

Monitoring of self-healing cementitious materials through contactless ultrasound

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Abstract. Self-healing cementitious composites provide an alternative to labour-intensive and costly manual repairs. While a cementitious blend possesses an inherent ability to repair its own damage through autogenous healing, an enhancement of the self-healing capacity can be obtained through the inclusion of superabsorbent polymers (SAPs). The implementation of such innovative materials within the construction industry requires proper evaluation methods to ensure a safe environment for the user. Over the past few years, contact ultrasonic measurements have proven their potential in assessing the self-healing progress. The sensitivity of ultrasonic waves to the elastic properties of the material under study allows for a direct link with the regained mechanical performance. Additionally, its non-destructive nature enables in-situ evaluations. However, the coupling of the sensors leads to a certain variability in the obtained results, as the application of the sensors is not identical between measurements. In an effort to increase the reliability of the results, contactless ultrasound can be applied, which is investigated in the present research.

1 Introduction

Concrete is one of the most used construction materials, due to its high compressive strength. However, when tensile stresses occur, cracks are easily formed. As these cracks provide pathways for the ingress of deleterious substances, repair and maintenance of concrete structures are of utmost importance. To limit the costs related to restorations, self-healing cementitious materials have been investigated. These mixtures present the ability to automatically repair incurred damage through so-called autogenous healing [1]. The main mechanisms that contribute to autogenous healing are the continued hydration of unhydrated cement and the precipitation of calcium carbonate. For these processes to take place, water is essential to be present, which limits the extent and effectiveness of autogenous crack healing. To promote the closure of cracks, improvements can be made to the mixture design through the addition of various additives. An example of such inclusions that are thoroughly investigated are superabsorbent polymers (SAPs) [2-4]. As their name implies, SAPs are able to absorb high amounts of water. In case of moisture ingress through the cracks, the SAPs swell and retain the water for a certain period of time. Later, upon dry periods, this water is released again to the matrix and the chemical processes leading to autogenous healing are initiated.

Over the past years, measuring techniques based on elastic wave propagation have gained ground for the characterization of cementitious materials. The strength of such techniques lies in the sensitivity of ultrasonic waves to the elastic properties of the material they travel

through. In this way, various features such as setting time [5], mechanical performance [6] and repair effectiveness [7-8] can be investigated in a non-destructive, easily applicable way. Concerning the evaluation of self-healing specifically, ultrasound showed the potential to distinguish between the uncracked, cracked and healed stages through an analysis of the wave parameters. For instance, the longitudinal wave velocity reduced upon cracking, whereas after a healing period a partial restoration of this parameter was noticed that could be linked to the visual closure of cracks [8]. Until now, mostly contact ultrasonic measurements have been used, despite the fact that the coupling of the sensors induces some variations in the obtained results. As a solution, contactless ultrasound could be applied [9-11], but presents the disadvantage of high attenuation, due to the air gap between sensor and specimen, which leads to low signal-to-noise ratios.

In this study, the evolution of the crack closure was monitored through microscopic analysis and surface wave ultrasonic measurements. Additionally, contactless measurements were compared to more common coupled ultrasonic measurements, in order to benchmark this new technique. The signal-to-noise ratio of the contactless results was increased by applying stacking of few hundreds of waveforms. This technique reduces the random noise captured by the contactless transducers and increases the reliability of the obtained results. Concerning the cementitious mixtures studied, mortar specimens with and without superabsorbent polymers were investigated. The outcome revealed the potential of contactless ultrasound to monitor the self-

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healing progress, as well as the improved crack closure effectiveness within mixtures with SAPs.

2 Materials and methodologies

2.1 Materials

To investigate the effect of SAPs on the self-healing ability, a reference mixture and a mixture with SAPs (called VP400) were cast. The mortar consisted of a high-strength Portland cement, namely CEM I 52.5 N from Holcim. River sand 0/2 was added in an amount of 2 to 1 with respect of the weight of cement. The water-to-cement ratio was equal to 0.35. Superplasticizer MasterGlenium 51 from BASF was added in an amount of 0.4% w.r.t. the weight of cement to increase the workability.

The SAP used is called VP400, obtained from BASF, Germany. It is a copolymer of acrylamide and sodium acrylate and is produced by bulk polymerization. The dry particle size is equal to $100 \pm 21.5 \mu\text{m}$. The amount of SAPs included was equal to 0.5% by weight of cement. As the SAPs absorb water during mixing, an additional amount of 26 g of water per gram of SAP was included on top, so that an equal workability was obtained for both mixtures. The workability was measured through a flow table test, following NBN EN 1015-3 [12].

