Residual mechanical properties of heated concrete incorporating different pozzolanic materials

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This paper aims at showing the effect of high temperatures on mechanical properties of concretes in which Portland cement (PC) has been partially replaced by pozzolanic materials. Three types of pozzolanic material, one natural pozzolana and two lignite fly ashes (one of low and one of high lime content) were used for the replacement. Concrete specimens were tested at four temperature levels of 200, 400, 600 and $800^{\circ}C$ without any imposed load. The specimens for each of the chosen temperatures were heated under the same heating regime, so a comparison of their behaviour during heat exposure concerning their mechanical properties was possible. Tests of compressive strength, splitting tensile strength and modulus of elasticity were carried out on specimens cooled slowly to room temperature 1 day, 7 days, 1 month and 3 months after heating. Based on the results of this experimental work it can be said that concretes with pozzolanic materials added to the mixer in partial replacement of PC are more sensitive to exposure to fire than conventional concretes. A relatively greater drop in the strength of the concretes with pozzolanic materials was found especially in the temperature area of 200°C when they were compared with conventional concrete made from PC of Greek type (10% insoluble residue). This particular sensitivity was attributed to heat deformations of hardened pastes, as all the other testing parameters concerning consistency and rate of heating remained the same for all specimens. It is suggested that this is caused by the escape of the non-evaporable water that exists as combined water in hardened pastes of pozzolanic binding agents. These pastes are richer in compounds such as calcium aluminates, calcium aluminate sulfates and tobermorite gel (C-S-H) which are decomposed between 110 and 150°C.

1. INTRODUCTION

Concretes containing pozzolanic materials are known worldwide for their good performance in structural applications. In many countries pozzolanic materials have been used to a great extent for ecological and economic reasons. In Greece there is an increased interest in taking advantage of the two types of lignite fly ash produced by the Greek Public Power Corporation.

A literature survey on the effect of high temperatures on concretes with pozzolanic materials showed that there is a limited amount of data dealing with blended types of cement [1–3]. The purpose of this work was to provide experimental data with regard to the residual mechanical properties of heated concretes containing pozzolanic materials added in the mixer, usually in high percentages. These properties are obviously very important in the repair of concrete structures such as buildings subjected to fire, aircraft runways, chimneys or shielding in nuclear reactors.

2. EXPERIMENTAL DETAILS

2.1 Materials

In the tested concrete mixes, Portland cement (PC) was partially replaced by pozzolanic materials. Three types

have been used:

(i) Santorin Earth (SE), a natural pozzolana from Santorin Island deposits;

(ii) lignite fly ash from the area of Ptolemaida (LFAP), ground and unground; and

(iii) lignite fly ash from Megalopoli (LFAM).

Control mixes made with PC of Greek type (10% insoluble residue), which is the best known cement for conventional concrete structures, were also prepared.

The chemical analyses and physical properties of these materials are given in Table 1. Crushed limestone aggregate was used for the coarse fraction (max. size 31.5 mm) and the fine fraction. Their grading curves are shown in Fig. 1.

2.2 Concrete mix proportions

Six series of concrete mixes were prepared with the following constant mix proportions:

1. Water/(cement + pozzolanic material) = 0.65. This ratio was selected by taking into account the strength range of normal concrete structures, of 20.0-30.0 MPa.

2. Cement + pozzolanic material content: 300 kg m^{-3} .

3. Aggregate/(cement + pozzolanic material) = 6.2 (by weight). However, the following differences should be

Constituent	PC (Greek type)	LFAP	LFAM	SE
SiO,	23.20	42.80	55.00	61.80
Al ₂ O ₃	6.87	10.30	24.50	15.60
Fe ₂ O ₃	3.58	3.45	2.50	5.90
CaO	61.70	Total 36.70 Free 10.80	9.50	4.00
MgO	3.80	3.40	2.90	2.10
SO ₃	2.51	6.50	2.0	0.70
Alkalis	2.21	1.20		
Loss on ignition	2.10	4.30	2.80	3.10
Insoluble residue	10.0	21.50	35.50	85.10
Specific gravity (kg m ⁻³)	3.10	2.42	1.97	2.45
Blaine fineness $(kg m^{-3})$	250-300	Without grinding 290–350 Ground 650–850	145	180-230

Table 1	Chemical	composition and	l some physical	characteristics o	of Greek	Portland cement,	LFAM and S
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noted: (i) in the first control mix, Greek-type cement was used, and (ii) in the subsequent mixes, normal PC was replaced in the mixes by (a) 30% LFAP, (b) 30% ground LFAP, (c) 40% ground LFAP, (d) 30% LFAM and (e) 30% SE.

