Cement and Concrete Research 83 (2016) 70–78

Contents lists available at ScienceDirect

Cement and Concrete Research

journal homepage: www.elsevier.com/locate/cemconres

Evaluation of self-healing of internal cracks in biomimetic mortar using coda wave interferometry

Shukui Liu [a], Zeynep Basaran Bundur [b,1], Jinying Zhu [c,*], Raissa Douglas Ferron [b]

[a] School of Mechanics and Civil Engineering, State Key Laboratory for Geomechanics and Deep Underground Engineering, China University of Mining and Technology, Xuzhou 221008, PR China
[b] Department of Civil, Architectural, and Environmental Engineering, The University of Texas at Austin, 301 East Dean Keeton Street, C1748, Austin, TX 78712, USA
[c] Department of Civil Engineering, The University of Nebraska-Lincoln, Omaha, NE 68182, USA

ARTICLE INFO

Article history:
Received 26 April 2015
Accepted 21 January 2016
Available online xxxx

Keywords:
Self-healing
Mortar (E)
Microcracking (B)
Curing (A)
Ultrasonic wave

ABSTRACT

Calcium carbonate biomineralization is a bio-chemical process in which calcium carbonate precipitation is obtained by leveraging the metabolic activity of microorganisms. Studies have shown that biomineralization can be used to repair surface cracks in cement-based materials. One of the challenges in determining whether biomineralization is a feasible option for internal crack repair pertains to how to monitor and quantify self-healing of internal microcracks. In this study, mortar samples with and without microcracks and microorganisms were cured in different environments until 50 days. Coda wave interferometry measurements, a nondestructive method that is very sensitive to small changes in material, were conducted on these samples to evaluate the extent of self-healing during the entire curing period. Compressive strength tests were performed after 7 and 28 days of curing. The results indicated that the cracked mortar samples with microorganisms showed significantly higher strength development and higher relative velocity change than samples without microorganisms.

1. Introduction

Internal stress in concrete might induce microscopic cracks, which then provide pathways for ingress of harmful chemicals and lead to loss of strength of concrete. Biomineralization is a process in which microorganisms stimulate the formation of inorganic minerals deposited in biological systems [1]. The list of biominerals can be extensive but calcium carbonate ($CaCO_3$) and calcium phosphate minerals are thermodynamically stable, thus commonly observed in nature [1]. In particular, $CaCO_3$ obtained via microbial induced calcium carbonate precipitation (MICCP) is promising for a varied range of engineering applications [2,3]. One attractive application of MICCP is remediation of defects and cracks in cement-based materials. Biomineralization has been used as an approach to repair cement-based structures and ornaments for restoration via external application of a bacterial agent [4–6]; however, it is still not clear if the embedded microorganisms can heal the cracks by the means of strength recovery. The width of the cracks might influence the efficacy of the biogenic self-healing process [7]. So far a maximum crack width of 0.97 mm was reported for biogenic self-healing applications on mortar surface [8]. The depth of the cracks may also have an influence on crack healing ability of microorganisms; as such, the majority of the current research on using biomineralization for self-healing applications in cement-based materials has been focused on surface cracks. However, internal microcracks in cement-based materials are also a concern, since these cracks may eventually coalesce and cause detrimental effects to the durability of the material.

