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INTRODUCTION

Concrete structures are often constructed for industrial purposes, and may be exposed to aggressive environments (for example, wastewater). The chain of chemical reactions triggered by biogenic bacteria is the main source of concrete deterioration in wastewater infrastructure. As an attribute of deterioration, sulfuric acid may exist in wastewater, treatment plants, and sewer systems[1-3]. Existing structures subjected to sulfuric acid are rapidly degraded and, as such, their service life is shortened. The chemical reaction between the concrete and sulfuric acid may be represented by:

$$Ca(OH)_2 + H_2SO_4 \rightarrow CaSO_4 + 2H_2O \tag{1}$$

It is a dissolution reaction accompanying gypsum ($CaSO_4 \cdot 2H_2O$), and ettringite is further generated[4]. Over \$25 billion is spent annually for the operation and maintenance of wastewater infrastructure in the United States[5]. A budget of \$390 billion may be required to repair damaged structures over the next 20 years[6]. Accordingly, the research community has been studying the effect of sulfuric acid on the performance of various structural members. Durning and Hicks[7] discussed the deterioration mechanism of concrete caused by chemical exposure (that is, sulfuric acid, acetic acid, formic acid, and phosphoric acid). Test results revealed that the use of a calcium silicate hydrate paste with microsilica formulated durable concrete by increasing resistance to those detrimental chemicals; specifically, the polymerization of the paste bound reactive ions. Bassuoni and Nehdi[8] examined the behavior of self-consolidating concrete exposed to sulfuric acid. The concrete specimens had three categories: non-air entrained, air-entrained, and fiber-reinforced. A change in concrete-mass with exposure time was reported along with some results attained from chemical investigations, employing differential scanning calorimetry and X-ray powder diffraction. The inclusion of steel and polypropylene fibers improved the integrity of the concrete. A direct relationship between the mass-loss and strength-decrease of the concrete was not found. Monteny et al.[9] examined the resistance of concrete to sulfuric acid: concrete cylinders were mixed with various admixtures, namely silica fume, blast furnace cement, polymer, and superplasticizer, in addition to conventional concrete components. The dimensional stability of the specimens was assessed using a laser sensor. The concrete mixed with blast-furnace cement exhibited the best performance, followed by the one with polymers. The concrete with silica fume was least recommendable because of the vulnerability to sulfuric damage. It may be of interest to note that the concentration of sulfuric acid varies from 1% to 5% for accelerated durability testing[10].

Although various technical methods have been proposed to enhance the sustainability of concrete structures in aggressive service environments, including the addition of supplementary cementitious materials, effective solutions are still required, since calcium hydroxide inside concrete fundamentally reacts with surrounding environments (for example, sulfuric acid). Conventional retrofit approaches such as concrete jacketing may postpone the deterioration of the structural system, whereas a similar problem could reoccur owing to the aforementioned chemical reaction mechanism. Despite some new binders being considered to address durability requirements for concrete members, there appears to be no broad consensus about extending the longevity of concrete exposed to sulfuric acid. The state-of-the-art of concrete exposed to sulfuric acid illustrates that the majority of research focuses on the design method of concrete mixtures to achieve durable performance. Little information is available on enhancing the behavior of existing concrete members deteriorated by sulfuric acid. This paper discusses a retrofit method using carbon fiber reinforced polymer (CFRP) composite sheets, which can extend the service life of concrete members exposed to sulfuric acid. Chemical and mechanical tests are conducted to characterize a difference between the concrete with and without CFRP-strengthening when deterioration occurs.

RESEARCH SIGNIFICANCE

Provided that structural concrete constructed in industrial regions is vulnerable to sulfate-related damage and many of them are physically deteriorated, the need for rehabilitation arises. It is a particularly crucial subject for concrete columns whose failure can lead to the collapse of the entire structural system. Structural strengthening with CFRP composites has been implemented since the 1990s to upgrade the performance of existing axial load-bearing concrete members. Numerous experimental and theoretical studies were reported from fundamental mechanics to accelerated durability testing. Nonetheless, extremely limited technical results are available when CFRP-strengthened structural members are subjected to potential damage associated with sulfuric acid. The present experimental research evaluates the effectiveness of composite-strengthening for load-bearing concrete situated in a sulfuric acid environment.

