Self-healing evaluation by optical microscope: An Approach to image acquisition, processing and analysis

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Abstract: Buildings are subject to weather and continuous use for human activities. Between its implications is cracks, which reduce these buildings' life cycle, especially reinforced concrete structures. Hence, several technologies have been developed to perform the self-healing phenomenon of cement-based materials, among which is the use of crystalline admixtures, supplementary cement materials, and bacteria that promote microbiologically induced calcium carbonate precipitation (MICP). Optical microscopy and image processing are used for crack closure analysis and are extensively used to monitor the self-healing agent efficiency in cementitious matrices. The self-healing measurement is directly dependent on the correct light exposure, imaging, and image processing. Due to its intrinsic characteristics - geometry, roughness, and porosity -, concrete cracks are tricky to work with. Thus it is necessary to determine a standard methodology that allows establishing a correlation between the approaches adopted by different researchers. This article compares two pipelines based on image analysis for crack closure behavior quantification through the number of pixels - the area (by scale factors). For such, two concretes with a 0.4 water/cement ratio were evaluated with and without crystalline additive (CA), exposed to wetting and drying cycles (2 and 5 days, respectively). The cracking process was at day three and monitored by optical microscopy until the age of 45 days. Two approaches are proposed in this study, both employing the ImageJ software, covering two magnifications (80x and 160x) and the same capture parameters. The wetting and drying cycle favors the self-healing process, for both concretes. Magnification influences the crack area quantification. Proposed methods cannot be differentiated statistically, though automated qualitatively seems to be more promising when compared to the manual method.

Keywords: Optical Microscopy, self-healing, crystalline Additives, supplementary cementitious materials.

1. Introduction

Among the pathological manifestations of civil constructions, according to Nunes, Vaz, Tavares (2017, p. 113), the most common are cracks of several factors that may result from the construction process and the mechanical stresses during their life cycle. Cracks can impair the building's structural performance, being the gateway for aggressive agents, so there is a need to promote their closure in an easy, fast and reduced cost way for the maintenance.

Therefore, self-healing of cementitious materials is a resource capable of promoting the crack closure and reestablishing the integrity of the elements, promoted by different approaches, and as main examples are the use of bacteria (WIKTOR, 2016; TIZIVIOGLOU, 2016; GONZÁLEZ, 2020), crystalline admixtures (ARNDT, 2019; RAVITHEJA, 2019), supplementary cementitious materials, and superabsorbent polymers (SNOECK, 2014; SILVA 2018).

The self-healing phenomenon can be classified, according to Van Titelboom and De Belie (2013) as autogenous self-healing or autonomous self-healing. Autogenous healing is considered to be the natural process, in which the crack is filled and/or sealed without the aid of any external action, with only intrinsic components of the concrete. Autonomous healing is defined as the repair phenomenon promoted by mechanisms that are not found in the main raw material of cementitious matrices.

Several techniques are employed to evaluate the self-healing phenomenon development, which involves the assessment of mechanical properties and tightness recovery, and crack closure - both superficial and internal. As for the tests used to evaluate the recovery of mechanical properties, the most used are compression resistance, flexural tensile strength, three-points flexural test, among others. Permeability tests are normally used in the analysis of leakage recovery, such as the migration or accelerated chloride ion penetration tests (LITOROWICZ, 2006; ZIEGLER, 2020; HOLLMANN, 2020), and ultrasonic pulse propagation (DURGA et al., 2019). Also, the superficial closure can be evaluated by the presence of internal and external compounds in the edges of the crack, in which scanning electron microscopy, electron microscopy, and dispersive energy spectroscopy techniques are widely used (SNOECK et al., 2016; CUENCA et al., 2018).

Optical microscopy is often employed to evaluate the formation of compounds superficially on cracks (CAPELLESSO et al., 2019; SULEIMAN, NEHDI, 2018; MAES et al., 2016), however image acquisition, treatment, and analysis vary according to each researcher, which can lead to a disagreement regarding the quantitative analysis of self-healing and the correlation between similar approaches.

Disagreements concerning the quantitative analysis of the self-healing process may occur over time, arising from the images post-processing and the parameters used in this analysis. Talab, Huang, and HaiMing (2016) present processing based on the identification of the crack edges, using the Otsu method for image thresholding and Sobel filter to detect the edges.

