Microscopic and Durability Evaluation of In-Situ Extracted Internal GFRP Reinforcing Bars After Temporal Exposure

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Abstract. This study assesses the durability of GFRP bars in concrete bridges exposed to a real-time weather environment. In order to observe any possible mechanical and chemical changes in the GFRP bars and concrete, several tests were conducted on the GFRP bars and surrounding concrete of the extracted cores. Carbonation depth, pH, and chlorides content were performed on the extracted concrete cores to evaluate the GFRP-surrounding environment and see how they influenced certain behaviors of GFRP bars. Scanning electron microscopy (SEM) was performed to observe any microstructural degradations within the GFRP bar and on the interfacial transition zone (ITZ). Energy dispersive spectroscopy (EDS) was applied to check for any chemical elemental changes. In addition, glass transition temperature (TA) and fiber content tests were carried out to assess the temperature state of the resin and check any loss in fiber content of the bar after these years of service. The results showed that there were no microstructural degradations in both bridges. EDS results were positive for one of the bridges, and they were negative with signs for leaching and alkali-hydrolysis attack on the other. Fiber content results for both bridges were within the permissible limits of ACI 440 standard. Carbonation depth was found only in one of the bridges. In addition, there were no signs for chlorides attack in concrete. This study adds new evidence to the validation of the long-term durability of GFRP bars as concrete reinforcing used in field applications.

Keywords: Glass fiber-reinforced polymer (GFRP) · Durability · SEM · FTIR · EDS · TA · pH · Chlorides content

1 Introduction

One of the major issues resulted from using ordinary steel reinforcement is corrosion. The necessary preventive and corrective actions for corrosion are usually costly and require a continuous monitoring (Al-Khafaji et al. 2020). Different procedures including cathodic protection, epoxy coating, and galvanizing were utilized to mitigate and avoid corrosion problems, but they have not been entirely effective (Byars et al. 2003). Therefore, the search for an alternative to replace steel reinforcement has been of main interest for many

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researchers across the globe. One of these alternatives is glass fiber-reinforced polymer (GFRP) bars, as it presents themselves as a solid solution to replace steel bars owing to their fantastic features including low-conductivity, non-corrosiveness, and high strength-to-weight ratio in addition to being economically feasible (Achillides and Pilakoutas 2004). In the last ten years, the applications of GFRP bars has considerably increased including barriers (El-Salakawy et al. 2003), parking garages (Ahmed et al. 2017), and bridges decks (Mufti et al. 2007a, b).

Utilizing accelerated laboratory regimes to evaluate the GFRP durability performance via subjecting the bars to concentrated alkaline solutions does not reflect the real condition that those GFRP bars are exposed to in the field. In fact, accelerating regimes are majorly harsher on the GFRP bar's entity and performance than if they were to be exposed to a real-time weather. Porter and Barnes (1998) carried out an investigation on GFRP bars exposed to accelerated regimes to evaluate their long-term tensile strength. Both high temperatures around 60 °C (140 °F) and alkaline solutions were applied on GFRP bars for three months. Their study results showed that the residual strength of bars was between 34% to 71%.

On the other hand, a more realistic condition to use in the GFRP durability evaluation can be taken from monitoring their performance as a reinforcement in concrete structures. Mufti et al. (Mufti et al. 2007a, b) carried out a study to evaluate the durability of GFRP bars extracted from five bridges across Canada after having a service life of 8 years. Multiple examinations were utilized to assess the performance of the extracted bars including: scanning electron microscopy (SEM), energy dispersive X-Ray spectroscopy (EDS), glass transition temperature (TA), and Fourier transform infrared spectroscopy (FTIR). The tests results depicted that there were neither microstructural deteriorations nor chemical degradations after the eight years of service.

Even though GFRP bars mechanical, physical, and chemical properties have been thoroughly investigated, there is still a very limited information about the durability performance of these bars under the real-time weather effect (Mufti et al. 2007a, b) (Benmokrane et al. 2018). In this study, another real-time weather-based durability investigation was carried out to evaluate the performance of GFRP bars installed and extracted from two bridges in the USA after almost two decades of service. Several examinations were carried out on both the GFRP bar and surrounding concrete. For concrete, carbonation depth, pH, and chlorides content were utilized. While for GFRP bar, SEM, EDS, TA, FTIR, and fiber content (FC) were performed.

