Caratterizzazione di materiali edili, con il metodo integrato termografico-ultrasonico-analitico

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SOMMARIO
Lo studio è focalizzato sull'uso combinato della termografia IR e delle onde elastiche per la caratterizzazione non-distruttiva di materiali edili. Il modulo elastico viene ricavato da misure di velocità di impulso di onde elastiche (UPV).
Si presentano i risultati ottenuti in laboratorio integrando una specifica tecnica termografica con misure UPV sia dirette che indirette, che permettono di valutare le caratteristiche meccaniche e termiche di materiali porosi, comunemente impiegati.
In particolare, la diffusività termica di cemento cellulare prodotto in autoclave (AAC, AKG Gazbeton®) è stata misurata con una tecnica che opera su di una sola superficie. In questo modo, si ottiene un metodo che non richiede il prelievo del campione e potrebbe essere applicato in opera. Alcuni provini di differenti composizione, anche invecchiati artificialmente in acqua sono stati analizzati. Questi vengono caratterizzati misurandone la massa volumica, la porosità e capacità di assorbimento d’acqua.
La diffusività termica si ricava producendo un riscaldamento localizzato della superficie ed elaborando nel dominio delle frequenze una sequenza di immagini termografiche, che registrano l’evoluzione della temperatura superficiale.
Infine, vengono analizzati in modo integrato i dati ottenuti, per correlare le proprietà meccaniche con quelle termiche soprattutto in relazione al decadimento dei materiali.
I risultati di ulteriori test analitici sono presentati per validare i metodi proposti.

Abstract:
This study was focused on the use of Quantitative IR Thermography (QIRT) together with ultrasonic pulse velocity (UPV) for the determination of the thermal-mechanical properties of building materials as a non destructive testing (NDT) methods. The thermal diffusivity in Autoclaved Aerated Concrete (AAC, AKG Gazbeton®), which is a highly porous and non-homogeneous material, was evaluated using a single face procedure. An experimental set up based on a laser shot of the surface was monitored in space and time by IR Thermography and the data reduction in the frequency domain allowed achieving the thermal diffusivity. The state of deterioration of the water-aged AAC samples were also examined by NDT methods, such as using QIRT and UPV measurements. Fresh and water-aged AAC samples were examined in terms of physical and mechanical properties variations. Finally, other analytical methods, as XRD and optical polarised microscopy have been used to validate the proposed NDE methods.
Introduction

Autoclaved Aerated Concrete (AAC) is one of the light-weight construction materials for contemporary buildings, especially due to its low density, unique thermal and breathing properties [1,2]. Within wall frames, such porous materials are more and more frequently used, improving the thermal performance of buildings and thereby contributing to the energy efficient design. The study was, especially, focused on the use of Quantitative Infrared Thermography (QIRT) for the determination of the thermal properties of AAC produced in Turkey, AKG Gazbeton®. The study was also conducted to monitor the changes in mechanical and thermophysical properties for the aged AAC samples, by non-destructive methods. Some changes in the mineral composition and mechanical properties, such as surface carbonation on exposed surfaces and decrease in modulus of elasticity (Emod) were expected for the water-aged AAC samples [1,3,4]. For the assessment of such variations, both fresh and artificially aged AAC samples have been used. Ageing is given by saturation of samples and leaving in water for some days before further tests by means of thermal diffusivity and ultrasonic pulse velocity (UPV) measurements. These studies were also supported by further physical and mineralogical analyses. The correlation between the thermal diffusivity and UPV measurements was evaluated by taking into account the surface deterioration of water-aged samples.

Materials and methods

Two types of AAC used in the study are produced: one as an infill (G2) and the other one as load-bearing material (G4). For the analyses, three sets of samples for each type of AAC were prepared in different dimensions, such as the thinner ones in the form of rectangular prisms of 2cm x 3.5cm x 5cm, the thicker ones as cubic samples of 5cm x 5cm x 5cm and 5cm x 5cm x 20cm (see Fig.1).