The objective of this study is to investigate feasibility of self-healing by microorganisms embedded in cement paste matrix. Self-healing corresponds to the recovery of mechanical properties (e.g. strength) after damage, whereas self-repair will be the general term used if crack closure/crack sealing occurred but it was not explicitly confirmed that the crack closure was accompanied with an increase in mechanical properties. One approach to observe the internal cracks is from taking cross-sectional slices and examining the sample with a microscope [9]; however, this method is destructive. As to nondestructive methods, airflow, water permeability, resonant frequency, acoustic emission and ultrasonic pulse velocity (UPV) tests are usually used. For instance, Gagne and Argouges investigated the natural self-repairing ability of mortars using airflow measurements through a single crack of controlled geometry [10]. Water permeability test was adopted by Reinhardt and Joss to characterize the influence of temperature and crack width on crack repair [11]. Yang et al. [12] performed the resonant frequency test to characterize the self-repairing properties of engineered cementitious composites under wet-dry cycles. Van Tittelboom et al. [13] carried out the acoustic emission analysis for the quantification of autonomous crack repair in concrete. UPV method has been applied to assess the self-repair of cracks caused by freeze–thaw cycles [14,15] and compression loading [16]. Although it has been found that these methods could detect the occurrence of repair to some extent, many of these methods are not sensitive

* Corresponding author. Tel.: +1 4025544459.
E-mail address: jyzhu@unl.edu (J. Zhu).
[1] Current affiliation: Ozyegin University, Department of Civil Engineering, Cekmekoy, 34794, Istanbul, Turkey.

http://dx.doi.org/10.1016/j.cemconres.2016.01.006
0008-8846/

enough to continuously characterize the small changes in materials. For example, in the UPV test, variability due to different sensor coupling conditions may exceed the velocity change level caused by self-healing [17]. During the self-repair process, small changes happen gradually, thus nondestructive methods of higher sensitivity are needed in order to track these small changes.

Coda wave interferometry (CWI) is a recently developed ultrasonic wave analysis technique to determine the relative velocity change of diffused wave fields which are measured from a fixed wave source and a fixed receiver at two different moments [18]. It has been known that coda waves, despite their noisy and chaotic appearance, are highly repeatable [19] and carry rich information about the medium. Due to multiple scattering [20], coda waves travel through a longer path and interact multiple times with the microstructure of the same region within the medium, thus are extremely sensitive to small changes in the medium. The sensitivity of coda waves has been mainly used by seismologists [21] to estimate slight velocity changes in the earth crust due to seismic effects [18], mining influence [22], volcanoes' activity [23] or seasonal variations [24]. It was not until very recently that CWI was applied to detect small changes in soils [25] and concrete materials [26–28].

In this study, the capability of the CWI method for quantifying the degree of self-healing of internal cracks in mortar samples will be assessed. The self-healing ability will be determined via compressive strength tests conducted on the cracked mortar samples and compared with the results from the CWI methods. The results of this study will provide an understanding for self-healing of internal microcracks in cement-based mortar and provide a new approach to monitoring self-healing of internal microcracks via the CWI method.

2. Materials and methods

2.1. Microorganism growth

American Type Culture Collection: *S. pasteurii* (ATCC® 6453™) [29] was grown in Urea-Yeast Extract (UYE) medium, which was prepared according to ATCC 1376 medium instructions [30]. Specifically, UYE medium contained 0.13 M Tris base, 10 g of urea, and 20 g of yeast extract per liter of distilled deionized (DDI) water. When solid media was necessary, 20 g of agar was added to a liter of liquid UYE medium. The pH of the medium was adjusted to 9.0. *S. pasteurii* cells were grown aerobically at 30 °C and 150 rpm shaking conditions in a 600 mL UYE medium until the desired concentration was obtained. Over time, 1-mL sample aliquots were removed for analysis. Absorbance of the aliquots was measured by optical density at 600 nm (OD_{600}) using a BioTek Synergy™ HT plate reader (Winooski, VT, United States). The absorbance was correlated with viable plate counts from the same sample aliquots. Viable plate count is a technique to determine the number of viable and growing cells in a medium and is conducted by plating the liquid sample onto UYE agar medium plates. The UYE agar plates were incubated at 30 °C for 48 h; then colony forming units (CFUs) was counted. Colony forming units is a general term to express the estimation of number of viable cells grown on agar plates [31]. A correlation between OD_{600} and CFU/mL was developed, and OD_{600} was used thereafter for determining the cell concentration prior to mixing.

