Use of decomposition of portlandite in concrete fire as indicator of temperature progression into the material

Application to fire-affected builds

Esperanza Menéndez · Luis Vega · Carmen Andrade

CEEC-TAC1 Conference Special Issue © Akadémiai Kiadó, Budapest, Hungary 2012

Abstract In field structures affected by fire, the temperature progress through the material. The progression of temperature in the concrete material can be determined by simultaneous differential thermal analysis and thermogravimetry. Also, the analysis of the behaviour of concrete in real concrete, by different techniques, permits the corroboration of the hypothesis of cover calculation. In this study, the analysis of concrete exposed to a very severe fire is studied in order to corroborate the calculus hypothesis and to determine the progression of the temperature inside the affected structure. In this study, the potentiality of the thermal instrumental techniques is studied to determine the situation of the concrete exposed to fire. These results can be used to calculate the residual strength of the concrete structural elements. Also, other auxiliary techniques are used to have some supplementary information about the situation of the concrete exposed to fire. The results are based in concrete samples from a real fire in the Windsor Building in Madrid. The Windsor Building in Madrid was project in 1974 and built between 1975 and 1979. This building was severely damaged by a serious fire on the 12th of February 2005, which lasted approximately 12 h.

E. Menéndez (⊠) Institute Eduardo Torroja of Construction Science (IETcc-CSIC), Madird, Spain e-mail: emm@ietcc.csic.es

L. Vega Ministry of Development of Spain, Madird, Spain

C. Andrade

Centre of Investigation in Security and Durability of Structures and Materials (CISDEM–CSIC–UPM), Madird, Spain

Keywords Concrete · Fire · Portlandite decomposition Temperature progression

Introduction

The concrete has different types of transformation when has been exposed to fire with increase of temperature from the exposed surface. Some of its transformations are the loss of water and the decomposition of hydrated products of the bulk, like C–S–H gel or portlandite. Other transformations are related with the carbonates that can be formed due to the natural carbonation or by recarbonation processes associated with the CO_2 or CO formation during the fire.

The decomposition of portlandite is produced between 450 and 550 °C, and the decomposition of calcium carbonate is produced between 650 and 800 °C. Both of these phenomenons can be analysed by simultaneous differential thermal and thermogravimetric analyses (DTA-TG). On the other hand, the fire engineering uses some simplified methods to evaluate the residual service life of the concrete structural elements after fire. Also these methods are useful to dimension the rebar covers in the reinforced concrete. One of the most used simplified methods is denominated method of the isothermal 500. In this method, the residual strength of concrete is calculated only with the material not exposed at higher temperature than 500 °C. Also, some calculations to determine the rebar cover are estimated based on the theoretical behaviour of the concrete and the steel at different isothermal temperatures.

When the concrete becomes subjected to higher temperatures, it goes through a series of physical-thermal transformations that bring about a modification in the products of the hydration of the cement. The decomposition of the C–S–H gel starts at 120 °C, with a loss of water which brings about a contraction as a result of this loss, but it is necessary to exceed 900 °C to complete the decomposition of this CSH. Likewise, a decomposition of the portlandite comes between 450 and 550 °C and the loss of CO₂ of the carbonates usually goes from 600 to 800 °C. Also, the possible changes in the aggregate as an effect of the temperature, it depends on other factors such as size, porosity, permeability, etc. In general, we can consider that the less porous the aggregate, the less susceptible it is to the action of the fire. In general, the concrete in ambient temperature constitutes a suitable protection for the reinforcement due to a low thermal conductivity and an elevated specific heat, which is why [1–7].

Different instrumental techniques are used with the aim of analysing the behaviour of the concrete exposed to a real fire or laboratory tests. Specially, thermal analysis, scanning electron microscopy and X-ray diffraction are useful to analyse the concrete exposed to fire. Some of these instrumental techniques, such as thermal analysis, are usually used to study the behaviour of certain materials when exposed to the action of fire and high temperatures. As a rule, the fundamental objective of these analyses is to look at the possible improvements that could come about through the use of certain materials that could substitute or complement others [8].