For each mixture, four mortar prisms were cast, measuring 40 mm x 40 mm x 160 mm, and cured for 28 days in plastic foil at $20 \pm 2^\circ\text{C}$. On one of the long faces, a carbon fiber reinforced polymer (CFRP) laminate was glued two days before cracking, in order to keep the specimens together after breaking. After the 28-day curing period, the specimens were cracked in a three-point bending set-up. To limit the crack widths, a metal frame was placed around the cracked specimens, which pushed the two halves together. By placing the specimens underneath the microscope, an average crack width around $150 \mu\text{m}$ was obtained after restraining.

The cracked specimens were placed in wet-dry curing cycles to improve the autogenous healing mechanisms. The cycles consisted of 23 hours in dry conditions at $20 \pm 2^\circ\text{C}$ and $65 \pm 5\%$ RH and 1 hour submersion in water at $20 \pm 2^\circ\text{C}$.

2.2 Microscopic analysis

Microscopic analysis was performed to follow-up the visual crack closure during wet-dry curing. An optical microscope Leica S8 APO, mounted with a DFC 295 camera was used. Crack width measurements were conducted in two positions along the crack length. For every position, one picture was taken and per picture five measurements of the crack width were conducted. A first measurement took place during restraining of the specimens, leading to an average crack width equal to $150 \mu\text{m}$. Afterwards, repetitions were executed in the exact same locations after 3, 7, 14 and 28 days of healing.

2.3 Surface wave ultrasonic measurements

The healing progress was monitored through ultrasonic surface wave measurements. The coupled ultrasound set-up is depicted in Fig. 1 and consisted of three piezoelectric R15a sensors, having a resonant frequency of 150 kHz. The sensors were attached to the mortar specimens by using Vaseline as a coupling agent. The distance between the sensors was fixed at 35 mm. The excitation of the wave signal was done using a waveform generator, which sent a single cycle sine wave with frequency of 150 kHz to one of the transducers. The other two sensors were used as receivers. For each measuring moment, five waveforms were sent and analyzed.

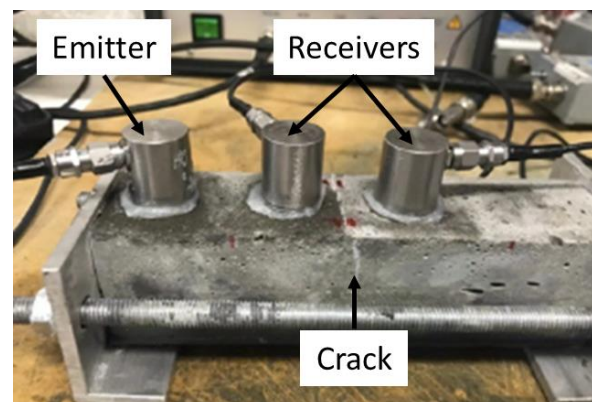


Fig. 1. Coupled ultrasonic set-up.

In case of the contactless ultrasonic set-up, again a coupled R15a sensor was used as emitter, because the contactless transducers used in this study can only be utilized as receivers. The receivers were now changed to contactless transducers of type AM41 (Fig. 2). The placement of the sensors onto the specimens was done by using a PVC mould, to ensure a distance of 35 mm between the receivers. The amplitude of the emitted signal was increased up to 200 V and the number of cycles was augmented to 20, taking into account the higher attenuation occurring during contactless testing. Concerning the frequency of the sent signals, a preliminary analysis was performed and showed maximum sensitivity at a frequency equal to 140 kHz. Five hundred signals were emitted for the contactless set-up. From these signals, an average response was calculated, showing a reduction of the random noise captured during the experiments and an improvement in the signal-to-noise ratio from the broadly 50 to 250.

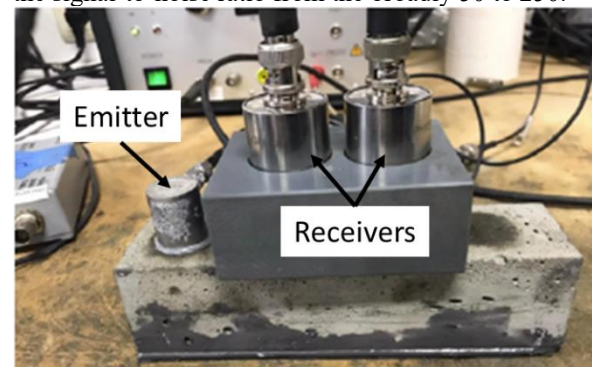


Fig. 2. Contactless ultrasonic set-up.