The percentages of pozzolanic material used were selected in compliance with their properties and the optimum cost-saving for concrete production [4]. LFAP has pozzolanic and hydraulic properties. Concrete mixes incorporating neat ground LFAP (to a fineness 650- $750 \,\mathrm{m^2 \, kg^{-1}}$) as a binder develop at 28 days at least 40% of the compressive strength of control mix [5]. Bearing this in mind, two percentages of ground LFAP, 30 and 40%, were also studied.

2.3 Casting and curing of test specimens

100 cylinders, $150 \text{ mm} \times 300 \text{ mm}$, were cast for each mix series. All cylinders were cast, capped and cured according to ASTM C192-81 recommendations. Although large specimens usually favour the development of high stresses across their section, cylinders of $150 \text{ mm} \times 300 \text{ mm}$ were chosen since they were considered to be more representative of the quality of conventional concrete.

After 28 days of moist curing, the specimens were transferred to the room climatic conditions until required for testing.

Fine Chromel-Alumel wire thermocouples, covered with a special ceramic tube, were incorporated in ten specimens along their axis.

2.4 Heating and testing

An electric furnace was used. The furnace temperature was controlled by an electronic controller. Three specimens were heated each time in the furnace. The temperatures were continuously recorded by two similar thermocouples: one positioned at the mid-height centre of



Fig. 1 Grading curves of aggregates used.



Fig. 2 Temperature-time curves used for the heating of concrete specimens: (--) furnace, (---) centre of specimen.

the specimen and the other at the centre of the furnace. Such temperature-time curves are shown in Fig. 2. Preliminary experimental work was carried out to decide upon the temperature range and duration of specimen heating. The following test procedure was followed:

1. Four temperature levels were examined: 200, 400, 600 and 800°C.

2. The rate of heating was increased step-by-step as shown in Fig. 2. Three cylinders of each of the six mixes were heated for each temperature level.

3. The heating period was 3 h. The maximum final difference allowed between the centre and the outside of the specimens was 50° C for 200 and 400° C temperature levels, and 60° C for 600° C, and 70° C for 800° C levels.

4. None of the specimens at the time of heating was allowed to have a moisture content of more than 3%. Otherwise, there was a risk of explosive spalling. The specimens with higher moisture were dried before testing.

The specimens were tested at six months after casting.

A total number of 648 specimens were heated. 21 specimens of each of six mixes and for each of the four temperature levels were tested after having been left to cool slowly to room conditions. Groups of three of them were crushed at 1 day, 7 days, 1 month and 3 months after heating. Groups of three specimens were also used to determine the one-day residual splitting tensile strength

and modulus of elasticity according to ASTM C496-71 and C469-81, respectively.

At the same time, nine unheated specimens selected at random were tested in compression and the mean compressive strength was taken as the normal strength of the mix.

2.5 Properties of fresh concrete

All concrete mix slumps ranged between 80 and 120 mm. For concrete mixes incorporating 30 and 40% ground LFAP a loss in slump was expected owing to its fineness. The slump of these mixes ranged from 50 to 80 mm.

3. TEST RESULTS AND DISCUSSION

The residual compressive strength $f_{c,\theta}$ after heating at different temperatures was expressed as a ratio $f_{c,\theta}/f_{c,20}$ where $f_{c,20}$ is the strength of concrete of the same age without heating. In Figs 3 to 6 the changes in residual strength 1 day, 7 days, 1 month and 3 months after heating are plotted so that concretes with pozzolanic materials can be compared with concrete with PC of Greek type which is largely used in Greece.