The physical-thermal transformations undergone by the concrete as an effect of the temperature are translated into a loss of performance, especially when the material exceeds 600 °C. As the temperature increases, a modification in the creep comes about, which can also be seen as a strong dependency on its resistance and elasticity [9]. On the other hand, the conditions of putting out the fire must be taken into account, as the speed of cooling or the contact with the water has a significant influence on the physical-mechanical conditions present in the concrete once the fire is extinguished [10]. Specifically, the physical-thermal transformations that the components of the concrete go through serve to characterise it after it has been subjected to the fire [3, 11, 12]. Other cement-based materials like the MDF materials are also studied by these techniques in order to analyse their composition and the behaviour of these under in different conditions of moisture [13, 14]. Also, the effect of addictions to the cements is studied by thermal techniques, like the study of mixes of burnt clay and silica fume or the effect of the superplasticizers on matrixes with fly ash and silica fume [15, 16].

During the project, step of the buildings are introduced the estimating calculus with relation to the fire resistance of the structural elements of the reinforced concrete. With regards to the high buildings in Spain during 1970s decade, the concrete fire resistance about 120–180 min was established. Usually, in the calculation are used the isotherm 500 °C method as the calculation hypothesis. To estimate the potential depth of concrete affected by fire usually the parametric fire curves are used. In these curves, the depth of concrete affected are established for different time of exposition in function of the temperature [17, 18]. Due to this, it is important to determine the depth of the concrete in which the temperature has been higher than 500 °C.

The analysis highlights the need for a residual bearing capacity in a structure affected by fire with a view to determine the progression of the temperatures in the interior of the elements realistically, for which certain instrumental techniques can be used. These techniques allow real information to be obtained on concrete samples that have been exposed to high temperatures. Obviously, the distribution of the temperatures in the interior of the area affected by the fire is not homogeneous, which is why the determination of the number of testing points, their distribution, etc., so that the results are representative and constitute a sound basis for the structural analysis is a relevant question. However, when samples taken from a real fire are analysed, it is not always possible to have the suitable number or disposition samples available. Nevertheless, the analysis of the materials that have been subject to a real fire, by means of certain instrumental techniques, can bring about a significant amount of knowledge on their behaviour [19].

The concrete samples come from the Windsor build, burned in 2005. The concrete was manufactured with an OCP. The building was constructed around a central reinforced concrete core with a facade consisting mainly of glass and steel. The building was 37 storeys high with a technical floor on the 17th floor. The fire broke out on the 21st floor. The fire caused most damage to the floors above and brought about the collapse of part of the upper external facade over the technical floor upwards.

Experimental

Concrete samples

All the samples analysed come from the concrete of the Windsor Building. The concrete cores have been taken from the concrete elements after the fire. Sections of the construction elements of the reinforced concrete were made available during the demolition process, consisting mainly of pillars taken from the 12th, 13th, 14th, 15th, 18th and 19th floors. Samples were taken of the different structural elements in order to study the concrete and analyse it using different instrumental techniques.

In each concrete core, different sections were taken at different depths, until 8 cm in depth. Also, a reference sample of concrete is taken in each case, 15 cm inside from

 Table 1
 Characteristics of the concrete samples and the structural elements which have taken the cores

Concrete samples		Structural elements		
Denomination	Depth from the fire exposed surface/cm	Floor	Construction element	Name
H-1	0–2	12	Pillar (2 pillars)	P12A and P12B
H-2	2–3	13	Pillar	P13
H-3	3–4	14	Pillar forged	P14 and F14
H-4	4–5	15	Pillar	P15
H-5	5–6	18	Pillar	P18
H-15	15–16	19	Pillar	P19

the exposed surface to the fire. Other contrast or reference areas or unaffected depths have also been analysed from the concrete. In Table 1, one can see the characteristics of the samples of the concrete analysed, indicating the depth from the surface exposed to the fire. Also, Table 1 shows the floor and the type of construction elements which have taken the concrete elements to extract the concrete cores.

It is considered that the concrete from the inside samples, situated at a depth about 15 cm have been affected by the fire. And, these results are used as a reference of the original concrete. While, the concrete samples taken from the surface exposed to fire provides the state of post-fire concrete. Being able to qualify the degree of alteration allows us to associate these segments to the 500 $^{\circ}$ C isotherm situation.