3 Results and discussion

3.1 Microscopic analysis

In Fig. 3, the evolution of the average crack width measured in reference and VP400 specimens is shown. These average values are based on the 10 measurements conducted per sample and this for four specimens per mixture design. Immediately after cracking, a crack width of about 150 μm was obtained thanks to the restraining of the specimens. The mortar samples were then placed in wet-dry curing cycles and a fast decrease of the crack opening was noticed during the first three days of curing for both mixture series. Afterwards, the crack size continued to reduce, but at a lower pace. When comparing the results of the reference and VP400 blends, a faster crack closure mechanism took place within the mixtures with SAPs. Finally, after 28 days of wet-dry curing, the VP400 mortar revealed an almost complete crack closure, confirming the improved healing ability upon SAP inclusion.

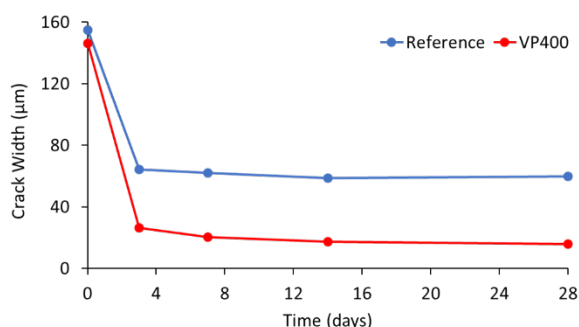


Fig. 3. Evolution of the average crack width during healing for reference and VP400 mortars.

3.2 Surface wave ultrasonic measurements

Before detailing the analysis of the healing evolution, a view of the raw data received during the ultrasonic measurements is shown in Fig. 4. Fig. 4 (a) reveals the waveforms received by the first (closest) and second (furthest) sensor after a single ultrasonic experiment on an uncracked reference mortar. The signal received by the first sensor is nearly identical for all experiments conducted, as the wave arrives at this sensor before travelling through the crack. The moment of arrival is shown by the blue arrow and is taken as the first deviation from the threshold level, which is the maximum noise received during the pre-trigger stage. The wave received by the second sensor exhibits a lower amplitude due to the attenuative effect of the material. Also, the arrival of the wave is delayed compared to the first receiver (orange arrow), due to the longer travel path. Using the distance between the sensors of 0.035 m and the time difference between both arrivals, the longitudinal wave velocity can be calculated by dividing the distance by the travel time. In this specific example, a travel time of 8.7 μs was found, leading to a velocity of approximately 4022 m/s. Next to the wave velocity, the attenuation was calculated by eq. 1:

$$\text{Attenuation} \left[\frac{\text{dB}}{\text{mm}} \right] = -\frac{1}{35\text{mm}} * 20 * \log \left(\frac{A_{\text{furthest}}}{A_{\text{closest}}} \right) \quad (1)$$

where A signifies the amplitude of the signal, being the highest voltage received. The attenuation for the signals in Fig. 4 (a) is equal to 0.23 dB/mm.

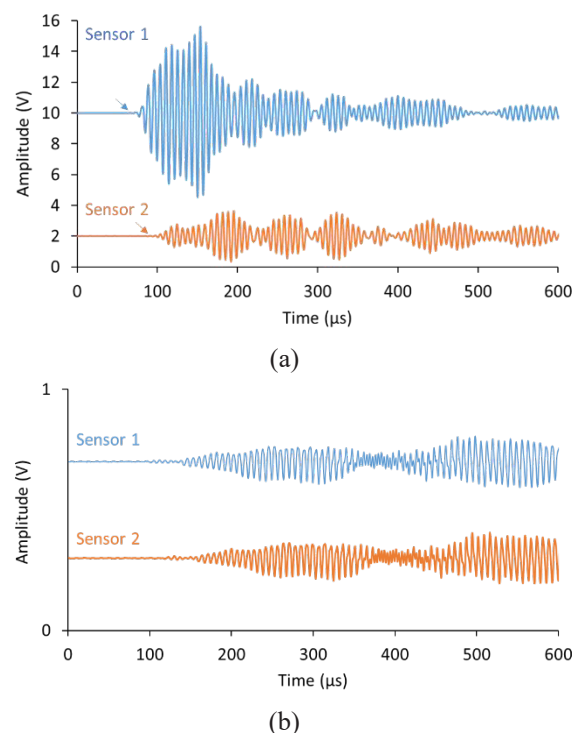


Fig. 4. Representative waveforms received for (a) coupled and (b) contactless ultrasonic testing on a reference specimen.