Figure 1 - Samples prepared for the analyses: the first set with dimensions of 2-2.5cm (thickness) x 3.5cm x 5cm (at the bottom), the second set with dimensions of 5cm x 5cm x 5cm (at the left top) and the third set with dimensions of 5cm x 5cm x 20 cm for the indirect UPV measurements (at the right top)

Some samples were aged by leaving them in soaked condition for 6 days, 20 days and 30 days. The differences between the fresh and water-aged AAC samples were monitored in terms of basic physical and mechanical properties, such as bulk density, effective porosity, water absorption capacity, thermal diffusivity, UPV and modulus of elasticity (Emod). The thermal diffusivity values were determined by QIRT [5,6]. The Emod expresses the deformation ability of a material under external forces. Emod was determined indirectly.
by means of computation according to equations described in the standards: RILEM and ASTM D 2845-00, applied to UPV and density [7,8]. The relationship between the Emod and thermal diffusivity values was investigated for the fresh and water-aged AAC samples. Some supportive laboratory analyses were also done to examine the compositional differences between the surface and bulk of water-aged AAC ones. In fact, it is supposed that the water immersion affects the thermophysical and mechanical properties of the material at the surface. The existence of any deterioration product at the surface of the aged samples was investigated by optical polarised microscopy and XRD analyses. The procedure of each study is briefly explained below.

**Quantitative Infrared Thermography**

Thermal diffusivity of AAC was evaluated using a single face procedure [4]. On this purpose, the experimental set up is based on a laser NdYAG (1.064 µm wavelength) that shots the surface of the sample in a circular area of 1 cm diameter with a short pulse whose duration is less than 1 ms. An IR camera (FLIR SC3000) grabs a sequence of images at a frequency of 50 Hz. Heat propagates inside the material and spreads over the surface. The diffusion on the surface is used here to evaluate the diffusivity of the material along a chosen direction, integrating the temperature map, perpendicularly. The spatial Fourier Transform (FFT) is computed for every temperature profile in the sequence. After that, each FFT component is divided by the zero\(^{th}\) component (the continuous one) to get rid of the effect of in-depth diffusion. Then, its behaviour in time is considered. It follows theoretically an exponential decay whose time constant depends on the material diffusivity and the square of the selected space frequency \((k)\) [4]. Therefore, the slope of the data linearly fitted in logarithmic scale gives the thermal diffusivity, for each \(k\). Figure 2 shows some temperature profiles of the heated spot in time (left) and on the right the logarithmic decrease of the temperature vs. time, expressed in the frequency domain for: \(k_2, k_6, e k_{10}\).

![Figure 2. surface temperature profiles varying during the diffusivity measurement on the heated spot, in space at different time (left) and frequency-time domain (right).](image)

**Ultrasonic Pulse Velocity**

The UPV measurements both for the fresh and water-aged AAC samples were taken in the direct transmission (cross direction) and indirect transmission modes (parallel to the surface) by using a portable **PUNDIT PLUS CNS FARNELL** instrument with 220 kHz transmitters (see Fig.3). Direct UPV measurements were taken on smaller samples with two different thicknesses to use this technique determining the soundness of AAC and apply for monitoring the weathered zone/depth. The indirect UPV measurements were also taken from the fresh samples in the form of rectangular prism with 20 cm length. In this case, the probes were used at the same surface: the transmitter was placed on a fixed point of the surface and the receiver was placed at successive positions along a line. The
transmit time was plotted in relation to the distance between the centres of transducers. Any change on the slope of the plot would exhibit the change in the velocity in depth [9]. The depth attainable with the indirect UPV measurements was calculated according to the technique developed by the Christaras [9]. The UPV measurements were taken on dry samples. The direct UPV measurements were conducted on eleven samples and five readings were taken for each measurement from all directions. For the indirect UPV measurements, three surfaces/sides of sixteen samples were examined by taking five readings for each measurement.

Figure 3. Schematic drawing of UPV measurements in the direct (transmission) and indirect modes, showing the location of Transmitter and Receiver and the distance between probes travelled by the wave (at the top); views of the experimental setup for the UPV measurements, both for direct (left bottom) and indirect transmission modes (right bottom).

Analytical Analyses

The physical properties of the fresh and water-aged AAC samples were determined in terms of bulk density, effective porosity and water absorption capacity by weight according to the RILEM standard [7]. The mineralogical/compositional properties of the surface both for the fresh and water-aged samples were studied by means of image analyses of thin sections and XRD analyses. Thin sections were examined by optical microscope, LEICA DM EP, in x2.5, x10 and x20 magnification. Such a petrographical examination of thin sections is done under normal light (single nicol) and under polarized light (cross nicol). The XRD analyses were done on the powders taken from the surfaces of the samples by using a Phillips PV 3710 X-Ray Diffractometer, with CuKα X-Rays.

Results and discussion

The non-destructive examination of fresh and weathered AAC were done in terms of the assessment of the mechanical strength and thermal diffusivity. The combined interpretation of results were summarized below under respective headings and Tables 1-3.
For all sets of samples: the thin and the thick ones, an increase in effective porosity and water absorption capacity was observed at the water-aged samples while their densities were found to be almost constant.

