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Abstract. Carbonation causes a physicochemical alteration of cement-based materials, leading to a decrease of porosity and an increase of material hardness and strength. However, carbonation will decrease the pH of the internal pore water solution, which may depassivate the internal reinforcing steel, giving rise to structural durability concerns. Therefore, the proper selection of materials informed by parameters sensitive to the carbonation process is crucial to ensure the durability of concrete structures. The authors investigate the feasibility of using linear and nonlinear dynamic vibration response data to monitor the progression of the carbonation process in cement-based materials. Mortar samples with dimensions of $40 \times 40 \times 160$ mm were subjected to an accelerated carbonation process through a carbonation chamber with 55% relative humidity and >95% of CO_2 atmosphere. The progress of carbonation in the material was monitored using data obtained with the test setup of the standard resonant frequency test (ASTM C215-14), from a pristine state until an almost fully carbonated state. Linear dynamic modulus, quality factor, and a material nonlinear response, evaluated through the upward resonant frequency shift during the signal ring-down, were investigated. The compressive strength and the depth of carbonation were also measured. Carbonation resulted in a modest increase in the dynamic modulus, but a substantive increase in the quality factor (inverse attenuation) and a decrease in the material nonlinearity parameter. The combined measurement of the vibration quality factor and nonlinear parameter shows potential as a sensitive measure of material changes brought about by carbonation. © *2015 Society of Photo-Optical Instrumentation Engineers (SPIE)* [DOI: 10.1117/1.OE.55.1.011004]

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1 Introduction

Carbonation is a diffusion-based process that involves a progressive physicochemical transformation of cement-based materials. Carbon dioxide (CO_2) present in the atmosphere diffuses through concrete and reacts with different constituents of the cementing matrix, which yields mostly calcium carbonate ($CaCO_3$) from its reaction with portlandite $[Ca(OH)_2]$.[1] The carbonation of portlandite takes place in the presence of moisture, so the actual reaction occurs with carbonic acid (HCO_3) as[2]

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O. \qquad (1)$$
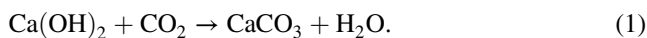
Other cementing matrix constituents react in the presence of CO_2, producing silica gel, alumina, and ferric oxide.[2–8] As a result, the total material porosity decreases, the elastic modulus and strength increase, and the pH level of the pore water solution decreases.[9–13] Decrease of the pore water pH causes the passivation film of any internal embedded steel to become unstable,[14] thus making it susceptible to corrosion. Figure 1 shows an idealized representation, depicting the progressive corrosion of the reinforcement steel and subsequent destruction of the concrete cover.

The proper selection of materials, together with the assessment of cement-based materials in the laboratory, is essential to ensure the durability of structures throughout

their service life.[15] Thus, it is useful to have characterization tools that can monitor the progression of material changes in cement-based materials caused by the carbonation process. Usually, monitoring of the carbonation is based on alteration of the chemical composition of concrete, for example, by the use of a pH indicator,[16] thermogravimetric analysis, or Fourier transform infrared spectroscopy.[17,18] On the other hand, tests based on the variation of the physical properties, such as the decrease of porosity, have been based on the measurement of the permeability, water absorption, and sorptivity characteristics, as well as through the mercury intrusion porosimetry technique.[19–21]

In this study, the modification of the properties of a standard portland cement mortar upon carbonation is monitored using vibration data obtained using the standard resonance frequency test setup.[22] The standard resonant frequency method has been traditionally used to assess the mechanical integrity of cement-based materials upon different durability distresses such as freezing-thawing damage[23] or alkali-silica reaction.[24] However, the standard resonant frequency test has been less broadly used to monitor carbonation. This is probably because of the limited sensitivity of linear-based measurements to the material's physical parameter variations due to carbonation. However, unlike linear-based measurements,[25,26] nonlinear acoustic and wave propagation-based techniques have demonstrated greater sensitivity to detect subtle microstructural modifications within the cement-based

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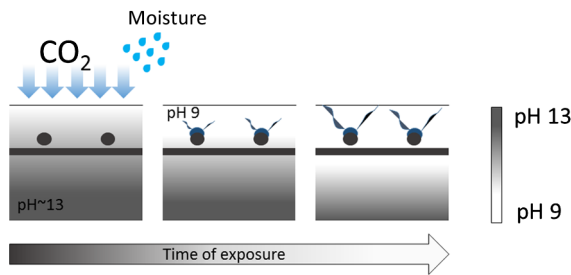
Fig. 1 Schematic depiction illustrating progressive pH reduction owing to carbonation in steel-reinforced concrete. The corrosion of steel bars, which are indicated in black, causes cracks and delamination of the protective layer of concrete.