In Fig. 4 (b), example waveforms for contactless ultrasonic experiments are depicted. Compared to coupled ultrasound, the amplitudes received are much smaller, even though the emitted signal has an amplitude of 250 V. Due to the relatively low signal-to-noise ratio, picking of the arrival time is difficult. Therefore, stacking of hundreds of waveforms was applied to reduce the random noise received. From these stacked waves, a wave velocity of 2200 m/s was obtained and an attenuation equal to 0.01 dB/mm.

For further analysis of the self-healing evolution, the longitudinal wave velocity and attenuation were calculated for every ultrasonic measurement conducted and an average value was reported per mixture composition and per measuring day. The results of the wave velocity are shown in Fig. 5. On day zero, two velocity values per mixture are obtained. The higher values represent the uncracked state, while the lower ones are linked to the cracked situation. Focusing on the coupled ultrasonic results, the SAP mortar exhibited a lower velocity compared to the reference mixture in the uncracked state. This decrease in velocity can be explained by the higher porosity within these mixtures, as the SAPs shrink and leave behind voids. After cracking, a strong reduction of the wave velocity was noticed, which is linked to the discontinuity between the two receivers. The specimens were then placed in wet-dry curing cycles to promote the healing ability. Over time, a restoration of the velocity can be observed for

both mixtures, confirming the closure of cracks as seen by microscopy.

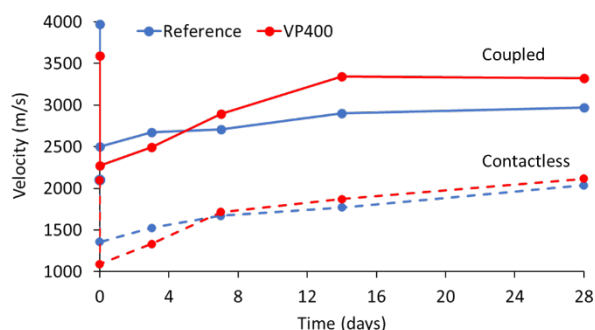


Fig. 5. Wave velocity vs. time of reference and SAP mortars during coupled and contactless ultrasonic experiments.

Concerning the contactless ultrasonic results, a similar trend with respect to coupled ultrasound was seen, though all values were significantly lower. The values of sound material are of the order of 2100 m/s. It is seen that the contactless sensors are capable of recording the leaky Rayleigh waves, since surface waves have stronger out of plane component, being at approximately 55% of the longitudinal wave velocity.

To obtain a more direct evaluation of the healing ability over time, the restoration of the velocity was calculated by the difference between the healed and cracked velocity values, divided by the difference between the uncracked and the cracked velocity values. In this way, velocity variations due to different porosity and crack size and geometry are omitted. Fig. 6 details the restoration percentage obtained from both ultrasonic test series. It can be noticed that the contactless results (dotted lines) always show higher recovery with respect to the coupled outcome. Still, when comparing the healing trends for the investigated mortars, both techniques systematically reveal a higher restoration for the VP400 mixture. This increased healing capacity was also seen during microscopic analysis and is due to the inclusion of the SAPs. Besides a confirmation of the visual crack closure, these results indicate the characterization potential of contactless ultrasound for self-healing.

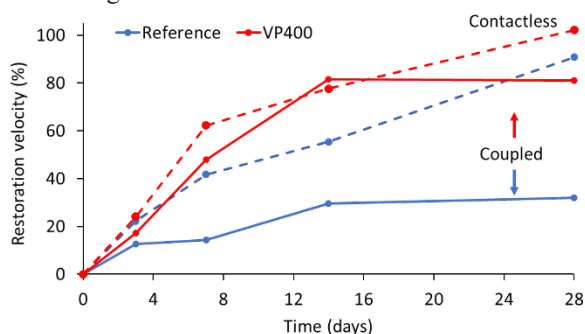


Fig. 6. Restoration of longitudinal wave velocity vs. time of reference and VP400 mortars.

Secondly, the attenuation was studied over time. The results for both coupled and contactless measurements are shown in Fig. 7. The attenuation of VP400 mixtures was slightly higher than the one of the reference material, caused by the higher porosity. After cracking,

the attenuation increases, as the air gap between the sensors provides a discontinuity. Thanks to the wet-dry curing cycles, the attenuation decreases again after day zero. This trend provides an additional confirmation on the partial closure of the cracks, as seen through microscopy.

