1 INTRODUCTION

Cement hydration plays a critical role in the temperature development of early-age concrete due to the heat generation\textsuperscript{1–4}. It also has significant effects on the material properties and performances at an early age, including material strengths\textsuperscript{5}, critical stresses\textsuperscript{6} and distresses like cracking\textsuperscript{7}. Therefore, it is essential to capture the hydration property and temperature development of concrete at an early age to prevent premature failures. It is known that the durability of cement-based products and concrete structures is highly influenced by the early stages of hydration\textsuperscript{8}. A precise knowledge of the micro-mechanical properties during the successive phases of the hydration process provides information on the concrete resistance and allows an assessment of its durability\textsuperscript{9}. Several non-destructive techniques have been developed and applied in that respect, most of them based on ultrasonic-wave measurements\textsuperscript{10}. The recording of passive energy using acoustic-emission techniques was used to evaluate the structural activity in concrete during the early ages, showing periods of intense micro-structural changes during the curing process\textsuperscript{11}.

Microstructural changes occurring in freshly poured concrete during the curing were monitored on a laboratory scale using a combination of the acoustic-emission technique and the monitoring of electrical properties\textsuperscript{12}. The acoustic-emission method is a passive-ultrasonic-
signal recording technique allowing an online monitoring of the internal microstructural activity of young concrete during the hydration process. The acoustic-emission technique, which is one of the non-destructive evaluation techniques, is a useful method for investigating local damage in concrete during its lifetime. The micro-changes in concrete structures can be easily estimated, taking account of the number of acoustic-emission hits and counts of the acoustic-emission signal. Acoustic-emission signals are detected, due to micro-cracks (i.e., structural changes in the material) by the acoustic-emission sensors attached to the surface of a concrete specimen. The acoustic-emission parameters such as the number of hits, counts, duration time, amplitude, energy and rise time are recorded by the acoustic-emission measurement system.

Impedance spectroscopy, called also dielectric spectroscopy, the monitoring of electrical properties and nuclear-magnetic-resonance spectroscopy have become promising techniques for probing cements and concretes. Cement paste is electrically conductive due to its interconnected pore network filled with an aqueous phase containing mobile ions such as Na+, K+ and OH−. The impedance of the material under investigation is obvious. The capacity C of a parallel-plate capacitor is computed with:

\[ C(f) = \varepsilon_r \cdot \varepsilon_0 \frac{S}{d} \]  

where \( \varepsilon_r \) is the relative permittivity, \( \varepsilon_0 \) is the vacuum permittivity (8.854 \cdot 10^{-12} F/m), S is the measuring-electrode area, d is the distance and f is the frequency. The resistance, R, of the electrodes is given by:

\[ R(f) = \rho \cdot \frac{d}{S} \]  

where \( \rho \) is the resistivity. Microstructural changes in a material make the material’s permittivity change. The permittivity value can also be affected by macrocracks, depending on the frequency.

Two cylindrical steel electrodes of a diameter of 6 mm, buried 65 mm under the specimen surface, were used for measuring the resistance. To measure the capacitance, two rectangular metal-plate electrodes with dimensions of 25 mm × 45 mm were applied. All the electrodes were fixed to a plastic slab so that their constant configuration was guaranteed. The electrodes and the temperature-sensor outputs were connected to an automated measuring device. The measurements of the capacitance, the temperature and the impedance were started within 15 min after the mixture preparation. This phase of the experiment was carried out using a TESLA BM 595 RLCG bridge and a selector switch. The measurement was carried out at selected points with frequencies in the range from 100 Hz to 20 kHz. Each of the electrical quantities (resistance, capacitance, temperature, etc.) was measured separately.

2 EXPERIMENTAL SET-UP

An acoustic-emission measuring system called LOCation ANalyser (LOCAN) 320 made by PAC (France) was used for the measurements. Wide-band sensors (made by the 3S Sedlák Company) were used. The sensor output was connected to pre-amplifiers (the PAC Company) with a 2 kHz high-pass filter. The proprietary PAC program package was used to record the acoustic-emission parameters. The acoustic-emission signals were taken by a LOCAN 320 acoustic-emission localizer. The LOCAN 320 system was fine-tuned at a well site and with a sonic-logging tool in the wellbore, with the acoustic threshold set slightly above the background-noise level as measured by LOCAN 320 and the internal gain set according to the manufacturer’s recommendations. This device was designed to record and evaluate ultrasonic signals. It was used to analyze and store the acoustic-emission parameters. It can process signals coming from four measuring points. Each recorded event can be characterized with the following acoustic-emission parameters: recording date and time, (maximum) amplitude, acoustic-emission energy, count, rise time and mean frequency.