materials. Because of the complexity of cement-based composites across a range of length scales, the hysteretic nature of the constitutive behavior is enhanced. A nonlinear hysteretic modulus has been proposed as[27]

$$E = E_o\left\{1 + \beta\varepsilon + \delta\varepsilon^2 + \alpha\left[\Delta\varepsilon + \varepsilon \cdot \text{sign}\left(\frac{\partial\varepsilon}{\partial t}\right)\right]\right\}, \qquad (2)$$

where the elastic modulus E_o is expanded to include quadratic and cubic terms of strain β and δ as well as the so-called nonclassical behavior due to dispersive media; ε is the strain, $\Delta\varepsilon$ is the strain amplitude, and α is a parameter that controls the magnitude of the strain rate-dependent hysteresis. Considering Eq. (2), a downward shift of the resonant frequencies associated with and proportional to increasing strain amplitude is predicted.[28] The hysteretic parameter α is related to the downward frequency shift through[28]

$$\frac{\Delta f}{f_o} = \alpha \cdot \Delta\varepsilon, \qquad (3)$$

where f_o is the linear resonant frequency (measured at very low strains) and Δf is the variation of resonant frequency with increasing strain amplitude. This behavior can be investigated using different nonlinear resonant techniques whose basic principle is to excite the materials at their resonance frequencies at different strain amplitudes. For concrete-like materials, previous studies reported that the nonlinear behavior shows enhanced sensitivity to detect alkali-silica reaction,[29] thermal damage,[30,31] and freezing-thawing damage,[32] when compared with the standard values of the dynamic modulus or relative modulus of elasticity. The present paper evaluates the variation of the linear and nonlinear dynamic properties upon carbonation of mortar samples, using resonant vibration data obtained according to the standard ASTM C215-14.[22] Previous research conducted by the authors[33] showed how to extract and quantify the nonlinear behavior due to mechanical hysteresis using a standard test configuration setup (see Sec. 2.2). The approach was demonstrated on freezing-thawing damaged mortar, which enhances the mechanisms of material hysteresis due to the accumulated damage with an increasing number of cycles. Conversely, the authors expect a progressive decrease of material nonlinearity, as initially noncarbonated samples absorb CO_2 within their microstructures. Previous work conducted by Kim et al.[34] investigated the variations of material nonlinearity upon carbonation of concrete samples, through the relative quantification of the quadratic nonlinearity

(or second harmonic generation) of Rayleigh waves. The results reported therein showed a monotonic increase of Rayleigh wave velocity, and also a decrease of material non-linearity (reduction of the second harmonic) as the carbonation of concrete progressed. Bouchaala et al.[35] investigated the effect of carbonation on the linear dynamic modulus and nonlinear hysteretic properties of cylindrical core concrete samples of varying composition, at a dichotomous state: noncarbonated and partially carbonated concretes. Their results demonstrated an increase of dynamic modulus and a decrease of material nonlinearity when noncarbonated and partially carbonated samples were compared. Conversely, this study monitors the evolution of the dynamic modulus and quality factor (inverse attenuation), as well as material nonlinearity through the quantification of the downward frequency shift, upon carbonation of portland cement mortar samples, from a pristine to almost fully carbonated state. The progress of the carbonation was confirmed on additional mortar replicates that are mechanically tested and sprayed with a pH indicator (phenolphthalein) to ascertain the actual level of carbonation. The results confirm an increase of mechanical properties (compressive strength and dynamic modulus) and a reduction of material nonlinearity. Further, the results show that the variations of quality factor, and more so, the variations of material nonlinearity upon carbonation, exhibit greater sensitivity to detect the carbonation process, especially at early periods of carbonation. For longer periods of carbonation (partially carbonated mortar), the material nonlinearity does not significantly evolve, but the quality factor does.

2 Experimental Details

2.1 Materials and Accelerated Carbonation

Five series of three prismatic mortar samples with dimensions of $4 \times 4 \times 16$ cm^3, a water-to-cement ratio of 0.50, and aggregate to cement ratio of 3 by weight, were fabricated according to European standard EN 196-1.[36] The aggregate was of siliceous nature and the cement was type CEM I-52.5R. The samples were cured at 20°C and 95% of relative humidity (RH) for 315 days after casting. The samples were then subjected to an accelerated carbonation process in a carbonation chamber with a >95% CO_2 atmosphere and 55% RH. The RH value of 55% within the desiccator was selected because the rate of carbonation is highest when RH is between 55% and 65%.[37] An aqueous solution of glycerin (75% by weight) helps to provide a 55% RH within the desiccator. However, since the samples were initially at 95% RH, and because the carbonation process involves releasing of water, the RH within the desiccator increased during early periods of carbonation, as the samples naturally reached the hygroscopic equilibrium in the desiccator. Nonetheless, a periodic renovation of the CO_2 atmosphere was conducted every 24 h to help to keep the >95% CO_2 atmosphere and 55% RH.