2 Bridges Investigated

In this study, two bridges were investigated. The first one is Southview Bridge and is situated in Missouri. This bridge consisted originally of four box culverts and topped with steel reinforced deck. An expansion occurred in 2004 where another four box culverts were added, but this part was topped with GFRP reinforced deck. The bridge is exposed to a temperature ranged from −5° to 35 °C (22° to 95 °F). Additionally, it is subjected to recurrent wetting, drying, freezing, and thawing cycles. In winter seasons, deicing salt is frequently used.

The second bridge is Sierrita de la Cruz Bridge and is situated in Texas. This bridge was originally reinforced with mild steel bars and was majorly corroded; therefore, it was considered structurally not sound. A replacement in 2000 was carried out using GFRP bars. The GFRP bars were utilized in the deck of the bridge. This bridge is exposed to wetting, drying, freezing, and thawing cycles. In both bridges, sand-coated E-glass fibers/vinyl ester resin bar were used. Figure 1 shows both bridges.

Fig. 1. (A) Southview Bridge, Missouri (B), Sierrita de la Cruz Bridge, Texas

3 Cores Extractions and Samples Preparations

Multiple cores with 102 mm (4 in.) diameter were extracted by expert personnel from both bridges. The GFRP bars were encapsulated inside the cores. Ten cores were taken from Southview Bridge and five cores were extracted from Sierrita de Cruz Bridge. After extraction, the cores were filled immediately with a rapid-curing cementitious grout. The cores were then submitted to the university's laboratory for inventory. The GFRP bars extracted from both bridges were 19 mm (0.75 in.) diameter. Figure 2 depicts a core extraction and specimen. Specimen preparation differs from test to another. Since some of these tests require only a tiny piece of material, each core was cut into multiple slices parallel to the bar length orientation. After that, each slice was cut into several slices until what was left is the GFRP bar with a little concrete surrounding it. For SEM and EDS tests, GFRP coupons were properly prepared and polished for the examination using sand papering and polishing material. While in TA test, little pieces (around 10 mg) were cut from the bars. All the specimens were conditioned before conducting any test. The conditioning included placing the specimens inside an oven for 48 h at 40 °C to remove

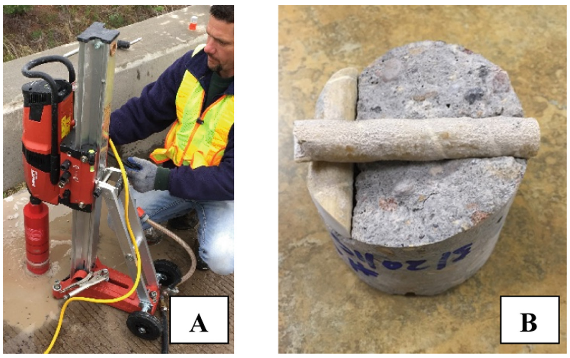

Fig. 2. (A) Cores extraction, (B), GFRP with its concrete core

any excess moisture that might be picked up from shipping and inventory, although the specimens were hermetically sealed.

4 Concrete Tests and Results

The concrete surrounding the GFRP bars was examined to see what environment surrounded the GFRP bars and how that influenced the overall performance of the bar after all these years of service. The concrete examinations conducted in this investigation were pH, carbonation depth, and chlorides content.

pH Test – This test was carried out to see the overall alkalinity level in concrete. The average pH level in such type of Portland cement-based concrete is between 11 and 12 (Grubb et al. 2007). However, pH level from the concrete surfaces lowers as a result of the reaction between the carbon dioxide (CO_2) from the atmosphere and alkalis in the concrete. Grubb procedure was used to determine the pH level (Grubb et al. 2007). In Southview Bridge, pH results were around 13 which is a little high, probably due to the ingress of hydroxide ion from the alkaline environment within and surrounded the concrete. In Sierrita de la Cruz Bridge, the pH results were found to be around 11 which complimented the expectation for such type of concrete and age.

