Experimental thermal characterization of concrete to be used in CP5.2 packaging system

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Abstract This work deals with the experimental evaluation of the thermal conductivity of a concrete matrix to be used for embedding LILW bituminised Wastes into the packaging system. Such a type of packaging, identified with the acronym CP5.2, has been also qualified by executing at the Lab. Scalbatraio of Dep. of Civil and Industrial Engineering of the University of Pisa, an experimental test campaign accordingly to the IAEA regulations.

In particular, the knowledge of the thermal conductivity is important because of the fire or furnace test to carry out in oven at 800 °C for 30 minutes. These data allowed to simulate pre-test conditions and to set up safety and operational fire test procedures.

The concrete thermal conductivity was obtained by performing hot wire tests on cylindrical concrete samples for temperatures ranging from 100° to about 800°C. Thermal conductivity is determined at steady state condition. Results indicate a monotonically reduction of the thermal conductivity as the temperature increases. The comparison with concrete thermal conductivity data available in literature indicates a quite good agreement. Finally, visual and X-ray inspection of sample did not highlight the presence of micro/macro damages that would have affected the thermal performance of the concrete under study.

1. Introduction

Concrete is widely used as a primary structural material both in conventional construction and in nuclear activities due to several advantages, such as strength, durability, fire resistance and non-combustibility properties. With reference to the fire exposure, that represents one of the most severe environmental conditions to which structures may be subjected, providing proper fire safety measures is an important outcome of design to fulfil.

Specifically, the present study deals with the thermal characterization of concrete to be used to immobilise bituminised LILW radioactive waste, stowed in drums, in turn stored in the inert concrete matrix of a CP5.2 packaging.

To this aim, the packaging integrity plays an important role, because a reliable structure must guarantee a safely storage in the repository during and beyond the institutional period. To be reliable, CP5.2 packaging must be qualified by submitting it to the tests foreseen by the IAEA SSR-6 rules [1], like fire test (engulffing fire condition foresees 800 °C for 30 minutes).

This means knowledge of deformations and of thermal and mechanical property changes [2] of the packaging constituting components, among which there is the concrete that, similarly to other materials, changes substantially at elevated temperature.

Although the availability of concrete and steel properties at elevated temperatures can permit a mathematical approach for pre-testing, to be confident that no abnormal situation will arise during the fire test execution, due to the peculiarity of bituminised wastes, the prediction of the thermal
conductivity of concrete becomes safety relevant (because of high burning risk) and mandatory. In fact, the bitumen may decompose forming flammable vapour, especially at temperature higher than about 300°C. On this basis, an experimental campaign aimed at the evaluation of the thermal conductivity of concrete was carried out at the Lab. Guerrini of Department of Civil and Industrial Engineering of the University of Pisa. The thermal performances of concrete samples were therefore evaluated for different temperatures up to 800°C.

In the following Section 2, the wire test methodology and the adopted test procedure are described, while the results obtained will be presented and discussed critically in Section 3, also by comparing them with the values of Kodur et al., Vodak et al. or Shin et al. [3, 4, 5, 6, 7, 8], which are available in literature.

2. Description of the experimental device
To thermally characterize the concrete, several cylindrical samples, grouted from the same cement paste, were prepared (Figure 1 a). They are 400 mm long and 100 mm in diameter (Figure 1 b).

Fig. 1 – Sample C3 soon after the grouting of the cement (a) and after 28-days curing (b).

Each sample is equipped with two thermocouples (positioned at a radial distance of 0.05 and 0.03 m from the centroid) and a central wire resistor. They were inserted in the mold before the grouting and held in the vertical position by means of a spacer device designed specifically to this purpose. In this way, the verticality/alignment of the sensors is controlled.

The two thermocouples (indicated in the following TC_INT and TC_EXT) are of type K and properly calibrated (calibration error less than 2%). They are immersed in the concrete at two different radial points, as shown in Fig. 2, in order to measure the temperature during the wire test-heating phase. In addition, attention was put to possible concrete de-hydration phenomena that would have degraded concrete properties, if material is exposed for long time at elevated temperature (higher than 500°C).

For this reason, an appropriate inspection of samples in post-test phase was carried out. After waiting 28 days for the maturation of the cement, thermal tests on samples were performed at the Lab. Scalbataro according to the following procedure:

a) opening and closure of the oven gate for the positioning of the sample (Fig. 3);

b) setting up of the oven temperature through PID controller;

c) starting of test by heating up uniformly the electric oven until the testing temperature is reached;

d) monitoring and control of temperature ramp by means of an adequate data acquisition system (DAS)

e) acquisition of temperature values by DAS when steady state condition is reached for at least 300 s.

Tests were performed between 100 and up to about 800 °C.
It is noteworthy that there are very few standardized methods available for measuring thermal properties of concrete. Therefore, to the aim of the study, the hot wire method is adopted; a more detailed description of which is given in [9].

Figure 4 exemplifies the acquisition data chain.

Each test is performed by heating up the sample homogeneously (thanks to the thermal resistances, distributed evenly along all walls of the oven, and the central resistor) in the oven until the steady state condition is reached. The temperature increase is adequately controlled through a PID system (GEFRAN© GF Promer) connected to the data acquisition system (DAS). The interface with PC is obtained via Labview©. GEFRAN system is a Microprocessor Controller that supports thermocouple configuration via the complete operator faceplate. It is equipped with a Lexan© membrane faceplate to provide a frontal IP65 rating.