2.2 Inspection Techniques

Three samples are tested using the standard resonant frequency method,[22] configured to promote the first bending mode. Figure 2 shows a schematic depiction of the test configuration. The impact excitation is sensed with an accelerometer PCB model 352A21 (PCB Piezotronics,
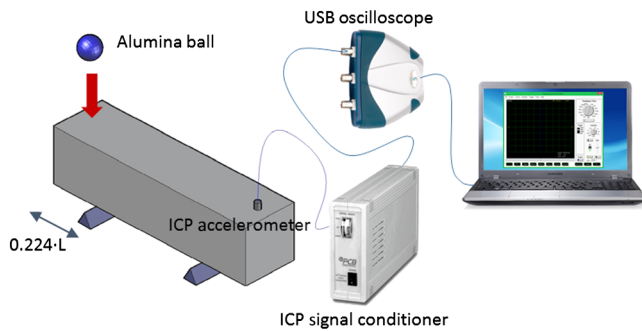
Fig. 2 Schematic depiction of the experimental setup.

Inc., New York) with a sensitivity of 0.956 mV/(m · s^{-2}). The samples are tested before starting the carbonation process, and as the carbonation progresses for 7, 28, 56, 79, and 94 days. Ten signals are acquired at every period of carbonation and transformed to the frequency domain using the fast Fourier transform algorithm, then the linear parameters such as fundamental flexural frequency and quality factor (Q) are derived. The material nonlinearity was investigated using a short-time Fourier transform-based approach, to track frequency changes as the signal amplitude decays naturally, following the procedure proposed in Ref. 33. A sliding window containing 10 cycles moves through a single-time response signal and transforms the time segment within the window to the frequency domain at every window position. The moving window analysis was stopped when the spectral amplitude was reduced below a preset threshold value, here set to 100 arbitrary units (au). Such a threshold value allows to analyze herein all the ring-down signals and extract the upward resonance frequency shift with decreasing signal amplitude. Moreover, the mechanical properties are determined according to the standard EN 196-1[36] on the additional replicates after 0, 7, 28, 79, and 94 days of carbonation; three samples per test result in an average of six values for the compressive strength.[36] A pH indicator (phenolphthalein in ethanol

solution) is used to distinguish between carbonated (pH < 10) and noncarbonated areas on the fractured surfaces.[16]

3 Results and Discussion

Figure 3 shows the representative fractured surfaces, after three-point bending loading, sprayed with phenolphthalein, and their respective binary images used to quantify the front of carbonation. The carbonation depth is measured after 7, 28, 79, and 94 days. This complementary test allows comparison of the actual progress of carbonation to the observed variation of the dynamic properties upon carbonation.

Figure 4 shows the mean results of compressive strength variation with carbonation duration. The results confirmed the increase of mechanical properties observed in portland cement-based materials. The compressive strength at pristine state (53.5 MPa) almost doubles after 94 days of carbonation (92.6 MPa). The mechanisms of mechanical strength increase due to carbonation of portland cement-based materials, described in the literature,[4] include the progressive deposition of calcium carbonates within the pore structures that reduces the capillary porosity[10]—and thus the mechanical strength increases[39]—and additional hydration, since carbonation reaction releases water molecules, mainly from the carbonation of $Ca(OH)_2$ [Eq. (1)], which can combine with yet unhydrated Portland cement particles.

Figures 5(a) and 5(b) show the representative frequency spectra of a mortar sample at noncarbonated state and after carbonation for 28, 56, 79, and 94 days. The first 28 days of carbonation resulted in a moderate reduction of the resonance frequency [Fig. 5(a)]. The initial reduction of the resonance frequency is most likely due to the reduction of moisture content in the sample, since the carbonation depth barely progressed [see Fig. 3(b)]. Further carbonation (time of exposure above 28 days) resulted in a monotonic increase of both resonance frequency and Q. From 28 to 94 days of carbonation, an increase of the material stiffness and a greater decrease of damping properties are observed. Figures 5(c) and 5(d) show the mean value of flexural frequency and Q for all samples. At early stages of carbonation
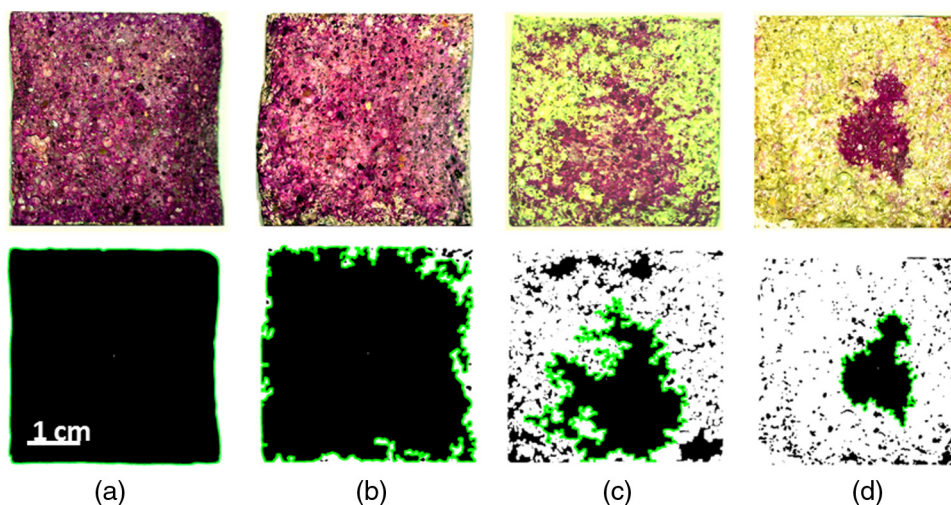