Carbonation Depth – This test was performed to see if there were any signs of carbonation reaction which is the reaction of CO_2 from the atmosphere with the alkalis from concrete. When carbonation reaction takes place, pH level of concrete decreases to around 10 or 9. As a result, the concrete layer became relatively acidic. It has been suggested that when the carbonation depth is equal to the concrete depth corrosion occurs (Wang 2017). RILEM-88 (RILEM and Materials 1994) was utilized in this study to perform the test where a 1% of phenolphthalein-70% ethyl alcohol solution was sprayed to a fresh cut of the concrete surface. If there were carbonations on the concrete surface, the surface color would not change due to the acidic environment, otherwise the surface color would turn into purple color due to the alkalinity of surface. The more purple the surface is, the more alkalinity is available. In Southview Bridge, the test results were negative which confirms the high results of pH found in that concrete. In Sierrita de la Cruz Bridge, some spots of carbonations about 13 mm (0.5 in.) was found. The results are shown in Fig. 3.

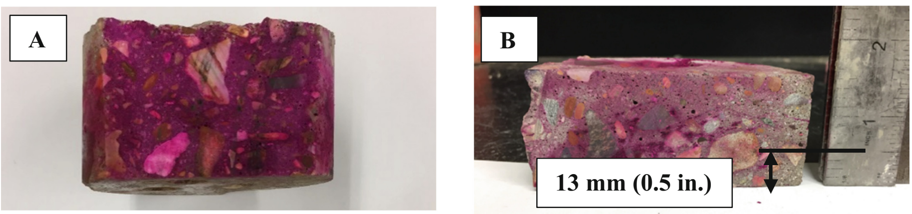

Fig. 3. Carbonation depth (A) Southview Bridge, (B) Sierrita de la Cruz Creek Bridge

Chlorides Content – This test was conducted to see the level of chlorides in concrete. In this study, acid-soluble analysis is carried out to find the total content of chlorides. To utilize this method, rapid chlorides testing (RCT) was carried out where a 1.5-g (0.05 oz.) of concrete powder was obtained from the concrete cores at several locations. Next, the samples were placed inside chloride-agent vails and left to react with the agent for one day. After that, testing for chlorides were taken place voltage-based meter. The test results were compared to the significance table from Broomfield (2007) where the chlorides level can be considered negligible if the content is lower than 0.03% and high if it is over 0.14%. The test results for both bridges were less than 0.03%, so chlorides effect can be neglected.

5 GFRP Tests and Results

The GFRP bars were assessed for durability using five tests including: SEM, EDS, TA, FTIR, and FC.

SEM Test – This test was carried out to see if there were any microstructural degradations resulted from the real-time weather exposure. Slices of 25 × 25 × 25 mm (1 × 1 × 0.25 in.) were cut from the bridges' cores and were then ground using different levels of sand papering. After that, the specimens were then polished and coated with gold to make the specimens conductive (since neither glass fiber nor concrete is conductive). After that, the specimens were conditioned to get rid of any excess moisture picked up from preparation of samples. The samples were then tested in the SEM apparatus where different magnification grades were explored to examine not only the surface of GFRP bar for microstructural deteriorations, but also the interfacial transition zone (ITZ) between the concrete and bar. The tests results showed that there were no obvious signs of microstructural deteriorations in fibers, resin, and neighboring areas of the bar. Also, there were no distinct signs of bond loss around the ITZ of the concrete-to-bar. Figure 4 shows SEM images from the tested bridges.

FTIR Test – This test was carried out to check for the hydroxyl levels in the GFRP. The pH levels of one of the bridges (Southview) was high, therefore this test was necessary to conduct. Small chunks of the GFRP bar were taken around 2 mg and were then ground with bromide potassium (KBr) to make a halide disk. The specimens were then exposed to infrared in the FTIR apparatus. In Sierrita de la Cruz, the hydroxyl group were found to be within the normal range which is between 3000 and 3600 cm^{-1}. On the other hand, in Southview Bridge, the results of hydroxyl group were a little high (a little over 3700 cm^{-1}). This confirmed the high pH seen in the concrete core. Figure 5 shows the FTIR results.