The impedance of the material under investigation changes in accordance with the structural changes, particularly the water absorption and evaporation. A change in the resistance is obvious. The capacity C of a parallel-plate capacitor is computed with:

\[ C(f) = \varepsilon_r \cdot \varepsilon_0 \frac{S}{d} \]  

where \( \varepsilon_r \) is the relative permittivity, \( \varepsilon_0 \) is the vacuum permittivity (8.854 \cdot 10^{-12} F/m), S is the measuring-electrode area, d is the distance and f is the frequency. The resistance, R, of the electrodes is given by:

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Figure 1: Experimental set-up before unmoulding, (samples S1, S2 tested with the acoustic-emission method and S3, S4 with impedance spectroscopy)

Slika 1: Eksperimentalni sestav pred razdrtjem (vzorca S1, S2 preizkušena z metodo akustične emisije in S3, S4 z impedančno spektroskopijo)
Sleeper concrete with a 32 mm crushed aggregate attained a compression strength of 107 MPa. Two of the specimens with dimensions of 100 mm × 100 mm × 400 mm were mould cast. Steel-mould upper sides were covered with PE foil (Figure 1). The components of the mixture are shown in Table 1. The mechanical properties of the monitored samples are statistically processed in Table 2. When the specimens were unmoulded, 24 h after the mixing, one specimen was covered with foil and the other one was not (Figure 2). In Figures 1 and 2, samples S1 and S2 are tested with the acoustic-emission method and samples S3 and S4 with impedance spectroscopy; samples S1 and S4 are covered.

Table 1: Concrete mixture

<table>
<thead>
<tr>
<th>Composition</th>
<th>Mass, m/kg</th>
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</thead>
<tbody>
<tr>
<td>Cement CEM I 42.5R</td>
<td>390</td>
</tr>
<tr>
<td>Water</td>
<td>125</td>
</tr>
<tr>
<td>Plasticizer Stachement 2060</td>
<td>4</td>
</tr>
<tr>
<td>Sand 0/4</td>
<td>700</td>
</tr>
<tr>
<td>Coarse aggregate 16/32</td>
<td>1275</td>
</tr>
</tbody>
</table>

3 RESULTS

The acoustic-emission activities of both samples during hydration, hardening and setting are shown in Figures 3 and 4. After the unmoulding, i.e., at the age of 24 h, when one sample was wrapped and the other one was left without any protection, the acoustic-emission activities were different. The increase in the cumulative acoustic-emission-count curve of the protected sample is lower than that of the sample without protection during the whole 120 d period, as shown in Figure 4. It should be noted that the acoustic-emission signals were picked up six hours after the mixtures were made.

The temperature of the samples after the unmoulding was kept close to the ambient temperature (Figure 3). The basic changes in the concrete structure during the hydration relate to the temperature peak, in this case after 12 h, as shown in Figure 4. The temperature curves for both samples are the same as before the unmoulding, i.e., slightly different.

Electrical measurements show a higher resistivity and a lower capacity, i.e., a higher capacitance of the specimen without protection after the unmoulding (Figure 5).

Different changes of both samples are also shown in Figure 6. On the first day the frequency characteristics of resistance (R) and capacitance (X_C) are the same, but thereafter they are different. There are three types of curves described in the legend in Figure 6. Each curve, as described on the curve at the 6th h, consists of eight points whose frequencies from the left-hand side of the horizontal axis are 100 Hz, 200 Hz, 400 Hz, 1 kHz,

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It is clear that the shapes of the curves are different after the unmoulding, therefore, the curves of both samples are the same on the first day (in Figure 6 marked with downward-pointing triangles).

Table 2: Main parameters of samples

<table>
<thead>
<tr>
<th></th>
<th>Units</th>
<th>Mean</th>
<th>Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Compressive strength</td>
<td>MPa</td>
<td>107</td>
<td>5</td>
</tr>
<tr>
<td>Flexural strength</td>
<td>MPa</td>
<td>10.0</td>
<td>0.2</td>
</tr>
<tr>
<td>Modulus of elasticity (bending)</td>
<td>GPa</td>
<td>37</td>
<td>3</td>
</tr>
<tr>
<td>Fracture toughness</td>
<td>MPa m$^{1/2}$</td>
<td>1.8</td>
<td>0.4</td>
</tr>
<tr>
<td>Fracture energy</td>
<td>J/m$^2$</td>
<td>200</td>
<td>10</td>
</tr>
</tbody>
</table>

4 CONCLUSION

The acoustic-emission method and impedance spectroscopy are powerful tools for determining the development of microcracks during the hardening and setting of concrete. It can be assumed that higher numbers of microcracks cause higher numbers of acoustic-emission events. The dependence of a cumulative acoustic-emission activity on the time shows the necessity of curing the concrete during its whole lifetime, especially during intense hydration and hardening. The big differences between the acoustic-emission activities of wrapped and unprotected samples clearly indicate the essentiality of the high moistness of hardened concrete. The number of microcracks in cement mortar affects the resulting mechanical properties of concrete.

Impedance, or dielectric, spectroscopy together with acoustic emission can also help with the monitoring and a detailed description of lifetime behaviours of concrete specimens during hydration.

As the acoustic emission (Figure 3) and the impedance (Figure 5) history of both samples are similar, the number of microcracks is higher in the samples without foil, which means that the curing of concrete during an early age is necessary.

Acknowledgement

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5 REFERENCES