Fig. 3 Images of test samples exposed to different extents of carbonation exposure. Top row—fractured surfaces (4 × 4 cm^2) sprayed with phenolphtalein solution indicator. Bottom row—binary representation of images with noncarbonated areas indicated by black color: (a) 7 days, 0.00% carbonated; (b) 28 days, 10.13% carbonated; (c) 79 days, 72.83% carbonated; and (d) 94 days of carbonation, 89.36% carbonated. (Adapted from Eiras et al.[38])
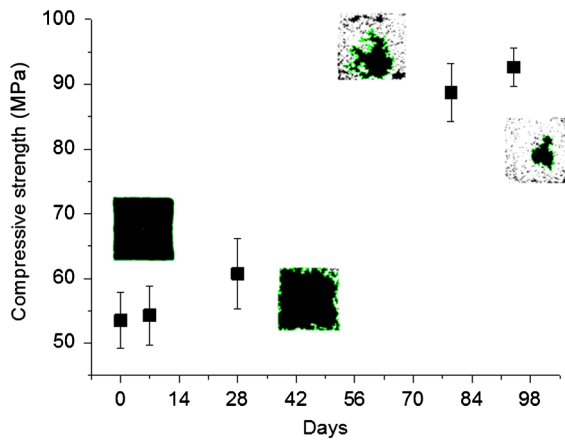
Fig. 4 Evolution of compressive strength with carbonation exposure duration; error bars represent standard deviation. (Adapted from Eiras et al.[38])

(from 0 to 28 days), it is observed that the resonance frequency decreases. The samples, initially at 95% RH, dry naturally inside the desiccator to 55% RH, thus reducing the internal moisture content of the samples. The carbonation rate during that period is very slow since CO_2 cannot diffuse into the samples when the pore structure is clogged with water. Previous research conducted by the authors[40] demonstrated that the presence of moisture in mortar samples gives rise to a stiffening effect on the pore structure, which increases the resonance frequency and viscous damping of the material (decrease of Q), whereas subsequent water removal caused a decrease of the resonance frequency and an increase of Q. Therefore, the initial variations of the resonance frequency and Q are most likely due to the variation of the moisture content and moisture distribution within the sample, rather than carbonation of the samples.

Usually, to quantify the shift of resonant frequency [Eq. (3)], the downward frequency shift as a function of dynamic strain is normalized by f_o. However, small errors in the selection of f_o would lead to great errors in the estimation of the parameter α in Eq. (3).[41] A related discussion on error in estimation of α due to incorrect or inaccurate selection of f_o can be found in Ref. 41. Alternatively, changes in resonant frequency with varying spectral amplitude can be evaluated through a least-squares minimization of the model such that no previous selection of f_o is required:

$$f = f_{\text{int}} + A \cdot \eta, \tag{4}$$

where f_{int} is the intercept and η is the slope of the relation between frequency (f) and spectral amplitude (A). In this
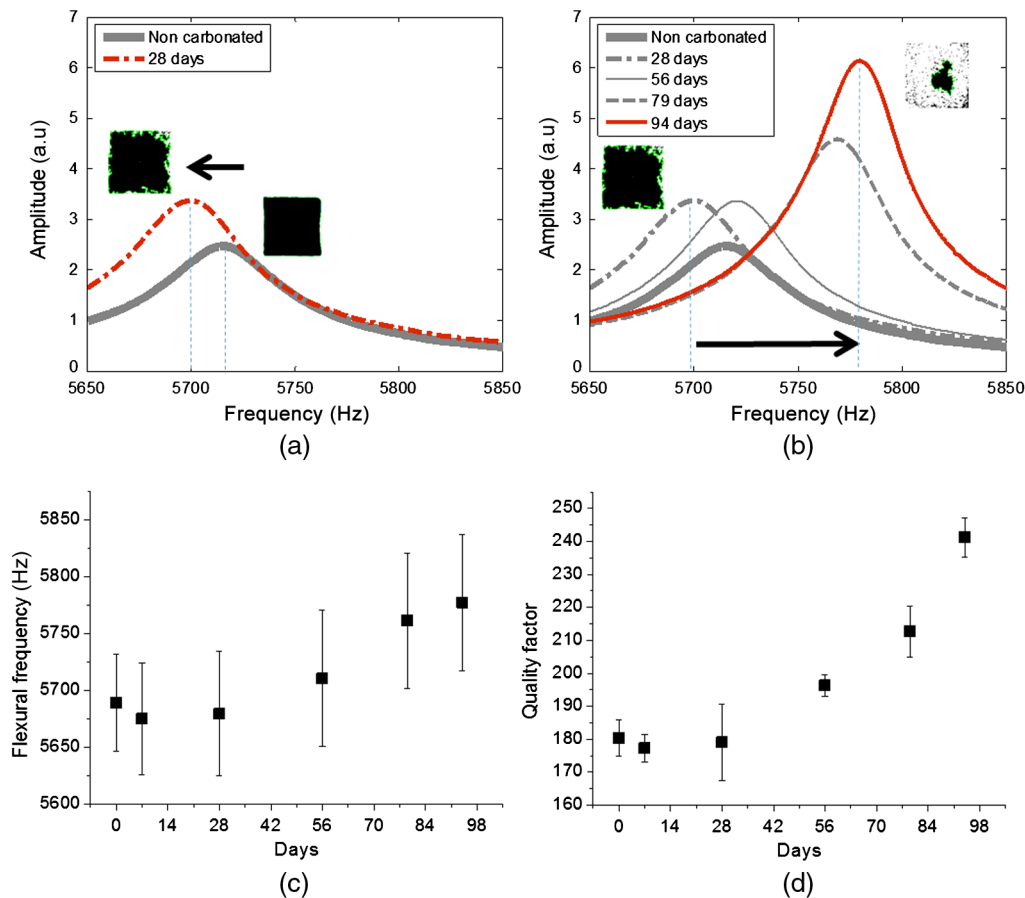

Fig. 5 Linear vibration resonance responses of the fundamental flexural mode obtained from varying degrees of carbonation exposure; inset images indicate noncarbonated areas as black on a $4 \times 4\ cm^2$ cross-sectional area: (a) representative resonance spectra of noncarbonated and after 28 days of carbonation; (b) resonance spectra for noncarbonated and after 28, 56, 79, and 94 days of carbonation; (c) mean values of the resonance frequency; and (d) mean values of Q. Error bars represent standard deviation. (Adapted from Eiras et al.[38])
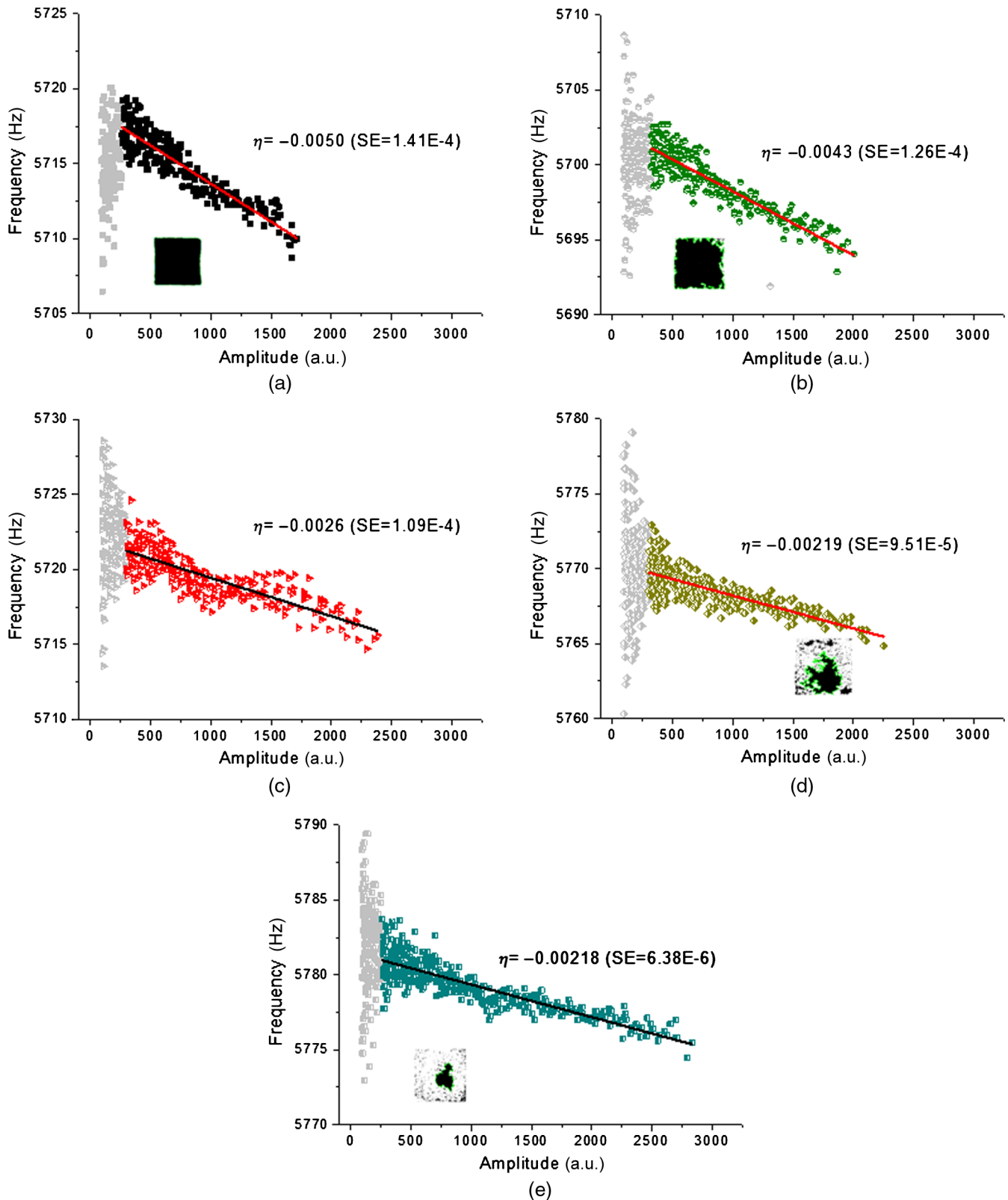
Fig. 6 Frequency shift observed on ring-down versus spectral amplitude for (a) pristine sample—0 day, $\eta = -0.0050$ ($\pm 1.41 \times 10^{-4}$) (standard error on the estimation of η is shown in parenthesis); (b) 28 days, $\eta = -0.0043$ ($\pm 1.26 \times 10^{-4}$); (c) 56 days, $\eta = -0.0026$ ($\pm 1.09 \times 10^{-4}$); (d) 79 days, $\eta = -0.00219$ ($\pm 9.51 \times 10^{-5}$); and (e) 94 days, $\eta = -0.00218$ ($\pm 6.38 \times 10^{-6}$). Gray-colored symbols represent discarded data that have insufficient signal-to-noise ratio to provide reliable results.