EXPERIMENTAL PROCEDURE

Materials and specimens

Unidirectional CFRP sheet was used with a two-part epoxy adhesive. Listed in Table 1 are the nominal properties of the CFRP and epoxy obtained from the manufacturers. Coupons were made to examine the effect of sulfuric acid at the material level: the CFRP coupon [Fig. 1(a)] was 15 mm [0.59 in] wide by 150 mm [5.9 in] long by 0.165 mm [0.006 in] thick, while the epoxy coupon [Fig. 1(b)] was 12.6 mm [0.50 in] wide by 100 mm [3.94 in] long by 6.5 mm [0.26 in] thick (dimensions were outside the gripping regions). Twelve concrete cylinders were cast with dimensions of 100 mm [4 in] in diameter by 200 mm [8 in] in height, based on a specified 28-day compressive strength of 20 MPa [2,900 psi] including 500 g [1.1 lb] of cement (Type III), 250 g [0.55 lb] of water, 811 g [1.79 lb] of fine aggregate, and 1,700 g [3.75 lb] of coarse aggregate per cylinder. All cylinders were cured in a water bath for 28 days at temperatures varying from 20ºC [68ºF] to 22ºC [72ºF]. The cylinders were then dried and the epoxy was applied circumferentially to wrap with the CFRP. Two groups of concrete specimens were considered [Fig. 1(c)]: plain concrete cylinders (Group 1) and concrete cylinders wrapped with one ply of CFRP sheet (Group 2). The top and bottom surfaces of the CFRP-strengthened concrete were sealed by the epoxy (t = 5 mm [0.2 in]) to avoid sulfuric damage (to be discussed).

Sulfuric acid environment

The high concentration of sulfate and dissolved sulfide are dominant attributes, leading to the deterioration of concrete in typical industrial regions or in a sewer system. It is a common practice in laboratory research that sulfuric acid is employed to simulate these service environments. The extent of an acid concentration is an important factor, because it will directly influence the deterioration rate of the concrete. A 5% concentration solution was exploited, which would reasonably deteriorate the prepared concrete specimens within a scheduled time period of 6 weeks representing an aggressive environment. Figure 2 shows two containers storing the specimens in a fume hood. Container 1 (without CFRP) included six plain concrete cylinders and Container 2 (with CFRP) encompassed six CFRP-strengthened cylinders. A digital pH meter was used to measure the variation of pH values from 0 to 6 weeks, as shown in Fig. 2. To measure the deterioration of material properties, six CFRP and epoxy coupons were immersed in the solution. Once the 6-week exposure period was completed, all specimens were rinsed and dried at room temperature for mechanical-testing and mass-measuring.

Chemical testing

Fourier transform infrared spectroscopy (FTIR) was conducted with the aforementioned specimens to chemically characterize the effect of exposure to sulfuric acid, based on absorbance responses and wavenumbers varying from 550 cm^{-1} [217 in^{-1}] to 4,000 cm^{-1} [1,575 in^{-1}]. Further details about the principle and data interpretation are available elsewhere[11]. The potential decomposition of the constituent materials owing to sulfuric acid was evaluated by thermogravimetric analysis (TGA), which measures a change in mass with temperature. The TGA equipment comprises four parts: temperature control and measurement, mass flow control, a purge gas system, and a computer post-processing program. Elevated temperatures were applied at a heating rate of 20°C/min [68°F/min] until a target temperature of 1,000°C [1,832°F] was reached.

Mechanical testing and instrumentation

Figure 3 shows a setup for mechanical testing using 90 kN [20 kip] and 900 kN [200 kip] capacity universal testing machines for the epoxy and CFRP coupons and the concrete cylinders, respectively. A laser extensometer was employed to measure the displacement (or strain) of each specimen marked by two reflective stripes [Fig. 3(a)], including a typical gage length of 1/3 of the specimen length. After completing the planned exposure period, three CFRP and epoxy coupons per category (that is, 0 and 6 weeks) were monotonically tensioned up to failure at a rate of 2.5 mm/min [0.1 in/min], as shown in Figs. 3(b) and (c). The conditioned and unconditioned concrete specimens were also loaded to failure [Fig. 3(d)] at a rate of 5 mm/min [0.2 in/min].

