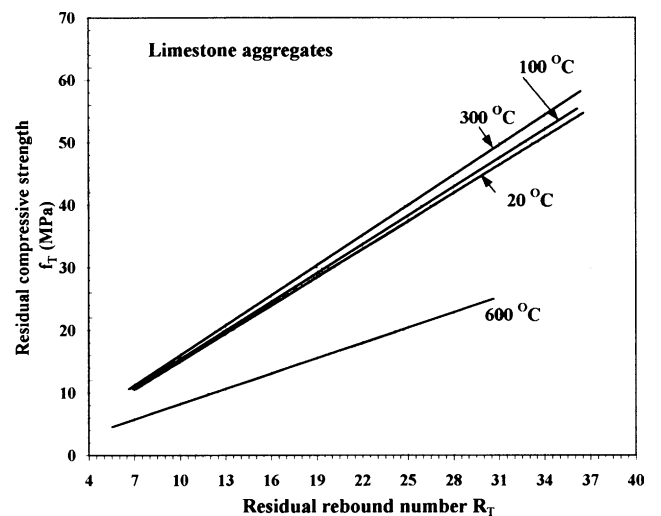Fig. 13. Relative residual pulse velocity of limestone concretes.

Fig. 15. Relation between rebound number and residual compressive strength after heating at various temperatures. Limestone concretes.
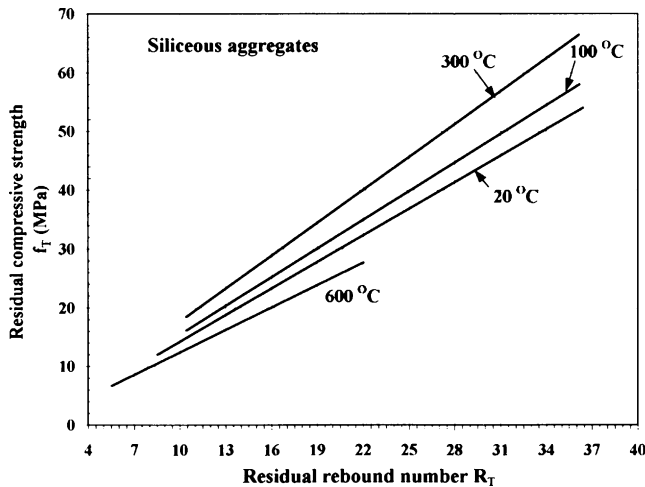
Fig. 16. Relation between rebound number and residual compressive strength after heating at various temperatures. Siliceous concretes.
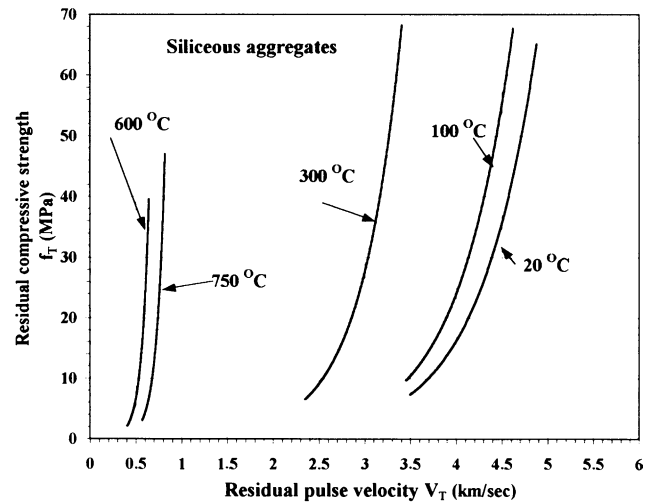

Fig. 18. Relation between pulse velocity and residual compressive strength after heating at various temperatures. Siliceous concretes.
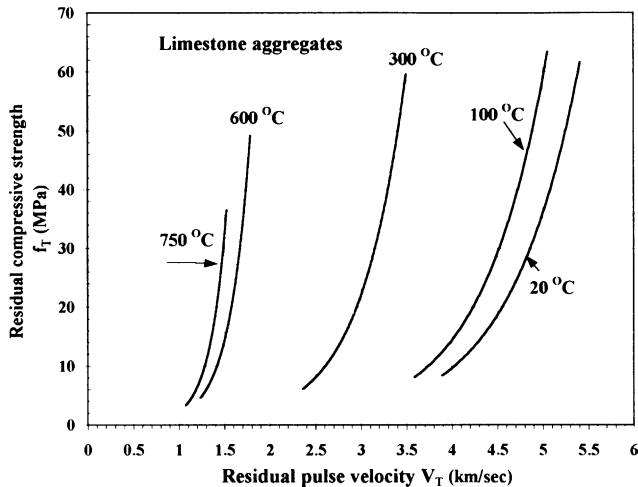

Fig. 17. Relation between pulse velocity and residual compressive strength after heating at various temperatures. Limestone concretes.

4. Conclusions

Strength, modulus of elasticity, rebound and pulse velocity are affected differently during heating.

Between 100 and 750 °C, concretes made with 10 MFA have the same behavior and the same changes of the initial strength with pure OPC, whatever the aggregate type is.

Concretes with pozzolanic materials show better strength results than the pure OPC concretes, up to 300 °C, while they seem to be more sensitive when exposed to heating above 300 °C.

Up to 300 °C, only a small part of the initial strength is changed. The type of binder and aggregate does not affect the strength change significantly in this temperature range. Between 100 and 300 °C, the initial strength

of almost all the mixtures increases. This increase is higher for siliceous concretes.

At temperature above 300 °C, a decrease in strength is observed, which is higher for the PFA (siliceous aggregates) concretes and for ME (limestone aggregates). At 600 °C, the strength of mixtures is reduced to about the half; at 750 °C, the reduction is from 75% to 93%.

Temperatures between 300 and 750 may be regarded as critical to the strength loss of concrete. At these temperatures, the greater the percentage of the replacement of OPC becomes, the greater the reduction in initial strength tends to be.

A continuous drop in the modulus of elasticity is noticed at all the temperatures. This drop is higher for the limestone concretes.

Based on the results of specimen tested at different temperatures, calibration curves have been proposed for the estimation of concrete strength after heating, by means of non-destructive measurements.

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