**Non-destructive assessment of the mechanical strength**

The water-aged thin samples, having a thickness around 2cm, presented a considerable decrease in the direct $UPV$ and $E_{mod}$ values, nearly halving their initial values (see Table1). Such a significant decrease was also pointed out in terms of wet-to-dry uniaxial compressive strength ratio for the 5 cm cubic-sized AAC samples [1,3]. According to the thin section analyses of the thin samples, no significant change was observed between the fresh and water-aged ones in terms of pore size and mineral structure (Figures 4 and 5). However, the images with larger magnification showed the existence of some capillary cracks between the pores of water-aged AAC samples (Figure6). These cracks may cause a considerable decrease in UPV measurements for the water-aged samples.

In contrast with this, the thermal diffusivity values were found to increase on the surface of the same water-aged samples (see Table 3). This may indicate the variations in the thermophysical properties from the exposed surface towards the depth.

![Figure 4](image1.png)  ![Figure 5](image2.png)

**Figure 4 - Thin section images of G2 samples under cross nicol showing the pore size and distribution for the fresh one (at the left) [3, p.595] and for the water-aged one left in water for 20 days (at the right).**

**Figure 5 - Thin section images of G4 samples under cross nicol showing the pore size and distribution for the fresh one (at the left) [3, p.595] and for the water-aged one left in water for 20 days (at the right).**

![Figure 6](image3.png)

**Figure 6 - Thin section images of the water-aged G4 sample left in soaked conditions for 20 days showing the crack formation between the pores: single nicol (at the left); cross nicols (at the right).**

UPV values measured on the thinner fresh samples appear higher than on thicker one, and the more compact AAC type (G4) shows higher values than G2, as expected. Consequently, small changes in mechanical strength of water-aged AAC samples observed on cubic samples by using direct $UPV$ measurements seem not significative (see Table 2).
Generally, a moderate reduction on both the $UPV$ and $E_{mod}$ values for both the G2 and G4 samples has been detected.

Table 1: Physical and mechanical property for fresh and water-aged samples (water-soaked for 6 days and 20 days), the thicknesses range is 2.1-2.4 cm.

<table>
<thead>
<tr>
<th>AAC samples: thickness 2.1-2.4 cm</th>
<th>sample code</th>
<th>bulk density $\rho$ [g cm$^{-3}$]</th>
<th>effective porosity $\phi$ [%]</th>
<th>water absorption capacity $\theta_{max}$ [% by weight]</th>
<th>$UPV$ in direct direction [m s$^{-1}$]</th>
<th>$E_{mod}$ [GPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fresh samples</td>
<td>G2K5</td>
<td>0.41</td>
<td>78</td>
<td>193</td>
<td>1974</td>
<td>1.456</td>
</tr>
<tr>
<td></td>
<td>G4K5</td>
<td>0.62</td>
<td>69</td>
<td>114</td>
<td>2283</td>
<td>2.967</td>
</tr>
<tr>
<td>water-aged in water for 6 days</td>
<td>G2K4</td>
<td>0.41</td>
<td>83</td>
<td>205</td>
<td>1850</td>
<td>1.283</td>
</tr>
<tr>
<td></td>
<td>G4K4</td>
<td>0.63</td>
<td>71</td>
<td>113</td>
<td>1610</td>
<td>1.499</td>
</tr>
<tr>
<td>water-aged in water for 20 days</td>
<td>G2K4/4</td>
<td>0.41</td>
<td>83</td>
<td>204</td>
<td>1493</td>
<td>0.833</td>
</tr>
<tr>
<td></td>
<td>G4K4/3</td>
<td>0.59</td>
<td>76</td>
<td>130</td>
<td>1566</td>
<td>1.329</td>
</tr>
</tbody>
</table>

Table 2: Physical and mechanical property for fresh and water-aged cubic samples (water soaked for 30 days; size is 5 x 5 x 5cm.