The samples were taken from the concrete cores at different depths in 1.5-cm thick sections. These sections were cut in dry in order to avoid the rehydration of the concrete or other interference with the material. Also, must be taken into account the potential rehydration of the cement paste during the extinction process due to the direct contact with the water and the potential carbonation of the concrete due to the CO or CO₂ formed as a consequence of the fire. In the case of partial rehydration of concrete from the extinction water, the concrete must show some portlandite close to the surface, the disappearance of it at certain depth and the normal quantity of portlandite inside of the material. In the analysed samples, we do not found portlandite in the concrete exposed to fire, while we can consider that this concrete has not been in direct contact with the water during the extinction or has not be rehydrated. With regard to the carbonation, this concrete was inside of the building and has not been carbonated.

Methods

The different concrete sample are analysed using the following instrumental techniques: Simultaneous DTA-TG, using Netzsch STA 409 equipment. The conditions of test are: 50 mg of sample, speed of heating 10 K/min until 1,050 °C, platinum crucible and 100 ccm of N₂ to avoid carbonation during the test. The principal results taken from these test are based on the evaluation of the following processes: dehydroxylation of portlandita between 450 and 550 °C approximately, decarbonation of calcium carbonates between 700 and 850 °C approximately and total loss of mass. The total amount of portlandite and calcium carbonates can be determined using the molar fraction and the loss of OH⁻ and CO₂, respectively.

Also, the microstructural aspect of the concrete has been made using backscattering electron microscopy combined with microanalysis of dispersive energy X-rays, with an equipment Jeol 5400 and a Link system from Oxford. Some samples have been analysed from the areas close to the exposed surface and the interior of the concrete. Finally, samples from the surface and interior of the concrete cores have been analysed using X-ray diffraction with a Phillips Mod. 1710 equipment. In this case, the majority crystalline compounds are found.

Results and discussion

Microstructural analysis of the damaged concrete phases by BSE–EDX techniques

Samples of concrete were analysed, taken at different depths, by means of backscattering electron microscopy combined with microanalysis using dispersive energy X-rays (BSE–EDX). According with the microstructural analysis, the concrete are made with an ordinary Portland cement without any type of additions and with siliceous and feldspars aggregates, typically used in the centre of Spain.

At different depths, we can observe some microstructural damage, especially at level of the cement paste, which is the most sensible component of the concrete to the fire action. The physical-chemical alterations of the concrete in the areas exposed to the fire are the analysis using these techniques. Close to the fire exposed surface, it is possible to see numerous microfissures, some of them parallel to the surface. Also, a loss of adherence with the cement paste in some of the aggregate can be observed. However, no significant alteration was observed in the aggregate which was of a quartz or feldspar type. In Fig. 1, it is possible to observe the microfissures and loss of adherence in the cement-aggregate interfaces. Also, the cement paste close to the exposed surface has a partial loss of cohesion in the C-S-H gel that is observed as well as the microfissures in anhydrous particles of clinker. Although in the concrete of all samples in depths higher than 5 cm, the unaltered aspect is observed. In Figs. 2 and 3, we can observe the aspect of the cement paste close to the exposed surface, with



Fig. 1 Microfissuring in concrete, parallel to the exposed surface to fire

alteration of the C–S–H gel and increase of porosity, and the cement paste in the interior with a normal aspect and aggregates without alteration inside or the paste–aggregate interface.

The damage at microstructural level is relatively moderate in comparison with the virulence of the fire. The microfissures parallel to the surface are limited to approximately 1 cm in depth. With respect to the cement paste, it is possible to observe some degree of alterations until higher depths. We can see some loss of mass and increase of porosity in C–S–H gel until approximately 5 cm in some samples.

Evaluation of the concrete damaged depth due to fire

For each concrete core, homogeneous samples were analysed, taken from the different depths, indicated in Table 1.

The samples were crushed and analysed by DTA–TG. Also, reference samples were studied to check that the concrete were not carbonated in the non-affected areas by the fire. In the analysis by DTA–TG, the presence of portlandite was the main analysis, this is observed by the loss of mass that brought about between 450 and 550 °C approximately, which is associated to the decomposition by the dehydration of the portlandite. The presence of portlandite and the quantity of it can be determined at the different depths of the concrete from the surface exposed to fire. The complete disappearance of the portlandite indicates that the concrete has exposed to temperatures higher than 500 °C during a certain period of time. This characteristic permits to know the depth at which the isotherm 500 °C is.