representation, η represents a dimensionless, relative material nonlinearity parameter that is appropriate for comparison of samples collected from a single test setup and signal processing approach. Figures 6(a) through 6(e) illustrate the measurement of η, where the relation between the upward frequency shift with decreasing spectral amplitude on ring-down for 10 vibrational responses is shown; the estimated model parameters [Eq. (4)] are also displayed across a range of carbonation durations. During the first 28 days of carbonation, no noticeable variation of the hysteretic behavior is observed. The nonlinear parameter varied from $\eta = -0.0050$ au to -0.0043 au. However, for longer carbonation periods, 56 to 94 days, the material nonlinearity decreased when the parameter η increased to -0.002. From the measurement of the material nonlinearity upon carbonation, one can conclude that the carbonation results in the reduction of material nonlinearity and the samples reach an almost constant value of η, even if the sample is not fully carbonated, so further carbonation does not modify the material nonlinearity.

Figure 7 summarizes the linear and nonlinear dynamic phenomena observed for progressive carbonation. At early stages of carbonation (from 0 to 28 days), it is observed that the resonance frequency moderately decreases, and Q and the material nonlinearity remain almost same. The samples, initially at 95% RH, dry naturally inside the desiccator at 55% RH, reaching progressively the hydric equilibrium. Variations of the dynamic properties at early stages of carbonation are most likely due to variations of internal moisture content in the material. The material nonlinearity was not sensitive to such moisture reduction in the samples during the first 28 days of carbonation. The values of η remained almost constant during this period. This implies that the features that caused the material nonlinearity are not significantly affected by the moisture loss and slight carbonation during the first 28 days. Once the internal moisture within the samples equilibrated at 55% RH, the carbonation process progressed at a higher rate. During advanced stages of carbonation, the resonance frequency and Q increased and the material nonlinearity decreased. During these stages, the modification of the dynamic properties is most likely due to progressive carbon dioxide absorption and formation of calcium carbonate within the pore structure that increases the mechanical stiffness, decreases its compressibility, and also decreases the nonlinearity of the material. No significant
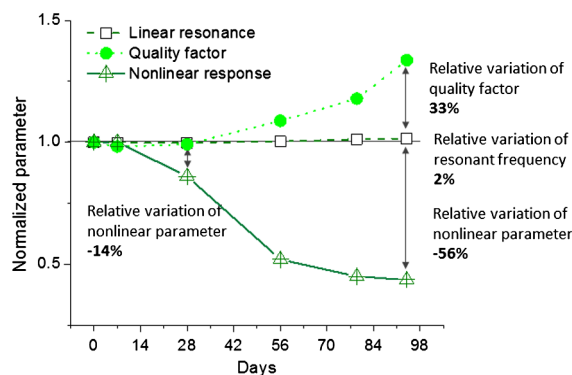
differences in material nonlinearity are observed, however, for the carbonation durations between 56 and 94 days. Further carbonation, after 56 days, did not cause significant variations of material nonlinearity, but a decrease of material damping (increase of Q) and still, a slight increase of the material stiffness (increase of resonant frequency).