<table>
<thead>
<tr>
<th>AAC samples: thickness 5 cm</th>
<th>sample code</th>
<th>bulk density $\rho$ [g cm$^{-3}$]</th>
<th>effective porosity $\phi$ [%]</th>
<th>water absorption capacity $\theta_{max}$ [% by weight]</th>
<th>$UPV$ in cross direction [m s$^{-1}$]</th>
<th>$E_{mod}$ [GPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fresh samples</td>
<td>G2U3</td>
<td>0.40</td>
<td>78</td>
<td>193</td>
<td>1802</td>
<td>1.202</td>
</tr>
<tr>
<td></td>
<td>G4U2</td>
<td>0.61</td>
<td>69</td>
<td>114</td>
<td>1952</td>
<td>2.134</td>
</tr>
<tr>
<td>Water-aged in water for 30 days</td>
<td>G2U3</td>
<td>0.40</td>
<td>83</td>
<td>208</td>
<td>1792</td>
<td>1.183</td>
</tr>
<tr>
<td></td>
<td>G4U2</td>
<td>0.61</td>
<td>74</td>
<td>121</td>
<td>1907</td>
<td>2.037</td>
</tr>
</tbody>
</table>

The $E_{mod}$ values of fresh G2 and G4 samples calculated indirectly by using the $UPV$ measurements in cross transmission mode were found to be consistent with the $E_{mod}$ values defined in the specifications for AAC material [2,10]. This indicated that the method developed for the indirectly-determination of $E_{mod}$ values for natural stones by using the direct UPV measurements [7,8] can be used for the AAC material as well.

**Thermal diffusivity measurements**

The results of the thermal diffusivity measurements were reported in Table 3. What appears is the increase of the diffusivity from the fresh samples to the aged ones in the G2 serie, while the G4 present a maximum of diffusivity in 6 days aged sample. Data are substantially confirmed by the two tests, even if with a spread between the two, due to the difficulty of the measurement connected to the high porosity of the samples and the relatively small area affected by thermal diffusion. Another raising aspect from the diffusivity experiments is the support to a possible inhomogeneity of the material in depth. In fact, the thermal 1D model is expected to be that of a semi-infinite body, given the thickness of the samples, with a temperature versus time decreasing slope of -0.5 in the log-log scale. What we found experimentally was a deviation from such a model with evidence of a variation in the thermophysical properties from the surface to the depth of the material. We remember that the diffusivity measurement with the technique here used, for its possible in-field application is sensitive to a shallow layer close to the surface. That
may explain some discrepancy with the data coming from the ultrasound velocity measurements that are mainly related to the bulk.

Table 3: Diffusivity measurement by one side QIRT, on samples treated as in Tab. 1

<table>
<thead>
<tr>
<th>AAC samples with thicknesses in the range of 2.1cm and 2.4 cm thickness</th>
<th>sample code</th>
<th>diffusivity test 1 [m²s⁻¹]</th>
<th>diffusivity test 2 [m²s⁻¹]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fresh samples</td>
<td>G2K5</td>
<td>2.4×10⁻⁷</td>
<td>2.2×10⁻⁷</td>
</tr>
<tr>
<td></td>
<td>G4K5</td>
<td>2.8×10⁻⁷</td>
<td>2.5×10⁻⁷</td>
</tr>
<tr>
<td>Water-aged samples in water for 6 days</td>
<td>G2K4</td>
<td>3.9×10⁻⁷</td>
<td>3.4×10⁻⁷</td>
</tr>
<tr>
<td></td>
<td>G4K4</td>
<td>4.4×10⁻⁷</td>
<td>4.0×10⁻⁷</td>
</tr>
<tr>
<td>Water-aged samples in water for 20 days</td>
<td>G2K4/4</td>
<td>4.7×10⁻⁷</td>
<td>3.4×10⁻⁷</td>
</tr>
<tr>
<td></td>
<td>G4K4/3</td>
<td>4.0×10⁻⁷</td>
<td>3.3×10⁻⁷</td>
</tr>
</tbody>
</table>

Non-destructive examination of surface deterioration

It is known that, the main mineral in the composition of AAC is tobermorite-11Å [2,3], which may react with atmospheric carbondioxide (CO₂) gas in the presence of moisture and be converted to silica and calcium carbonate [10]. The results obtained from the XRD analyses of the surface powder and the thermal diffusivity measurements of the surfaces were interpreted together to examine surface carbonation on water-aged AAC samples. The indirect UPV data for the sound sample surface of AAC were also produced. It is expected to be useful for the assessment of the weathering state of AAC surfaces.

Figure 7. The XRD patterns of the surface powder taken from the fresh and water-aged G2 samples, showing the increase in calcite at the surfaces of the water-aged samples: tobermorite 11Å (T), quartz (Q) and calcite (C). The calcite peaks increased in intensity at wet-aged samples were shown in red.