From the results it is obvious that a decrease in the compressive strength of all tested concretes started at 200°C and progressed until a complete deterioration of

1.0 8.0 50 10 10 Compressive strength 0.6 Â 0.4 0.2 (150 x 300) mm 8 800 200 400 600 0 temperature 🦻 [°C]

Fig. 3 Residual compressive strength of concretes 1 day after heating: (×) PC (Greek type), (\bigcirc) 30% LFAP, (\square) 30% GLFAP, (\triangle) 40% GLFAP, (\odot) 30% LFAM, (\bigtriangledown) 30% SE.

concrete occurred at 800° C. Concrete with PC had a reduction in strength of the order of 25%, while concretes with pozzolanic materials showed a reduction which ranged from 38%, to 50% at 200°C. This loss of strength at around 200°C (which corresponds to 145°C in the centre of the specimens after 3 h heating) is greater than that referred to by Abrams [6] and Malhotra [7]. At around 400°C (which corresponds to 350°C in the centre of the specimens) the decrease in strength rose to 50% for



Fig. 4 Residual compressive strength of concretes 7 days after heating. (\times) PC (Greek type), (\odot) 30% LFAP, (\Box) 30% GLFAP, (Δ) 40% GLFAP, (\odot) 30% LFAM, (∇) 30% SE.



Fig. 5 Residual compressive strength of concretes 1 month after heating. (\times) PC (Greek type), (\odot) 30% LFAP, (\Box) 30% GLFAP, (Δ) 40% GLFAP, (\odot) 30% LFAM, (\bigtriangledown) 30% SE.

concrete with PC of Greek type and to 65% for concretes with pozzolanic materials.

At temperatures over 400° C all tested concretes suffered deterioration and they lost 70 to 80% of their initial strength. Considering Figs 3 to 6 it can be seen that there is in all cases (especially at 200°C) an upper limit line formed by concrete with PC, which indicates that concretes with pozzolanic materials are more sensitive to high temperatures.



Fig. 6 Residual compressive strength of concretes 3 months after heating. (×) PC (Greek type), (\odot) 30% LFAP, (\Box) 30% GLFAP, (Δ) 40% GLFAP, (\odot) 30% LFAM, (∇) 30% SE.



Fig. 7 Strength recovery of concretes heated at 200 and 400°C. (×) PC (Greek type), (\bullet) 30% LFAP, (\Box) 30% GLFAP, (\diamond) 40% GLFAP, (\odot) 30% LFAM, (∇) 30% SE.

At a temperature of 145° C (which corresponds to a furnace temperature of 200°C) the reduction of concrete strength is due to escape of non-evaporable water, since evaporable water is supposed to be removed after prolonged drying of specimens at 110° C [8].

Among the tested concretes the only difference in consistency was in the cementitious material. In some of them fly ashes and Santorine earth were used to replace PC. Consequently, differences in their behaviour on heating were thought to have come from their binding agent, as the heating regime remained the same for the specimens at each temperature level. At the age of testing (six months after casting) an additional tobermorite gel (C-S-H phases) was formed [9] due to pozzolanic reaction of the Ca $(OH)_2$ in PC with reactive silica in the pozzolanic material.

Regarding the literature [10, 11], the following changes



Fig. 8 Residual splitting tensile strength of concretes with pozzolanic materials 1 day after heating. (×) PC (Greek type), (\bigcirc) 30% LFAP, (\square) 30% GLFAP, (\triangle) 40% GLFAP, (\bigcirc) 30% LFAM, (\bigtriangledown) 30% SE.

in cement paste are referred to in DTA-TG studies:

(i) At 110 to 150°C, calcium aluminate sulphate hydrates dehydrate [10].

(ii) Over 105°C, tetracalcium aluminate hydrate loses part of its combined water [11].

(iii) At 120°C, amorphous tobermorite gel dehydrates [8].

It is generally known that paste mixtures of PCpozzolana or PC-fly ash are richer in calcium aluminates or calcium aluminate sulphate as well as in tobermorite amorphous gel [9, 11, 12]. Furthermore, DTA-TG diagrams referring to these pastes [13] indicate that at temperatures from 55 to 160°C a greater loss of mass occurs than that for paste made with PC of Greek type. It must also be said that calcium sulphoaluminate hydrates



Fig. 9 Residual modulus of elasticity of concretes with pozzolanic materials 1 day after heating. (\times) PC (Greek type), (\bigcirc) 30% LFAP, (\square) 30% GLFAP, (\triangle) 40% GLFAP, (\bigcirc) 30% LFAM, (\bigtriangledown) 30% SE.