However, according to Poobal (2017), the difficulties found in crack detection are mainly due to incorrect illumination of the samples, stains on the surface, irregular shape and size of the cracks, and splintering of concrete inside the aperture, which comes off during the treatment.

In this study, optical microscopy was used to capture crack images, subjecting them to two different image treatment pipelines using the ImageJ software (SCHINDELIN et al., 2012), comparing with standards made in the Adobe Photoshop software.

2. Materials and methods

2.1. Material characteristics

The binder utilized was Portland cement, type V, high initial resistance (CPV) according to NBR 16697 (ABNT, 2018), equivalent to Type III by C150/C150M (ASTM, 2019) and CEM 140R by EN 197-1. The characteristics provided by the manufacturer of this material are a specific area of 4.594 cm²/g, setting time 231 minutes (start) and 292 minutes (end), compressive strength 22.2 MPa (1 day), 40.2 MPa (3 days) and 44.7 MPa (7 days), and 5.1% loss of ignition.

The fine aggregate, a washed sand with a fineness modulus of 2.30, maximum dimension of 4.75 mm, specific mass of 2.49 g/cm³, and a unit mass equivalent to 1.61 g/cm³; respectively following the NBR NM 248 (ABNT, 2003), NBR NM 52 (ABNT, 2009), NBR NM 45 (ABNT, 2006). The coarse aggregate is a basaltic origin, fineness modulus of 6.10, the maximum dimension of 12.50 mm, a specific mass of 3.01 g/cm³, water absorption of 1.20%, and unit mass of 1.51 g/cm³ evaluated, respectively, according to the requirements of NBR 248 (ABNT, 2003), NBR NM 53 (ABNT, 2009) and NBR 45 (ABNT, 2006).

Concretes were produced without (REF) and with crystalline admixture (CA), an agent used in the evaluation of the self-healing phenomenon. CA is a commercial product with an average diameter of 10.72 μm , main chemical compounds indicated in the X-ray fluorescence test: Calcium Oxide (CaO , 68.85%), Silicon dioxide (SiO_2 , 8.94%), Aluminum oxide (Al_2O_3 , 2.67%), and 12.87% loss of ignition; and its content must be equal to 0.8% on the cement mass as indicated by the manufacturer.

2.2 Experimental matrix

The self-healing phenomenon was observed and compared for 2 different concretes. The composition of the mixtures is shown in Table 1, the first being considered a reference free of self-healing agent and the second with the addition of 0.8% of CA. The mix proportion comes from a previous dosage study carried by the NORIE/UFRGS Self-Healing Group. The workability of the mixture in the fresh state was fixed at 200 ± 20 mm slump index, and the superplasticizer additive content was defined according to this reduction.

After casting, the specimens were kept at room temperature for the first 24 hours, covered with a plastic wrap to avoid the exchange of moisture with the environment. Subsequently, they were unmolded and stored in a humid chamber with a temperature of $23 \pm 2^\circ\text{C}$ and humidity greater than or equal to 95%, where they remained until (i) segmentation, cut and crack (at day 3) for drying and wetting cycles (starting after crack), or (ii) compressive strength tests dates, according to NBR 5738 (ABNT, 2015).

Table 1 – Mix proportions

Concrete	Cement	Sand	Coarse Aggregate	Water/Cement	CA content (%)	Slump (mm)
REF	1	1.37	2.18	0.40	0.00	190
CA	1	1.37	2.18	0.40	0.80	195

2.3 Performed assays

Concrete in the hardened state was maintained in (i) wetting and drying cycles - until the date of the microscopy tests - or (ii) wet chamber. Specimens kept in a humid chamber were characterized by compressive strength tests at the ages of 3, 28, and 45 days, following the procedure described in NBR 5739 (ABNT, 2018).