4 Conclusions

Microstructural changes caused by carbonation in mortar were assessed by linear and nonlinear vibrational response data obtained using the standard resonance frequency test configuration. The three dynamic parameters investigated here exhibited different evolutions with, and sensitivities to, the material changes brought about by carbonation. Both Q and η showed substantive changes in value with increasing extents of carbonation, although η showed significant changes early in the carbonation process (first 56 days) while Q showed significant changes later on in the process. The resonant frequency showed only a modest change in value with carbonation. The nondestructive test used here is suitable for laboratory monitoring of test samples because of its ease of use and implementation; in particular, Q and η together serve as sensitive measures of material modifications brought about by carbonation across all stages of the process. Thus, the suitability of the material against carbonation can be deemed using the linear and nonlinear dynamic properties in laboratory tests. Further, these findings may also have relevance, more indirectly, for *in situ* tests with regard to the nature of linear and nonlinear dynamic test data as carbonation progresses. In this sense, the sole observation of the dynamic properties would inform about the mechanical integrity of the samples, since under natural weathering conditions, different degradation mechanisms take place. Therefore, since the variation of the physical properties of the samples is not specifically related to a carbonation process, additional complementary tests would be needed.

References

1. A. Hidalgo et al., "Microstructural changes induced in Portland cement-based materials due to natural and supercritical carbonation," *J. Mater. Sci.* **43**(9), 3101–3111 (2008).
2. V. G. Papadakis, C. G. Vayenas, and M. N. Fardis, "Fundamental modeling and experimental investigation of concrete carbonation," *ACI Mater. J.* **88**(4), 363–373 (1991).
3. Z. Sauman, "Carbonization of porous concrete and its main binding components," *Cem. Concr. Res.* **1**(6), 645–662 (1971).
4. A. M. Neville, "Elasticity, shrinkage and creep," in *Properties of Concrete*, 3rd ed., pp. 359–432, Longman Scientific and Technical, Harlow Essex, England (1981).
5. K. Kobayashi, K. Suzuki, and Y. Uno, "Carbonation of concrete structures and decomposition of C-S-H," *Cem. Concr. Res.* **24**(1), 55–61 (1994).
6. L. Fernández-Carrasco et al., "Carbonation of calcium aluminate cement pastes," *Mater. Constr.* **51**(263-264), 127–136 (2001).
7. E. Zornoza et al., "Accelerated carbonation of cement pastes partially substituted with fluid catalytic cracking residue (FC3R)," *Cem. Concr. Compos.* **31**(2), 134–138 (2009).
8. A. Morandeau, M. Thiéry, and P. Dangla, "Investigation of the carbonation mechanism of CH and C-S-H in terms of kinetics,

Fig. 7 Comparison of the evolution of the linear and nonlinear dynamic parameters upon progressive carbonation.

microstructure changes and moisture properties," *Cem. Concr. Res.* **56**, 153–170 (2014).

9. S. E. Pihlajavaara, "Some results of the effect of carbonation on the porosity and pore size distribution of cement paste," *Mater. Struct.* **1**(6), 521–526 (1968).

10. V. T. Ngala and C. L. Page, "Effects of carbonation on pore structure and diffusional properties of hydrated cement pastes," *Cem. Concr. Res.* **27**(7), 995–1007 (1997).

11. B. Johannesson and P. Utgenannt, "Microstructural changes caused by carbonation of cement mortar," *Cem. Concr. Res.* **31**(6), 925–931 (2001).

12. C.-F. Chang and J.-W. Chen, "Strength and elastic modulus of carbonated concrete," *ACI Mater. J.* **102**(5), 315–321 (2005).

13. S. T. Pham, "Effects of carbonation on the microporosity and macro properties of Portland cement mortar CEM I," *J. Mater. Sci. Chem. Eng.* **2**(7), 40–52 (2014).

14. M. Pourbaix, *Atlas of Electrochemical Equilibria in Aqueous Solutions*, Pergamon Press, Oxford (1966).

15. J. Bickley and R. Hooton, "Design for durability: the key to improving concrete sustainability," *Constr. Build. Mater.* **67**(Part C), 422–430 (2014).

16. RILEM Recommendations, CPC-18, "Measurement of hardened concrete carbonation depth," *Mater. Struct.* **21**(6), 453–455 (1988).