(a)

Fig. 10 Hair cracks on specimens heated at (a) 400°C, (b) 600°C, (c) 800°C.

(such as ettringite) were found in paste mixtures of PC and fly ash of Ptolemaida, some hours after their mixing [14].

The above may give an answer to the question of sensitivity of mortars with pozzolanic materials when heated at temperatures of between 110 and 200°C.

In Fig. 7 a further loss in strength was observed for all tested concretes 7 days after heating. This was in agreement with that of other researchers [3]. It is also obvious that there is a slight recovery in strength for concretes heated at 200 and 400°C, while for concretes with pozzolanic materials this recovery is less. This means that the rehydration process in these concretes is slower.

From Fig. 8 it can be said that there are differences in the splitting tensile strength of tested concrete at a temperature of 200°C alone. At this temperature, concrete with PC of Greek type lost about 15% of its initial strength, while the corresponding loss in strength of concretes with pozzolanic materials ranged from 20 to 40%. In Fig. 9 it can be seen that the test results for modulus of elasticity follow a somewhat similar pattern to those of splitting tensile strength. At a temperature of 200° C the modulus of elasticity of concrete with PC of Greek type decreased to about 30% of its initial value, while the corresponding decrease for concretes with pozzolanic materials ranged from 21 to 41%.

Differences among concretes with various pozzolanic materials were not very clear, although in most cases concretes with LFAP were nearer to the upper limit line of concrete with PC of Greek type (see Figs 3 to 9).

4. INSPECTION OF SPECIMENS

A network of hair-cracks was observed at 400° C which became deeper at 600° C, as shown in Fig. 10. At 800° C, spalling occurred in specimens during cooling which resulted in the concrete softening.

Through careful observation it was possible to distinguish at 400°C a specimen's colour change which increased in intensity at high temperatures.

As shown in Fig. 11, cracks in the section of concrete



Fig. 11 Cracks in sections of heated specimens. Stereoscope photographs of the section of a specimen heated at temperatures of (a) 200°C, (b) 400°C, (c) 600°C, (d) 800°C.

specimens started to propagate at 400°C and increased considerably at 600 and 800°C. These formed mainly around aggregate particles.

5. CONCLUSIONS

Concretes with pozzolanic materials added in the mixer in order to replace PC seem to be more sensitive to exposure to heating. They lose a great percentage of their mechanical strength compared to concretes with PC of Greek type. Especially at the temperature of 200°C the difference in the reduction of strength is obvious, and increases to a factor of two.

This is attributed to the thermal behaviour of PC-pozzolana pastes. It is suggested that these pastes contain larger quantities of compounds or crystal phases which are decomposed at 200°C.

All tested concretes show a slight strength recovery three months after testing, but the rate of rehydration is slower for concretes with pozzolanic materials.

There are no clear differences in the thermal behaviour of concretes in relation to the type of pozzolana, although the results for concretes with LFAP are closer to those of concrete with PC of Greek type.

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RESUME

Propriétés mécaniques résiduelles de béton chauffé renfermant divers matériaux pouzzolaniques

Cette étude fait partie d'une recherche sur l'effet de la température sur les propriétés mécaniques de béton dans lequel le ciment Portland a été remplacé au cours du mixage par des matériaux pouzzolaniques. Trois types de ces matériaux, soit deux cendres volantes et une pouzzolane naturelle, ont été utilisés afin de remplacer partiellement le ciment Portland dans le béton. Les éprouvettes de béton non modifié ont été soumises à différentes températures: 200, 400, 600 et 800°C. La résistance en compression, en traction, et le module d'élasticité ont été mesurés à un jour, sept jours, un mois et trois mois après l'essai thermique.

Les bétons renfermant des matériaux pouzzolaniques ont manifesté une forte diminution de la résistance et de l'élasticité. On peut dire que le remplacement du ciment Portland par des matériaux pouzzolaniques ne contribue pas à l'augmentation de la résistance du béton au feu, tout au moins dans le cas de températures peu élevées.