Microscopy samples were obtained by splitting 9.5x19 cm cylindrical specimens into 4 parts, and the two central parts (9.5x5 cm each) were chosen for subsequent steps - due to their greater homogeneity produced by the absence of the wall effect of the forms (Figure 1). The crack opening methodology followed the procedures previously adopted by the group (CAPPELLESSO, 2018; ARNDT, 2019; ZIEGLER, 2020; FRANK, 2020; SILVA, 2021). After the crack opening procedure, loose particles within their interior - that may influence the quantification of the cracked area if loosened between the two analysis moments (3 and 45 days) -, were removed by compressed air puffs, with caution not to damage the matrix. Samples were kept in wetting and drying cycles (2 and 5 days, respectively) until the age of 45 days (6 cycles).

Seven cracked samples were evaluated for each concrete - REF (autogenous self-healing only) and CA (autogenous and autonomous self-healing). The cracks were divided into 10 quadrants of 6mm each, captured at 80x magnification; the central portion was subdivided into four parts of 3 millimeters each, which were captured at 160x magnification (Figure 1). The cracked samples had their images captured using the Zen 3.2 Blue Edition software as the user interface. Images for each sample were captured immediately after cracking (3 days) and at the end of the follow-up (45 days).

The evaluation and monitoring of the crack closure were performed with the aid of an optical microscope, Zeiss Stemi 508, maximum magnification of 250x. Coupled with the microscope, a lighting chamber was used to maintain a constant light exposure condition for all samples.

The parameters used in the software were exposure time of 180ms, and constant color temperature to maintain uniform illumination in the samples. All samples remained in an acclimatized room for three hours

before image acquisition. The purpose of this procedure was to avoid the superficial color variation promoted by the water saturation index of the surface.

The images of each different quadrant of each crack were arranged sequentially with the aid of Adobe Photoshop software. This procedure can join, by recognizing common points, a sequence of images that describes each crack in its entirety. The procedures from obtaining the samples to the sequential joining of the microscopy images (for later analysis by the methods) are shown in Figure 1.

Figure 1 – Sample preparation sequence and microscopy data obtainment (dimensions in mm)

2.4 Image processing

Two image processing methodologies, with partially identical steps, were evaluated. A third procedure, defined as “Standard”, obtained through the manual delimitation of the cracked area in Photoshop, serves as a comparison between methodologies. The methodologies were called "Manual" and "Automated", the first being dependent on an operator for every step, the second - obtained Macro by programming - requires an operator only in the first and final steps.

In Figure 2 there is a sequential disposition of each of the steps adopted for the methodologies. Images were first acquired by microscopy in an RGB model (Initial). The procedures of the “Manual” and “Automatic” methodologies consist of:

- multiply the values of each pixel by themselves;
- transform the RGB image to 8-bit grayscale;
- application of the threshold tool to select pixels of a certain intensity range;
- select all the particles that describe the crack, excluding noise (small circular particles external to the region of interest);
- dilate the pixels to connect neighboring regions;
- filter the filling to cover the white pixels inside the crack area, transforming them into black pixels;
- apply erosion to the image, which eliminates displaced pixels and without connectivity to each other;
- apply the outline tool, which creates an outline to the region of interest;
- manually select the region of interest (crack) in the sample;
- exclude interest region outer points.

Finally, by the relationship between the total number of pixels in the image and black pixels, the cracked area can be obtained. Figure 2 shows the results of each of the steps applied in "Manual" and "Automated". Images were scaled through a known dimension (scale bar inserted at the capture moment with the Zen 3.2 Blue Edition program).

In Figures 3 to 5 samples are identified as to their composition: REF (reference concrete samples without addition of healing agent) and CA (concrete samples with addition of crystalline additive); 0.8 (magnification of 80x of the central part of the crack, composed of two captures) and 1.6 (magnification of 160x of the central part of the crack, composed of four captures).

Figure 2 – Treatment pipeline with example results of each step

2.5 Statistical Methods

Image analysis methods (Automated and Manual) were compared to Standard through equations 1 and 2:

$$Self - healing = 1 - \frac{final}{initial} \quad (1)$$

$$Accuracy = \frac{method - standard}{standard} \quad (2)$$

Figure 4 displays values obtained with equation 1. Values obtained with equations 1 and 2 were analyzed using ANOVA to assess the influence of all parameters. The definition of significance was set a priori to $p < 0.05$. For all models, residuals were checked for normality. All analyses were performed using the Statistica 8 program.

3. Results and discussion

The strength test was performed for concretes in a humid chamber (at ages 3, 28, and 45 days). Table 2 describes the average strength and standard deviation.