The thermal conductivity is determined measuring the radial temperature gradient in correspondence of the cylinder cross section located at half of its length [10, 11], once steady state condition is reached. Indeed, the superficial temperature of sample, the oven temperature and the current intensity circulating in the wire are measured; this latter is used to calculate the power generated within the cylinder due to the Joule effect.
The thermal conductivity is so calculated by means of the Fourier law of heat conduction assuming a homogeneous thermal power production within the sample (because of the combined control of samples heating made by means of the imposed ramp of the resistor and oven electrical resistances), in plane geometry:

\[
k = \frac{\varnothing}{2\pi LR^2 \Delta T} (X_{\text{ext}}^2 - X_{\text{int}}^2) \quad (1)
\]

where:
- \( k \) [W/m °C] is the thermal conductivity;
- \( \varnothing \) [W] is the power generated by Joule effect within the wire;
- \( L \) [m] is the cylinder length;
- \( \Delta T \) [°C] is the difference of temperature between TC_INT and TC_EXT;
- \( R \) [m] is the radius of cylinder; and
- \( X_{\text{ext}} \) and \( X_{\text{int}} \) [m] are the radial distances of thermocouples from the cylinder axis.

Moreover, before the execution of wire tests, samples were X-ray inspected in order to exclude, as confirmed, any misalignment of thermocouples from their own generatrix or of the hot wire from its centreline. Figure 5 shows the steady state condition for wire test at 600 °C: for an average temperature of about 615 °C, an average power of 192.4 W, the thermal conductivity of sample is equal to 1.49 W/m °C.
3. Results and discussion

The average values of thermal conductivity at each testing temperature are listed in Table 1. The (total) maximum error associated to these measurements is lower than 8%. Furthermore, the discrepancy between thermal conductivity values, obtained from different samples and at the same testing temperature, is lesser than 5%.

The thermal conductivity of concrete at room temperature is in the range of 1.6 and 2.2 W/m °C and varies with temperature and depends on concrete composition [8]. It complies with the value of the upper curve of the Eurocode and with that proposed for carbonate and siliceous high-strength concrete (HSC) by Kodur and Sultan [4]. By analysing the behaviour shown in Fig. 6, it is possible to note that the thermal conductivity of the concrete monotonically decreases as the temperature increases. Best-fit curve, based on linear interpolation, see the Eq. (2) below, provides provisions of thermal conductivity mean values.

\[
k = 1.9364 - 0.0007 T \tag{2}
\]

This trend is mirrored in similar data available in the literature, e.g. those pointed out by Kodur [3], even if for normal strength concrete.

| Temperature [°C] | Average value [W/m °C] | Standard deviation |
|------------------|------------------------|--------------------|
| 100              | 1.86                   | 0.04               |
| 200              | 1.80                   | 0.07               |
| 300              | 1.73                   | 0.10               |
| 400              | 1.63                   | 0.12               |
| 500              | 1.56                   | 0.06               |
| 600              | 1.51                   | 0.06               |

Moreover the behaviour of the thermal conductivity indicates 20% reduction of the thermal conductivity in the temperature range between 100 °C and 600 °C. This reduction can be explained by considering de-hydration process occurring at elevated temperature and that leads to an increase of porosity and simultaneously to a reduction of the thermal conductivity: the same behaviour was found in ASCE and Eurocode data, as reported by Kodur in [3].

Fig. 6 – Behaviour of the thermal conductivity (the black solid line identifies interpolation curve).
On the basis of the measured data, a qualitative trend of thermal conductivity has been extrapolated up to 1000 °C (Fig. 7) for predictive purposes; it indicates that at this temperature we would have about 36 % reduction in thermal conductivity. This result seems in agreement with the reduction found by Vodak [5] and Kodur [6] in the range between 20 and 800 °C; nevertheless, the greatest deviation respect with the ASCE values appears at elevated temperature.

![Comparison between measured and ASCE and Eurocode thermal conductivity values](image)

**Fig. 7** – Comparison between measured and ASCE and Eurocode thermal conductivity values [3].

Finally, Fig. 8 shows an overview of the C3 sample surfaces before and after testing: they did not indicate significant damage on the cylinder surface, a part from an increased micro porosity, main responsible of the thermal conductivity reduction at high temperature.

![Overview of C3 cylinder surface before and after testing](image)

**Fig. 8** – Overview of C3 cylinder surface before (a) and after (b) the exposition at 800 °C for 30 minutes.

4. **Conclusion and future activities**

The concrete experimentally investigated by adopting the hot wire method allowed determining the thermal conductivity of this material that will be as matrix for bituminised LILW packaging.

The relevance of the activity relies with the fire test (800 °C for 30 min.) to execute to qualify such type of container. In fact, although concrete and steel properties are known at elevated temperatures and available in literature for conventional application, to be confident that no abnormal situation (high risk of burning of bitumen vapour) will arise during the fire test execution, concrete thermal
performances/characteristics must be evaluate. This is also because these latter are dependent on the concrete matrix and constituents.

The hot wire method test performed at the Department of Civil and Industrial Engineering of the University of Pisa allowed to determine the thermal conductivity of concrete in a wide range of temperatures, ranging from 100° to about 800 °C.

The thermal conductivity of concrete at room temperature is between 1.6 and 2.2 W/m °C and monotonically decreases as the temperature increases. Results highlight also about 32.5% reduction in the temperature range between 100 and 600 °C, probably due to de-hydration process. This consideration was supported by visual and X-ray inspection of sample.

Finally, the results have been compared with Eurocode, ASCE and Kodur data, available in literature, highlighting a good agreement (same trend also). The experimental thermal conductivity of concrete was used in turn for pre-test purposes in planning the fire test of CP5.2 LILW packaging.

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