TEST RESULTS

Deterioration of concrete

Shown in Fig. 4 are the pH values measured from Containers 1 (without CFRP) and 2 (with CFRP). When the test program began (0 week), the pH values of the two containers were identical. The values, however, diverged at 6 weeks: an increase of 197% and 64% was noticed for the plain and CFRP-strengthened cases, respectively. This observation denotes that the sulfuric acid directly interacted with the plain concrete (alkaline). By contrast, the presence of CFRP-wrapping precluded the interaction, so that the core concrete was protected. Figure 5 compares the concrete cylinders at 0- and 6-week exposure. The plain concrete specimens revealed significant surface-degradation [Fig. 5(a)]; on the other hand, the strengthened ones showed marginal deterioration of the epoxy surrounding the concrete with a change in color [Fig. 5(b)]. The variation of mass associated with the exposure time is provided in Fig. 6: a decrease of 7% and an increase of 1% before and after the exposure for the plain and strengthened cylinders, respectively. The increased mass is attributed to the fact that the CFRP and concrete absorbed the acid solution.

Microscopic imaging

Figure 7 illustrates the microscopic images of the concrete surface at 0- and 6-week exposure periods. A digital microscope was used to magnify the image by 20 times. The surface of the 0-week-exposure concrete was generally smooth with minor pits [Fig. 7(a)], whereas the surface of the 6-week counterpart was remarkably degraded and aggregates were exposed due to the disintegration of the cement paste [Fig. 7(b)]. There was no microscale damage such as cracking between the cement binder and the aggregates. Shown in Fig. 8 is the magnified surface of the epoxy coupons with and without the exposure. An apparent change in color was observed from dark blue to light blue for those at 0- and 6-week exposure (Figs. 8(a) and (b), respectively). It is postulated that the oxygen component of the epoxy (typical epoxies comprise a three-member ring structure that includes oxygen and carbon atoms) interacted with the sulfuric acid, and a chemical chain reaction took place (that is, ring-opening reaction: i) oxygen atoms in the epoxy are intermingled with hydrogen atoms in sulfuric acid; ii) bond between the oxygen and H_3C breaks; iii) the H_3C attracts oxygen and hydrogen atoms in the acid and forms CH_3OH; and iv) a product consisting of H_3C, OH, and H is established). This process further explains the foregoing change in pH values of the strengthened concrete specimens exposed to sulfuric acid: the hydrogen atoms are a parameter influencing the chemical reaction associated with the pH scale. A similar observation was made for the CFRP where carbon fibers were bound by the epoxy resin (Fig. 9). Although the epoxy itself reacted with sulfuric acid, CFRP served as a protective layer that prevented sulfate-induced damage. The white spots visible in Fig. 9(b) seems to be some residual material leaked from the concrete, while further chemical investigations are necessary to identify the formation and nature of the spots.