17. C.-F. Chang and J.-W. Chen, "The experimental investigation of concrete carbonation depth," *Cem. Concr. Res.* **36**(9), 1760–1767 (2006).

18. G. Villain, M. Thiery, and G. Platret, "Measurement methods of carbonation profiles in concrete: thermogravimetry, chemical analysis and gammadensimetry," *Cem. Concr. Res.* **37**(8), 1182–1192 (2007).

19. E. Rozière, A. Loukili, and F. Cussigh, "A performance based approach for durability of concrete exposed to carbonation," *Constr. Build. Mater.* **23**(1), 190–199 (2009).

20. R. Neves et al., "Assessing concrete carbonation resistance through air permeability measurements," *Constr. Build. Mater.* **82**, 304–309 (2015).

21. J. J. Chang et al., "Suitability of several current used concrete durability indices on evaluating the corrosion hazard for carbonated concrete," *Mater. Chem. Phys.* **84**(1), 71–78.

22. ASTM C215-14, *Standard Test Method for Fundamental Transverse, Longitudinal, and Torsional Resonant Frequencies of Concrete Specimens*, ASTM, West Conshohocken, PA (2014).

23. M. Ohtsu, "Damage evaluation in freezing and thawing test of concrete by elastic-wave methods," *Mater. Struct.* **44**(10), 1725–1734 (2011).

24. R. N. Swamy and M. M. Al-Asali, "Engineering properties of concrete affected by alkali silica reaction," *ACI Mater. J.* **85**(5), 367–374 (1988).

25. V. M. Malhotra and V. Sivasundaram, "Resonant frequency methods," in *Handbook on Nondestructive Testing of Concrete*, V. M. Malhotra and N. J. Carino, CRC Press, Boca Raton, Florida (2004).

26. T. R. Naik, V. M. Malhorta, and J. S. Popovics, "The ultrasonic pulse velocity method," in *Handbook on Nondestructive Testing of Concrete*, V. M. Malhorta and N. J. Carino, CRC Press, Boca Raton, FL (2004).

27. R. A. Guyer and P. A. Johnson, "Nonlinear mesoscopic elasticity: evidence for a new class of materials," *Phys. Today* **52**(4), 30–36 (1999).

28. K. Van Den Abeele et al., "Nonlinear elastic wave spectroscopy (NEWS) techniques to discern material damage. Part II: single mode resonance acoustic spectroscopy," *Res. Nondestr. Eval.* **12**(1), 31–42 (2000).

29. K. J. Leśnicki et al., "Accelerated determination of ASR susceptibility during concrete prism testing through nonlinear resonance acoustic spectroscopy," U.S. Department of Administration, Federal Highway Administration, Publication No. FHWA-HRT-13-085, p. 76 (2013).

30. S.-J. Park et al., "Evaluation of residual tensile strength of fire-damaged concrete using a non-linear resonance vibration method," *Mag. Concr. Res.* **67**(5), 235–246 (2015).

31. C. Payan et al., "Applying nonlinear ultrasound spectroscopy to improving thermal damage assessment in concrete," *J. Acoust. Soc. Am.* **121**(4), EL125–EL130 (2007).

32. J. N. Eiras et al., "Evaluation of frost damage in cement-based materials by a nonlinear elastic wave technique," *Proc. SPIE* **9064**, 90641G (2014).

33. J. N. Eiras et al., "Non-classical nonlinear feature extraction from standard resonance vibration data for damage detection," *J. Acoust. Soc. Am.* **135**, EL82–EL87 (2014).

34. G. Kim et al., "Quantitative evaluation of carbonation in concrete using nonlinear ultrasound," *Mater. Struct.* (2014).

35. F. Bouchaala et al., "Carbonation assessment in concrete by nonlinear ultrasound," *Cem. Concr. Res.* **41**, 557–559 (2011).

36. EN 196-1, "Methods of testing cement—part 1: determination of strength" (2005).

37. D. Russell et al., "Effect of relative humidity and air permeability on prediction of the rate of carbonation of concrete," *Proc. ICE—Struct. Build.* **146**(3), 319–326, (2001).

38. J. N. Eiras et al., "Monitoring accelerated carbonation on standard portland cement mortar by nonlinear resonance acoustic test," *Proc. SPIE* **9438**, 94380D (2015).

39. X. Chen, S. Wu, and J. Zhou, "Influence of porosity on compressive and tensile strength of cement mortar," *Constr. Build. Mater.* **40**, 869–874 (2013).

40. J. N. Eiras et al., "The effects of moisture and micro-structural modifications in drying mortars on vibration-based NDT methods," *Constr. Build. Mater.* **94**, 565–571 (2015).

41. P. A. Johnson, "Dynamic measurements of the nonlinear elastic parameter α in rock under varying conditions," *J. Geophys. Res.* **109**(B2), B02202 (2004).

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