When analysed the different samples that come from the different concrete cores, we can classify these concrete according the depth at which the portlandite have disappeared. In this case, we can distinguish two groups. One group in which the portlandite has disappeared at a depth of 3 cm was observed, and another group in which it has disappeared at a depth of 5 cm. In both cases, the curves of DTA-TG have shown the disappearance of the portlandite close to the surface and portlandite that remain from certain depth to the interior. Figure 4 shows the DTA-TG curves of the concrete at the different depths for one of the core samples affected until 5 cm. Also, the quantity of portlandite that remain in the cement paste can be possible in relation with the total one, which we consider is the portlandite that are inside the concrete without damage. In the Fig. 5, the medium sequences of portlandite decomposition in the two groups of concrete defined are shown, quantified by the DTA-TG results.

Also, an analysis of surface and inside samples was made using X-ray diffraction in order to corroborate the presence or absence of crystalline portlandite. Samples of



Fig. 2 Aspect of the cement paste close to the exposed side to the concrete with some alteration and associated elemental composition



Fig. 3 Aspect of the cement paste inside the concrete core with a normal aspect and associated elemental composition



Fig. 4 TG and DTA curves of concrete with degradation of portlandite at a depth of 5 cm

concrete were analysed, from each of the samples extracted. In all of the cases, a total absence of portlandite in the surface samples was observed, which is associated with the concrete's exposure to high temperatures. While in the inside samples at depth higher than 15 cm, the presence of portlandite was observed, which would correspond to a concrete unaltered by the effects of the high temperature. It also identified the presence of quartz and feldspar, which correspond mainly to the aggregate present in the concrete. Figure 6 details the spectra from the X-ray diffraction corresponding to both the surface and inside areas in relation to the surface of the concrete exposed to the fire.

It can be seen from the analysis that the concrete exposed to the fire has undergone a significant alteration on its surface area which translates to a loss of properties in the cement paste, such as the decomposition of the portlandite and the partial dehydration of the C–S–H gel. The formation of microfissures mainly parallel to the surface



Fig. 5 Sequence decomposition of portlandite in concrete affected until 3 and 5 cm depth

exposed to the fire can be observed. Analysing the concrete of the different cores in intervals of depth, the concrete samples have been exposed to temperatures higher than $500 \,^{\circ}\text{C}$ (associated with the decomposition of the portlandite) to a depth of 3 or 5 cm. According with these results, we can define two families of samples P-12A, P-13, P-14 and P-19 affected until 3 cm and P-12B, F-14, P-15 and P-18 affected until 5 cm. And, at depths higher than 7.5 cm there are no evidences of physio-chemical or microstructural alterations and the concrete has not been affected by the temperature effect. In the Fig. 7, there is a scheme of the depth of concrete altered by temperatures higher than $500 \,^{\circ}\text{C}$ at the different floors studied.

The results of disappearance of the portlandite is well associated with exposure to high temperatures (approximately 1,200 °C), for a moderate period of time, or even a



Fig. 6 X-ray diffraction spectra for the surface (exposed to fire) and inside (15 cm depth) areas



Fig. 7 Scheme of depth of concrete damaged by fire according with the disappearance of the portlandite in concrete

prolonged exposure to more moderate temperatures (e.g. 750 °C), in accordance with the estimations carried out though the application of the parametric fire curves to the concrete, which confirms that the behaviour of a standard concrete is suitable in the case of it being subject to high temperatures, such as in a fire. The depths of affected concrete are according to a forecast for a fire resistance between 90 and 180 min, according to the parametric curves [14, 15]. According with these results, it is corroborated that the project exigencies were correct in order to ensure the required safety levels, using for these calculations the simplified method of the isotherm 500 °C.

Conclusions

• The effect of the fire in concrete exposed to real fire shows some physical-chemical alterations in the first few

centimetres of the concrete correspond to the appearance of microfissures and decomposition resulting from the dehydration of the portlandite and partially the C–S–H gel.