Chemical analysis

The results of the Fourier transform infrared spectroscopy are provided in Fig. 10, including comparative plots between the CFRP and epoxy specimens exposed for 0 and 6 weeks. Several peak absorbance regions were identified and corresponding chemical properties are listed in Table 2. The CFRP and epoxy specimens experienced analogous chemical responses, in that the selected five functional groups (Table 2) demonstrated an ascending trend with an exposure time of 6 weeks; however, their absorbance-change rates were different. The C-H stretch of alkanes in the epoxy increased by 47% at 6 weeks, which was higher than 26% of the CFRP. Similarly, the H-C=O:C-H stretch of aldehydes increased by 22% and 13% for the epoxy and CFRP, respectively; the -C=C- stretch of alkenes was augmented by 20% (epoxy) and 13% (CFRP); the C-N stretch of aliphatic amines arose by 44% (epoxy) and 25% (CFRP); and the -C≡C-H:C-H bend of alkyne increased by 33% (epoxy) and 15% (CFRP). Because the CFRP included the epoxy as a binder, all these chemical reactions occurred with reduced magnitudes owing to the contribution of the carbon fibers. In other words, the fibers tended to impede the chemical reactions between the composite (carbon fibers and epoxy) and sulfuric acid. Figure 11(a) reveals the thermogravimetric analysis of the CFRP composite and the cores of the concrete cylinders with and without CFRP-wrapping. According to the observation shown in Fig. 11(b), it is apparent to report that the epoxy component of the composite began to evaporate at around 200ºC [392ºF] and completely disappeared at 420ºC [788ºF] when the mass responses became plateaued, irrespective of elevated temperature, which was also confirmed by a visual examination of the tested composite (residual char). The specimen exposed to 6-week conditioning showed more mass losses in comparison with its 0-week counterpart [Fig. 11(b)], possibly because the fibers were partially interacted with the solution and the composite's thermal stability was influenced. As shown in Figs. 11(c) and (d), the effect of CFRP-wrapping was significant. The mass change of the concrete cores without acid exposure (0 week) was preserved up to 1,000ºC [1,832ºF] with a marginal decrease of 4.6%. This loss is attributed to several factors, such as the evaporation of moisture, dehydration of hydroxide, and decomposition of calcium carbonate[12]. The conditioned cores without and with CFRP-wrapping (6 weeks), however, demonstrated noticeable mass losses of 21.6% [Fig. 11(c)] and 12.8% [Fig. 11(d)] at 1,000ºC [1,832ºF], respectively. The thermal stability of the concrete cores was also distinguishable. For instance, the core without CFRP [Fig. 11(c)] showed a rapid drop in mass at about 100ºC [212ºF], which is different from the gradual decrease of the CFRP-wrapped case [Fig. 11(d)]. These observations illustrate that the chemical reactions between the concrete and sulfuric acid were affected by the presence of CFRP, and consequently the extent of water evaporation in the concrete cores changed (the water absorbed in the plain concrete began to evaporate at 100ºC [212ºF]). It is worthwhile to note that, although the CFRP effectively protected the concrete from sulfuric acid, a certain amount of acid permeated through the CFRP evidenced by the gradual mass loss shown in Fig. 11(d).

Constitutive behavior

The stress-strain behavior of the epoxy coupons is given in Fig. 12. The ultimate stresses of the 0- and 6-week cases were respectively 44 MPa [6,400 psi] and 19 MPa [2,750 psi], on average. Because of the exposure to sulfuric acid, the stiffness of the epoxy was reduced, which resulted in the increased strains of the conditioned specimens [Fig. 12(b)] compared with those of the unexposed ones [Fig. 12(a)] at the same load level. Figure 13 shows the constitutive response of the CFRP composites with and without sulfuric acid exposure. The average stresses at failure were 4,394 MPa [640 ksi] and 3,372 MPa [490 ksi] for the 0- and 6-week exposure categories, respectively. Unlike the cases of the epoxy mentioned above, the CFRP-strain at failure was not noticeably affected by sulfuric acid because the carbon fibers were protected, in part, by the epoxy.

Capacity variation

The variation of load-carrying capacity of the epoxy, CFRP, and concrete specimens is summarized in Fig. 14. The trend of sulfuric acid-induced capacity reduction between the epoxy and CFRP was similar [Figs. 14(a) and (b)], although the former showed a higher reduction than the latter (that is, 60% versus 23% in strength loss). In the case of the cylinders tested at 0 week, the ones wrapped with CFRP revealed a 440% higher compression capacity than their plain counterparts, on average (89.4 MPa [13.0 ksi] versus 20.3 MPa [2.9 ksi] for the CFRP-wrapped and plain concrete, respectively). Such a capacity-discrepancy was augmented to 600% when the cylinders were exposed to sulfuric acid (56.9 MPa [8.3 ksi] versus 9.5 MPa [1.4 ksi] for the CFRP-wrapped and plain concrete, respectively).