2.2. Cement and aggregates

Texas Lehigh Type I/II (Buda, TX) Portland cement was used for all mortar mixtures. Table 1 shows the mass percentage distribution for oxides; Type I/II cement has 3.2% free lime content, and 77.3% of the free lime is $CaCO_3$. The particle size distribution (PSD) of the cement was determined by using a laser diffraction particle size analyzer (Mastersizer 2000) equipped with a wet dispersion unit (Malvern, Worcestershire, United Kingdom). Fig. 1 shows PSD of Texas Lehigh Type I/II Portland cement and the mean particle size of the cement (d_{50}) was determined as 23.2 μm [32].

Table 1
Texas Lehigh Type I/II Portland cement oxide composition [33].

Oxides	% (w/w) composition
CaO	65.0
SiO_2	20.5
Al_2O_3	4.5
Fe_2O_3	3.0
MgO	1.6
SO_3	2.6
LOI	2.4
C_3A	7.0
Na_2O equivalent	0.79
Free lime%	3.2

For mortar samples, Colorado River sand was used. The gradation of sand was determined according to ASTM C136-06 [34]. The fineness modulus of Colorado River sand was calculated as 2.37. According to ASTM C128-07 [35], the absorption capacity of the sand and the specific gravity were determined as 0.65%, and 2.62 respectively. Fig. 2 shows the PSD for sand.

2.3. Mixing and initial curing

Texas Lehigh Type I /II (Buda, TX) Portland cement and Colorado River sand were used for all mortars. Two different sets of mortar mixes were prepared: *neat mortar* and *bacterial mortar*. A set of *neat mortar* samples was prepared by mixing distilled water and cement. For the *bacterial mortar* samples, the water content of the mortar was replaced by bacterial culture (see Section 2.1). Here we use a solution-to-cement ratio (s/c), which refers to the aqueous component used to prepare the paste and is considered a general term applicable to all the pastes. Thus, the solution portion for the *neat mortar*, and *bacterial mortar* are distilled water and the bacterial culture, respectively. The s/c for both *neat mortar*, and *bacterial mortar* were 0.50 and is based on a mass basis. It should also be noted that 94.5% (by mass) of the UYE medium is DDI water while the remaining 5.5% is the chemical ingredients. Bundur et al. [32] showed that even though the percentage of the chemical ingredients is low, the hydration kinetics were impacted. The authors showed that the induction period was extended significantly when the water content of the cement paste was replaced with UYE medium. This effect was found to be due to the addition of yeast extract and addition of vegetative *S. pasteurii* cells [32]. For the *bacterial mortar* samples, the vegetative *S. pasteurii* cells were grown in pH-9.0 UYE medium (see Section 2.1) culture and the concentrations of vegetative *S. pasteurii* cells varied in the range of 2–6 x 10^7 CFU/mL.

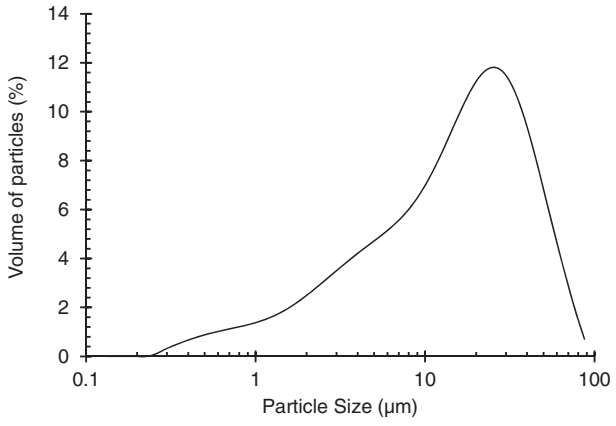

Fig. 1. PSD for Texas Lehigh Type I/II Cement. The x-axis is the particle sizes logarithmically between 0.1 and 100 μm and the y-axis shows the volume of particles between these sizes.