- Use of thermal analysis permits the establishment of the depths of concrete alterations when analysing sequential portions of concrete from the exposed surface.
- In this build, two depths of damage have been identified. These variable depths of the alteration of the concrete would indicate that the distribution of the fire in the building is not considered as homogeneous.
- The analysis of the concrete subjected to a real fire has allowed its behaviour to be contrasted to theoretical models based on the use of parametric fire curves. Also, according to the results obtained the simplified method of isotherm 500 °C and a demand of 120–180 min of parametric fire can be considered adequate to calculi the fire resistance of concrete structural elements.

Acknowledgements This study was supported by the CSIC Project PIE 200460E475 "Alteration of construction materials as a result of the development of reactions of an internal expansion. Identifications of the problem and its interactions. Monitoring and damage evolution".

References

- 1. Taylor HFW. Cement chemistry. 2nd ed. London: Thomas Telford; 1997.
- Mehta PK. Concrete. Structure, properties and materials. Englewood Cliffs: Prentice-Hall, Inc; 1986.
- Fletcher IA, Welch S, Torero JL, Carvel RO, Usmani A. Behavior of concrete structures in fire. Therm Sci. 2007;11–2:37–52.
- Menéndez E, De Frutos J. Equivalence between electrical measurements and X-ray diffraction in the formation of crystalline phases of cement paste. Bol Soc Esp Ceram Vidr. 2011;5:225–34.
- Charreau GL, Luna F. Efecto del fuego sobre los hormigones. Alteraciones sufridas por los agregados. Instituto Nacional de Tecnología Industrial. INTI-CECON. Jornadas de Desarrollo e Innovación. Octubre 2000.
- Hajpál M. Monuments exposed to fire or high temperature. Fire Technol. 2002;38:373–82.
- Lottman BBG. Fire in bored tunnels. Structural behavior, during fire conditions, of bored tunnels made with a concrete segmental lining. Delft: Delft University of Technology; 2007.
- Sarvaranta L, Elomaam M, Järvelä E. A study of spalling behaviour of PAN fibre-reinforced concrete by thermal analysis. Fire Mater. 1993;17:225–30.
- 9. Schneider U. Behaviour of concrete under thermal steady state and non-steady state conditions. Fire Mater. 1976;1:103–15.
- Nassil A. Postfire full stress-strain response of fire-damaged concrete. Fire Mater. 2006;30:323–32.
- Ercolani GD, Ortega NF, Señas L. Empleo de ultrasonidos y esclerometría en el diagnóstico de estructuras de hormigón afectadas por elevadas temperatura. Asociación Argentina de Ensayos no Destructivos y Estructurales. IV Conferencia Panamericana de END, Buenos Aires. Octubre 2007.
- Colombo M, Felicetti R, New NDT. Techniques for the assessment of fire-damaged concrete structures. Fire Saf J. 2007;42: 461–72.

- Drabik M, Galikova L, Zimermann P. Attack by moisture on advanced cement-based macroscopic defect-free materials. A thermoanalytical study. J Therm Anal Calorim. 1999;56:117–24.
- Mojumdar SC. Processing moisture resistance and thermal analysis of macro-defect-free materials. J Therm Anal Calorim. 2001;64:1133–46.
- Amin MS, Abo-El-Enein SA, Abdel Rahman A, Alfalous KA. Artificial pozzolanic cement pastes containing burnt clay with and without silica fume. J Therm Anal Calorim. 2011. doi: 10.1007/s10973-011-1676-5.
- Siler P, Kraiky J, De Belie N. Isothermal and solution calorimetry to assess the effect of superplasticizers and mineral admixtures on

cement hydration. J Ther Anal Calorim. 2011. doi: 10.1007/s10973-011-1479-8.

- UNE-EN-1991-Eurocódigo 1: Acciones en las estructuras. Parte 1–2: Acciones en estructuras expuestas al fuego. 1991.
- Documento Básico DB-SI: Seguridad en caso de incendio. Código Técnico de la Edificación. Ministerio de Vivienda– Gobierno de España 2005.
- Menéndez E, Vega L. Analysis of the behaviour of the structural concrete after the fire at the Windsor Building in Madrid. Fire Mater. 2010;34:95–107.