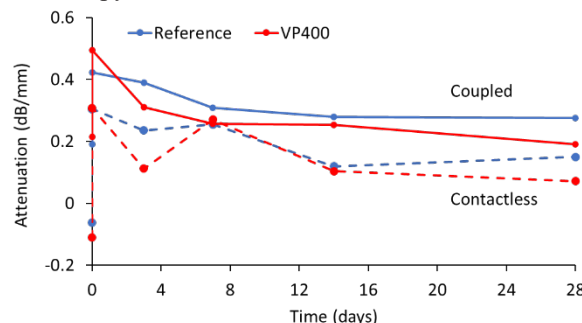


Fig. 7. Attenuation vs. time of reference and VP400 during coupled and contactless ultrasonic experiments.

When focusing on the contactless ultrasonic measurements, the general trends remain. Upon cracking, an increase in attenuation was seen, similar to the coupled experiments, as well as a decreasing trend upon wet-dry curing afterwards. The importance is on the trends that seem sensitive to the material status (cracked or healed). The initial attenuation is negative, something that could be the result of the small cross-section of the specimen, the limited beam spreading and possible reflections.

From the average attenuation values, a restoration percentage was calculated like the restoration of the wave velocities. The results are shown in Fig. 8. The trends obtained from coupled testing clearly reveal the improved healing ability upon SAP inclusion, which shows a final restoration of 110%. While microscopy shows only partial healing of the cracks, this high recovery can be possibly explained by deposition of healing products not only in the gap between the crack walls but also in the previously empty pores.

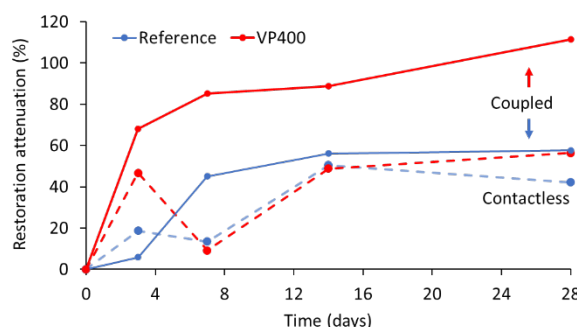


Fig. 8. Restoration of attenuation vs. time of reference and VP400 mortars.

When comparing the contactless results to the coupled data, a lower recovery was observed in the case of contactless testing. Nonetheless, a slightly higher restoration was obtained upon inclusion of VP400 after 28 days of healing. Again, these results reveal the potential of contactless ultrasonic measurements to follow-up the self-healing process. While the exact wave parameters calculated are not identical between

coupled and contactless testing, the evolution of the crack closure can be monitored through both techniques and is confirmed by microscopic measurements.

4 Conclusions

Within this study, an investigation of the self-healing ability of cementitious mixtures with and without superabsorbent polymers was performed. The healing process was promoted through the application of wet-dry curing cycles. An evaluation of the crack closure over time was done by means of microscopic analysis and ultrasonic testing, which included coupled and contactless measurements.

The visual closure of cracks was demonstrated through microscopy. While both mixtures studied showed a strong reduction of the crack width during wet-dry curing, the inclusion of SAPs improved the healing ability and an almost complete crack filling was obtained after 28 days.

The ultrasonic surface wave measurements enabled a characterization of various properties of cementitious materials by an analysis of the wave velocity and the attenuation, and this for both measurement techniques. The experiments conducted in the uncracked state revealed the increased porosity within mixtures with SAPs by a decrease in velocity and an increase in attenuation. Upon cracking, the presence of the discontinuity was noticed by a reduction in velocity up to 50%, while the attenuation increased strongly. The placement in wet-dry curing cycles induced a restoration of both parameters and confirmed the improved healing ability in mixtures with SAPs, as seen through microscopy.

A comparison between coupled and contactless ultrasound showed that similar trends were observed upon cracking and during healing, even though with different absolute values. Additionally, the promoted healing capacity of mixtures with SAPs could be noticed from both techniques. More research should be conducted on the mechanics of propagation in each case to validate the different restoration degrees in velocity and attenuation; however, these results validate the potential of contactless ultrasound to monitor the self-healing progress within cementitious materials. The adoption of this technique not only obviates the coupling variability on the ultrasonic results, but paves the way for automated measurements, leading to a faster and cost-effective assessment of present infrastructures.

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