Figure 8. The XRD patterns of the surface powder taken from the fresh and water-aged G4 samples, showing the increase in calcite at the surfaces of the water-aged samples: tobermorite 11Å (T), quartz (Q) and calcite (C). The calcite peaks increased in intensity at wet-aged samples were shown in red.
The XRD patterns of surface powder taken from the fresh and water-aged AAC samples were given in Figures 7 and 8. The XRD traces of the surface zones showed that the main minerals detected were tobermorite 11Å, quartz and calcite. The calcite in the fresh samples was also observed in the images of thin sections (Figure 9) [3]. The calcite pattern became more visible in the XRD traces of surface powder taken from the water-aged samples. This meant that the conversion of tobermorite-11Å into calcite and silica, as previously indicated by some researchers [10], happened in water-aged samples. Increase in the calcite can be expected to be more on the exposed surfaces, periodically getting wet by rainwater. The formation of surface carbonation as the weathering product on the water-aged samples can affect the thermophysical properties. Those effects may be followed by the thermal diffusivity measurements as proved by the results presented in Table 3.

Fig.8. (left image x10 magnification): Thin section under cross nicol for the fresh G2 sample; (right image x20) fresh G4 sample, showing the minerals: quartz (Q), muscovite (mu), mica (M), micritic limestone (ml), quartzite (Qt), calcite (C), opaque mineral of limonite (L) and clay-size sub microscopic mineral (S)

The extent of carbonation is expected to be followed by the indirect UPV measurements. For this purpose, the relationship between the sound and weathered AAC should be investigated. Preliminary results show that the variations in the indirect UPV measurements were the same for the fresh G2 and G4 samples. The fitting of data shows a linear relationship between the ultrasonic pulse travel time and the travel distance. The Table 4 gives speed data, considered correct up to the depth of 7.2mm [8]. The indirect UPV values for the sound surfaces of AAC samples were found to be 722±69 m/s, indicating results with a moderate accuracy. This statement must be borne in mind analyzing variation in the indirect UPV data for the sound and weathered samples.

Table 4: indirect UPV measurements on fresh AAC long samples (5cm x 5cm x 20cm)

<table>
<thead>
<tr>
<th>Travel distance [mm]</th>
<th>Travel time [ms]</th>
<th>Indirect UPV [m s⁻¹]</th>
<th>depth [mm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>15</td>
<td>24.1</td>
<td>622</td>
<td>-</td>
</tr>
<tr>
<td>30</td>
<td>42.5</td>
<td>706</td>
<td>2</td>
</tr>
<tr>
<td>45</td>
<td>58.1</td>
<td>775</td>
<td>3</td>
</tr>
<tr>
<td>60</td>
<td>75.2</td>
<td>798</td>
<td>3</td>
</tr>
<tr>
<td>75</td>
<td>105.5</td>
<td>711</td>
<td>7</td>
</tr>
</tbody>
</table>
Conclusions

The combined interpretation of the QIRT and UPV measurements gave information on mechanical and thermal properties of AAC. The grade of deterioration of water-aged AAC samples has been explored by means of the joint-use of these NDT methods, as well. Further supportive investigations have been applied to several samples of such a highly-porous and non-homogeneous materials and results analyzed in terms of physical mineralogical and mechanical properties.

The UPV measurements taken in the direct mode and derived mechanical strength exhibited a significant reduction for the water-aged AAC samples having the thickness around 2 cm. In contrast with this, the reduction in UPV on the thicker water-aged AAC samples was much lower. The reason for this difference and the slight increase in effective porosity is given to tiny cracks occurred mainly on a shallow layer, close to the surface of the water-aged AAC samples, which affect the average UPS with a weight inversely proportional to the thickness.

On the other hand, the thermal diffusivity values were found to increase on the surface of the same water-aged samples, which indicates the variations in thermophysical properties from the exposed surface towards the depth. The explanation of this behaviour is at the moment related with the differentiated ageing of the surface not significantly affecting the bulk of the material. Furthermore, the surface deterioration seems to start very soon in time, because the diffusivity increase at more or less the same value for all the aged samples, independently from the days of treatment.

Laboratory analyses as XRD revealed also an increase in calcite on the surfaces of water-aged AAC samples which may affect the thermophysical properties of the boundary surfaces. It is worth mentioning that the thermal diffusivity measurements seemed to reflect the change in the mineral composition of the water-aged AAC surfaces.

The $E_{mod}$ values have been calculated indirectly by the direct UPV and density values. The indirect UPV measurements joined to the QIRT diffusivity were thought to be promising for the assessment of the weathering state, for the sound and weathered surfaces.

Further studies are necessary on the development of this approach. In fact, some difficulty has been over passed in measuring the thermal diffusivity and UPV properties of a highly-porous and non-homogeneous material. Comprehensive studies are also necessary to better understand the formation of deterioration product(s) and their effect to the material properties and to interpret the data carefully obtained from those NDT techniques.

Bibliografía