Failure mode

Figure 15 demonstrates the failure mode of the individual test categories. The epoxy and CFRP failed by abrupt rupture, as shown in Fig. 15(a) and (b), respectively. It is qualitatively noticed that the degree of brittleness in failure mode (e.g., time to failure and sound at failure) appeared to be accelerated by the exposure. The failure plane of the plain concrete exposed to sulfuric acid for 6 weeks was almost perpendicular to the top and bottom loading plates [Fig. 15(c)], which is not generally observed in ordinary cylinder testing accompanied by inclined angles. This fact indicates that the cohesion of the cementitious bonder was deteriorated by the acid exposure and such disintegration of the concrete was resulted[5]. The concrete wrapped with CFRP failed by typical CFRP-rupture, as shown in Fig. 15(d). In accordance with a visual assessment between the concrete cylinders shown in Figs. 15(c) and (d), the concrete core was virtually intact because of the protection by the CFRP.

SUMMARY AND CONCLUSIONS

This study has investigated the physical and chemical behavior of a CFRP system and concrete cylinders with and without CFRP-strengthening, when subjected to a 5% sulfuric acid solution. The test approaches employed were

Fourier transform infrared spectroscopy, thermogravimetric analysis, and destructive mechanical loading. The following conclusions are drawn:

- Because of the interactions between the sulfuric acid and the alkaline component of the plain concrete, a divergence in pH scale was measured after 6-week exposure and a mass loss of 7% was accompanied. The CFRP-strengthened concrete, however, demonstrated an insignificant change in the pH and mass loss, because the CFRP sheet protected the core concrete.
- Microscopic examinations indicated that the damage caused by sulfuric acid was apparent at the surface level of the plain concrete, which revealed aggregates owing to the disintegration of the cement paste. The epoxy was deteriorated by the acid; however, no evidence of deterioration in the carbon fibers was observed at the 20 times magnification scale.
- Although the CFRP and epoxy specimens exposed to sulfuric acid showed similar chemical responses according to the Fourier transform infrared spectroscopy, their absorbance responses at specific functional groups were different due to the presence of the carbon fibers, which retarded the chemical reactions with the acid. The thermogravimetric analysis clarified that the concrete core extracted from the CFRP-strengthened cylinder was more stable than that from the plain cylinder, evidenced by the gradual and abrupt mass drops with temperature (12.8% and 21.6%, respectively).
- The exposure to sulfuric acid for 6 weeks resulted in an average strength decrease of 57% and 23% for the epoxy and CFRP coupons, respectively, and the failure strain of the former was significantly reduced. The average compressive capacity of the cylinders strengthened with CFRP was 600% and 440 % higher than the capacity of their plain counterparts with and without the sulfuric acid exposure, respectively. A visual evaluation on the failed concrete cores confirmed that CFRP-strengthening impeded the ingress of sulfuric acid into the concrete.

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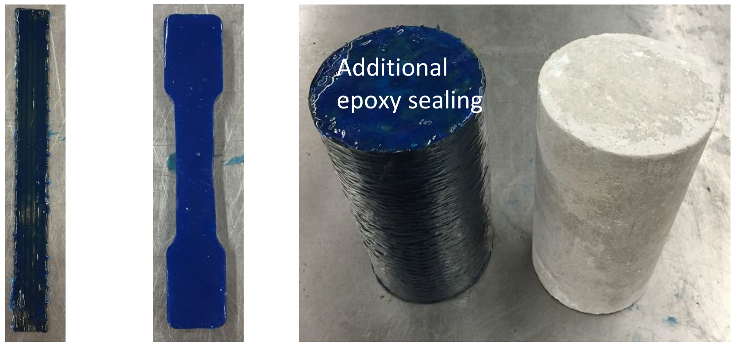
Table 1—Nominal properties of carbon fiber and epoxy

Carbon fiber		Epoxy	
Areal weight (g/m^2 [lb/ft^2])	300 [0.062]	Volatile organic compounds (g/liter [lb/gal])	20.0 [0.17]
Nominal thickness (mm [in]/ply)	0.165 [0.0065]	Mixed viscosity @ 20^{0}C (cps)	1,600
Tensile modulus (GPa [ksi])	227 [33000]	Tensile modulus (GPa [ksi])	2.6 [377]
Tensile strength (MPa [ksi])	3,800 [550]	Tensile strength (MPa [ksi])	52 [7.5]
Ultimate rupture strain	0.0167	Elongation at break	0.015