EDS Test – This test utilized to observe any changes from the environmental exposure on the chemical elemental concentration. The pore water solution of concrete is considered highly alkaline, as it includes abundant sodium, and potassium (Mufti et al. 2007a, b). Along with the alkalis of concrete's pore water solution, there are ones that are a constituent of the glass fiber. When there is an ample hydroxide, the pH level rises, and

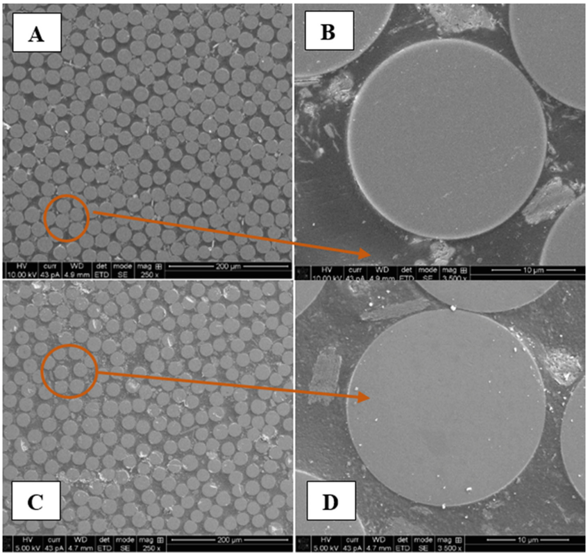

Fig. 4. SEM images (A) Sierrita de la Cruz at 250× magnification, (B) Sierrita de la Cruz at 3500× magnification, (C) Southview at 250× magnification, (D) Southview at 3500× magnification

leaching issue might take place. Leaching in fibers is the process where the alkalis of fibers are extracted by hydroxyl group to form a more stable chemically compound such as sodium hydroxide (Nkurunziza et al. 2005). It is important to note that EDS apparatus cannot detect elements lower than sodium, and appearance of alkalis in the resin matrix can be used as a sign of leaching. In this test, a 10–20 keV electron was exerted on the same specimens used for SEM test. The results showed that there were no zirconium which confirms the glass fibers were not alkali-resistant type of fibers (Kamal and Boulfiza 2011). Furthermore, the constituent's elements of fibers were detected including aluminum, calcium, silicon, sodium, and oxygen. In addition to those elements, magnesium was observed too which confirms that the fibers were E-glass and not ECR-glass (Gooranorimiet al. 2016). Elements such as gold and palladium were detected too, but they were present as a result of coating process of the specimens. For the resin, carbon, the main element, was found as well. In Sierrita de la Cruz, all the main elements of fiber and resin were detected. However, sodium, and silicon were seen in the resin too which can indicate for alkali-hydrolysis attack and/or leaching issue (Kamal and Boulfiza 2011), but those elements were detected too in the control specimens as well as, to further confirm, the pH results were within the normal range (not high enough to make the environment highly alkaline). In Southview Bridge, the main elements were seen too, but sodium and silicon were detected in the resin. The pH results of this bridge were a little high, so there is a significant chance for alkali-hydrolysis attack at least a little. To confirm the attack, FTIR test was performed to reveal the hydroxyl levels in the GFRP bar. The FTIR test results showed a high hydroxyl level, therefore there is a strong chance that alkali-hydrolysis occurred. However, even if it did occur, it is still within its embryonic stage, because SEM tests results did not show any obvious signs for chemical-related deteriorations.