Table 2 – Concrete average strength and standard deviation (MPa) at days 3, 28, and 45 days

Concrete	Humid chamber		
	3 days	28 days	45 days
REF	33,56±1,68	39,91±2,22	42,75±2,96
CA	32,76±1,08	38,40±3,21	36,20±3,04

Specimens without crystalline admixtures (REF) showed a better performance when compared to CA samples. At 3 days, all concrete showed great compressive strength, resulting from the high initial strength cement used in the mix proportion design.

Figure 3 – Crack area quantification results at days 3 and 45 for REF and CA samples at 0.8 and 1.6 magnifications

Crack dimensions immediately after their cracking influence the later stages of the process of monitoring the healing process, which is determined mainly by the manufacture, cracking methodology, and composition of the samples. Cracks' diverse geometric characteristics may be responsible for a large dispersion in area quantification results, a factor that may distort the real result of the variables analyzed in the present work. The ideal would be to minimize the variability between samples, obtaining similar profiles for all groups in the cracking assay.

To assess the influence of all analyzed parameters - 2 types of concrete, magnification of images, methodologies - on the crack area, an Analysis of Variance (ANOVA) was performed, where the type of concrete and magnification showed significance (p-values < 0.005).

Figure 3 shows the influence suffered by the crack area, in terms of the methodologies used, the magnification of the images, and the type of concrete used (each column is a mean of seven independent measurements). It is observed that the wetting and drying cycle causes a reduction in area for all analyzed combinations when comparing the ages of 3 and 45 days. This phenomenon may be associated with the carbonation of calcium hydroxide leached from hydration, consequently, with the high precipitation of calcium carbonate close to the edges of the fissures (CHOI et al., 2016). The same behavior for samples containing CA was perceived by Arndt (2019), when evaluating cracked concrete at the same age, maintained in similar exposure environments (wetting and drying cycles) but for different periods.

Figure 4 – Self-healing behavior of REF and CA samples at 0.8 and 1.6 magnifications

Values obtained in each of the groups (standard, manual, automated) present a specific pattern in REF and PRA for both extensions, which becomes clearer when expressed in terms of self-healing (Figure 4). Standard tends to have higher area values and smaller differences between days 3 and 45 (Figure 3), implying a lower self-healing - especially for CA - (Figure 4). Manual and automated, on the other hand, present very close crack area values, but automated detects higher healing values. This behavior of greater healing by automated can be explained by its greater sensitivity to capture narrower stretches of the fissure, discussed in greater depth in the discussion of Figure 5. The results found by Arndt et al. (2019) corroborate with the ones found in this work - higher healing values for REF than CA ($p < 0.0005$) -, that is, there may be a more suitable exposure environment for concretes with CA rather than wetting and drying cycles.

Figure 5 – Comparison between the results obtained by the methodologies for each magnification

For both magnifications, the Automated methodology presents a greater wealth of details when compared to the manual method (Figure 5). As for the 1.6 magnification, there is an increase in the definition of the edge region along the entire crack - resulting from the capture of narrower stretches of the crack -, especially for Standard and Automated, maintaining the same bias found for the 0.8 magnification, with the automated method making closer captures to the Standard.

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4. Conclusions

The wetting and drying cycle favors the occurrence of self-healing compounds, both for reference and for CA concretes.

Magnification influences the capture of the crack area ($p\text{-value} < 0.005$). As image acquisition with a magnification twice greater is made, the total number of captures also doubles, resulting in twice as much work to obtain, store and process the data. One way to get around this drastic increase is to limit the use of greater magnification to the central portion of the crack, as the behavior present in the central portion is comparable to that of the crack as a whole, but it depends on its crack opening width.

Proposed methods cannot be differentiated statistically, yet Automated qualitatively seems to be more promising when compared to the Manual method. Additionally, as it has few operator-dependent steps (only two: initial, where the input files are selected; and final, where the region of interest is selected), processing becomes faster, also diminishing steps that can cause an increase of variability due to the influence of the operator throughout the process.

Acknowledgments

Thanks to the Laboratory of Materials and Technology of the Built Environment (LAMTAC) for providing space and equipment for the development of this research.

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