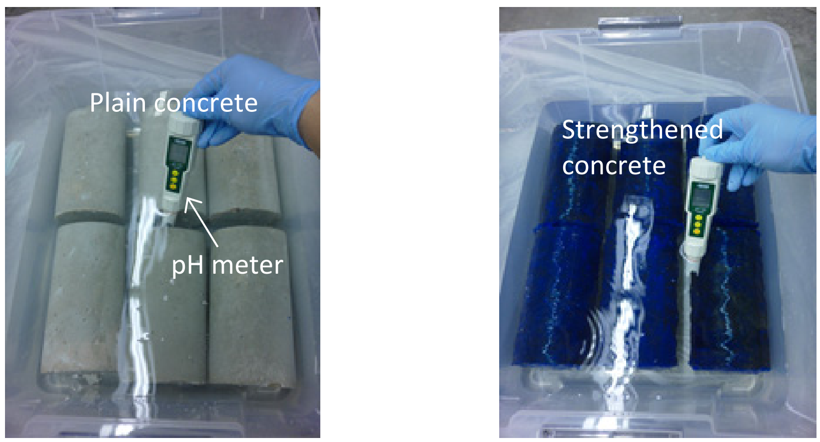
Table 2—Peak absorbance response and corresponding functional group

Wavenumber, cm^{-1} [in^{-1}]	Bond	Function group
3,000-2,850 [1,181-1,122]	C-H stretch	Alkanes
2,830-2,695 [1,114-1,061]	H-C=O:C-H stretch	Aldehydes
1,680-1,620 [661-637]	-C=C- stretch	Alkenes
1,250-1,020 [492-401]	C-N stretch	Aliphatic amines
700-610 [275-240]	-C≡C-H:C-H bend	Alkynes



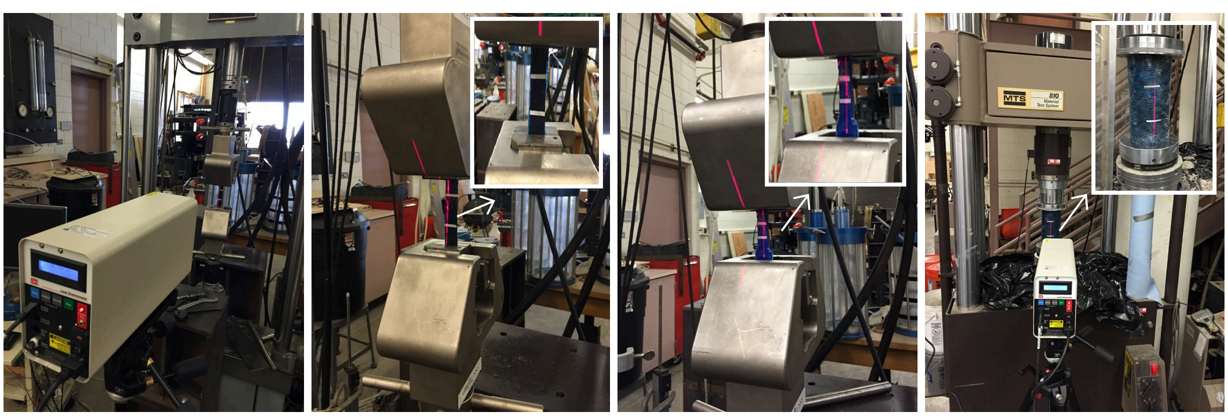
(a) (b) (c)

Figure 1—Test specimens: (a) CFRP coupon; (b) epoxy coupon; (c) concrete cylinders with and without CFRP



(a) (b)

Figure 2—Exposure to sulfuric acid: (a) container 1; (b) container 2



(a) (b) (c) (d)

Figure 3—Test setup: (a) laser extensometer; (b) CFRP coupon; (c) epoxy coupon; (d) concrete cylinder