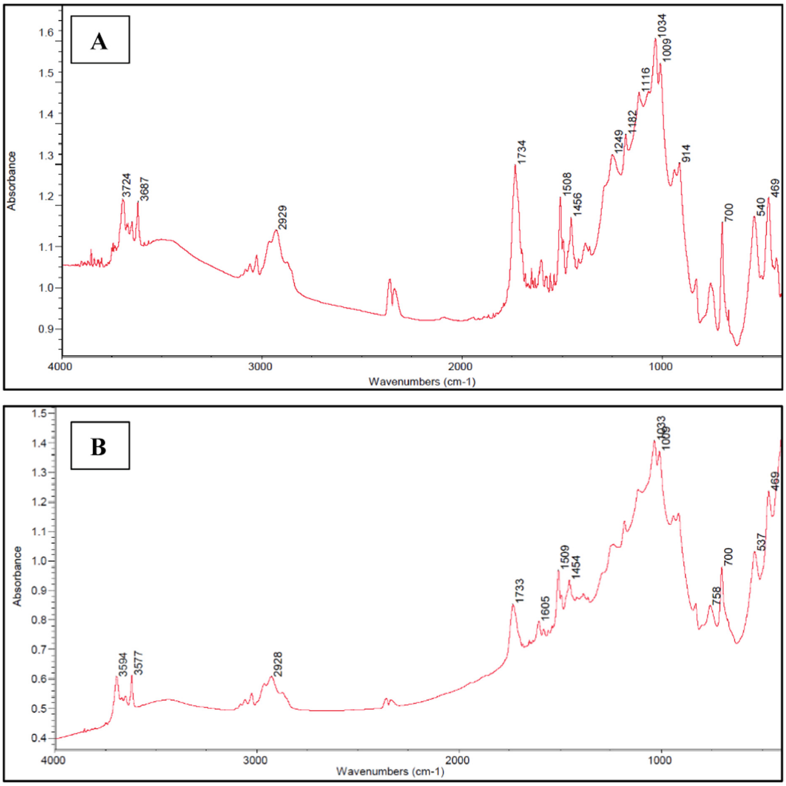

Fig. 5. FTIR test results (A) Southview Bridge (B) Sierrita de la Cruz Bridge

TA Test – This test was carried out determine the glass transition temperature (TA) of the bar as it can be affected by the hydroxyl group abundance. Glass transition temperature is defined as the temperature region in which the physical features of matrix alter from being hard to soft materials (Epoxy Technology Inc., 2012). The test can be performed using either dynamic mechanical analysis (DMA) or differential scanning calorimetry (DSC). In this research, DSC was utilized where small pieces about 10 mg (0.0004 oz.) were obtained from the bar. After that, they were installed inside the TA-DSC apparatus. A temperature ramp of 5 °C (41 °F) per minute with a total of elevated temperature of 200 °C (392 °F) from room temperature. In Sierrita de la Cruz, a slight reduction in TA was observed in compared to pristine bars. It is most likely the curing temperature used in the filed were a little different from that of the pristine bars, because neither its FTIR results showed a high hydroxyl level, nor its SEM results showed obvious signs for microstructural degradations. In Southview Bridge, there were no pristine bars to compare results with, but since vinyl-ester matrix was applied, the TA results were compared to the ASTM E1640 (2013). The results were less than the standard by approximately 25 °C (77 °F). Hydroxyl group was found to be a little high in this bridge, so this could be the reason for such reduction. TA results are shown in Table 1.

Table 1. TA test results

Southview Bridge	Core 1 - bar	Number of samples	3
		Average temperature °C (°F)	72 (162)
		Variation coefficient %	6.94
	Core 2 - bar	Number of samples	3
		Average temperature °C (°F)	75 (167)
		Variation coefficient %	3.3
Sierrita de la Cruz Bridge	Control bar	Number of samples	3
		Average temperature °C (°F)	81 (178)
		Variation coefficient %	17
	Core 1- bar	Number of samples	3
		Average temperature °C (°F)	74 (165)
		Variation coefficient %	9.2

Fiber Content (FC) – This test was performed to check if the content of fiber was affected by all these years of service under real-time weather exposure. This test is fashioned for polymeric matrices (Agarwal et al. 2015). This test was utilized following the recommendations of ASTM D2584 (2005). To perform the test, little chunks (about 5 g (0.18 oz.)) were taken from the bar and were then weighed. After that, the specimens were inserted in a muffle furnace at 575 °C (1010 °F) until the resin was totally disappeared. The burnt specimens, were then, taken out of the furnace and reweighed. Table 2 shows the FC results. The differences in weights yielded the fiber content. In both bridges, the fiber contents were more than 70% and had met the requirements of the ASTM D7957 standard (2017) (Figs. 6 and 7).

Table 2. FC test results

Southview Bridge	Core 1 - bar	Number of samples	3
		Fiber content %	69.9
		Resin content %	30.1
	Core 2 - bar	Number of samples	3
		Fiber content %	71.8
		Resin content %	28.2

(*continued*)

Table 2. (*continued*)

Sierrita de la Cruz Bridge	Control bar	Number of samples	2
		Fiber content %	80.5
		Resin content %	19.5
	Core 1 - bar	Number of samples	3
		Fiber content %	81.6
		Resin content %	18.4

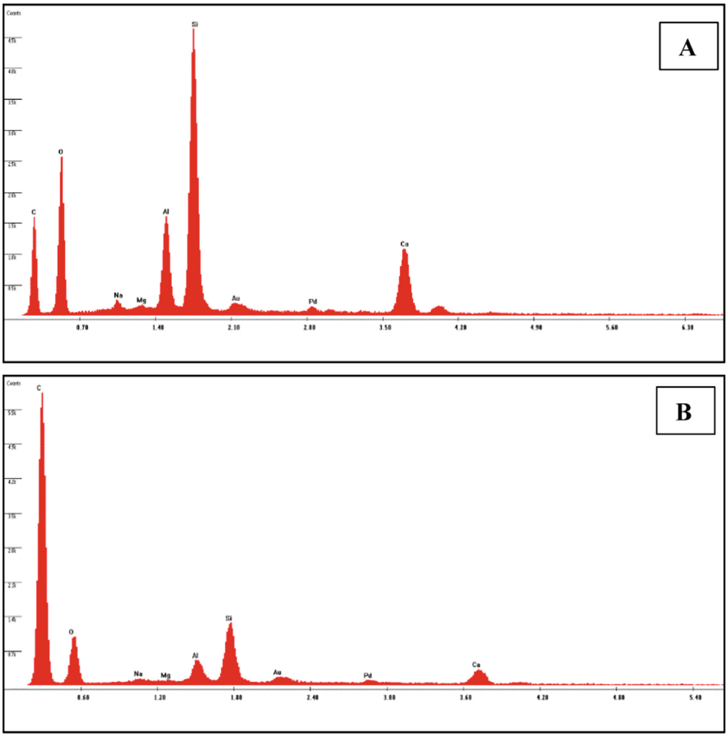

Fig. 6. EDS test results of Sierrita de la Cruz Creek Bridge (A) fiber (B) resin

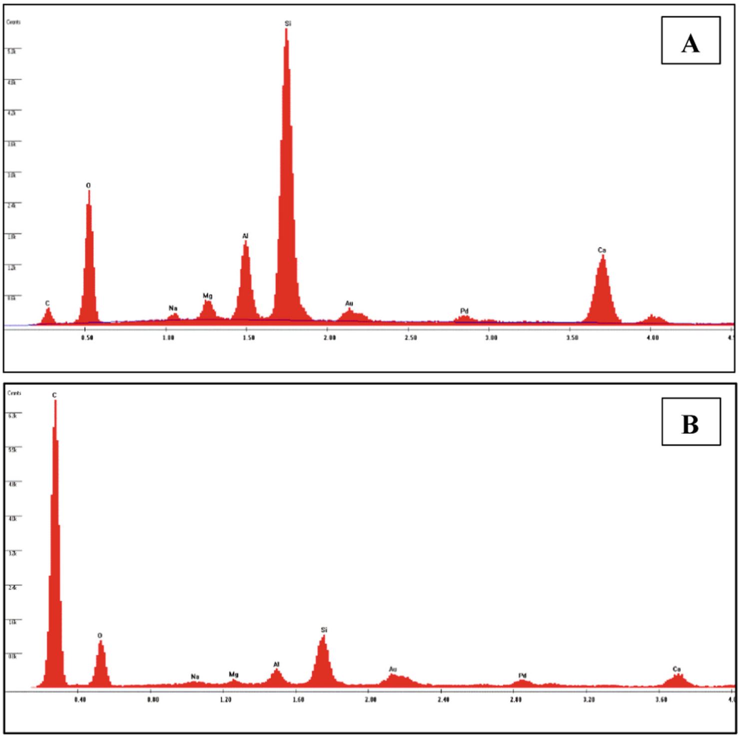

Fig. 7. EDS analysis of Southview Bridge (A) fiber (B) resin

6 Conclusion

A durability study was carried out to assess the performance of GFRP bars extracted from two bridges after being in service for almost two decades. For Southview Bridge, it can be concluded that there were some signs for alkali-hydrolysis attack, because the concrete pH test result was high for such concrete; meaning there is a chance for high level of hydroxyl. FTIR test results confirmed that the hydroxyl level was high in this bridge. In addition, TA results were less than the ASTM standard, and EDS results showed silicon and sodium in the resin. For Sierrita de la Cruz Bridge, SEM showed no obvious signs of deteriorations. EDS results did not show any chemical abnormalities, except that sodium and silicon were detected, but they were detected in pristine bars too. TA test results showed a slight reduction in glass transition temperature. Fiber content for both bridges were within the standard acceptable limits.

This study adds new evidence to the validation of the long-term durability of GFRP bars as concrete reinforcing used in field applications. Both bridges demonstrated the long-term performance and viability of GFRP bars in bridge decks after nearly 20 years of field exposure. The work also provides a framework for sampling and evaluation of in-service GFRP bar extraction to monitor and develop a larger database of field collected data.

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