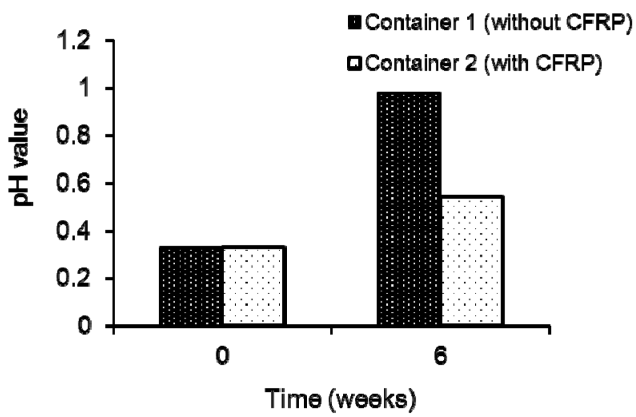
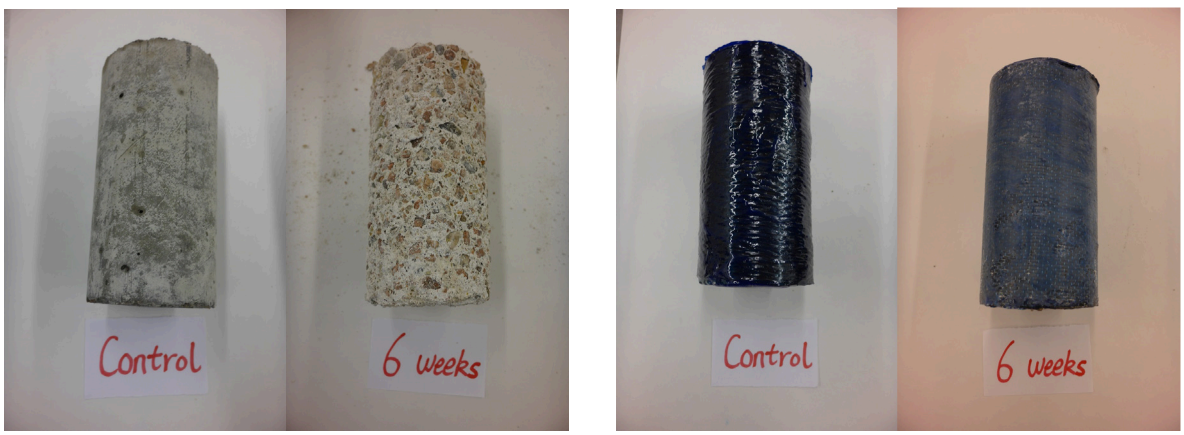
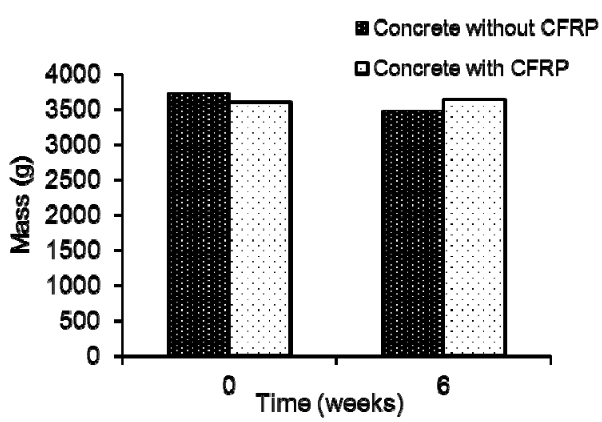


Figure 4—Measured pH values with time



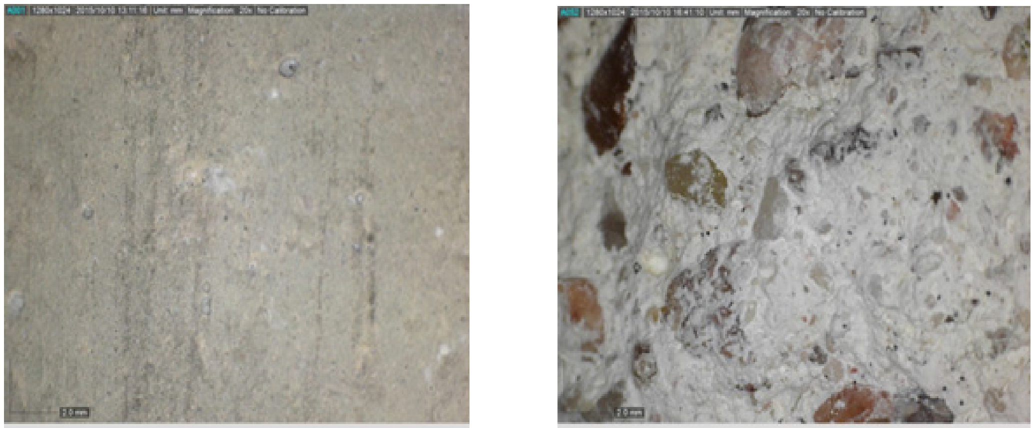
(a) (b)

Figure 5—Deterioration of concrete cylinders from 0 to 6 weeks: (a) plain concrete; (b) strengthened concrete



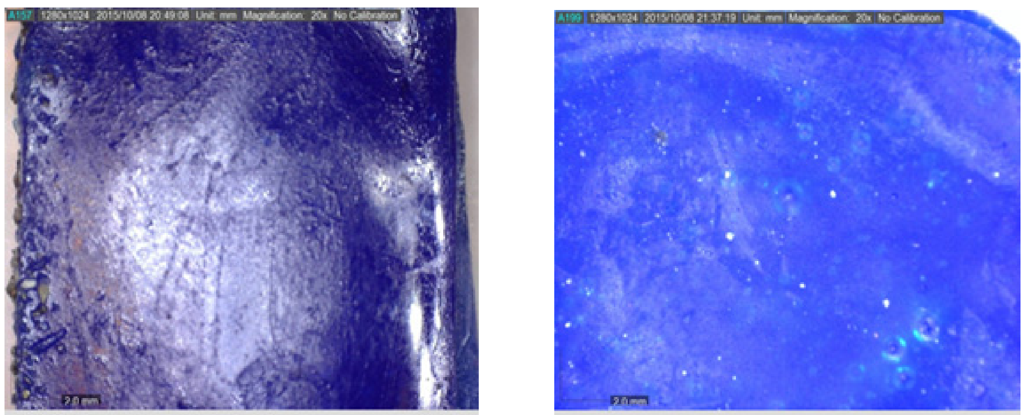
[1 g = 0.0022 lb]

Figure 6—Change in mass of concrete cylinders



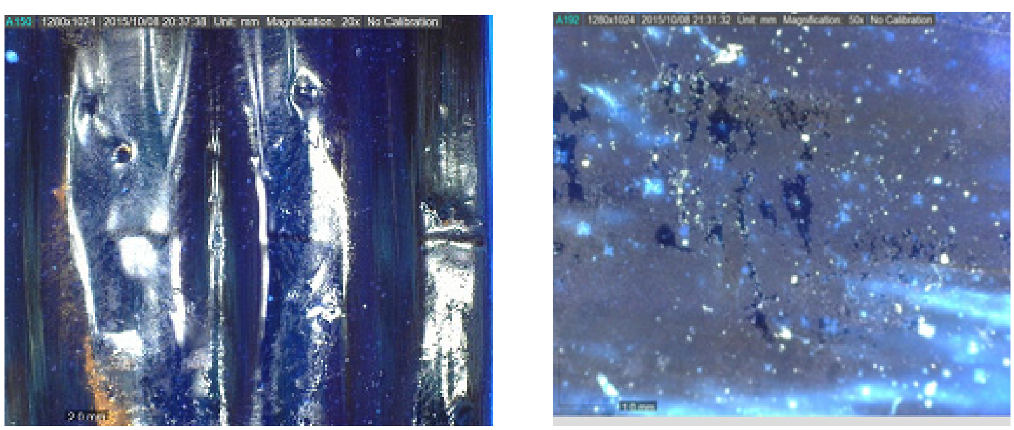
(a) (b)

Figure 7—Microscopic images of concrete surface at 20 times magnification: (a) 0 week; (b) 6 weeks



(a) (b)

Figure 8—Microscopic images of epoxy at 20 times magnification: (a) 0 week; (b) 6 weeks



(a) (b)

Figure 9—Microscopic images of CFRP sheets bonded to concrete at 20 times magnification: (a) 0 week; (b) 6 weeks