Enhancement of fabric–mortar interfacial adhesion by particle decoration: insights from pull-off measurements

Matan Birenboim · Amr Alatawna · Raghu Sripada · Lior Nahum · Lucas Luciano Cullari · Alva Peled · Oren Regev

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Abstract Polymer-impregnated carbon fabric is used as an alternative to metallic reinforcement bars in cementitious materials, which is then termed textile-reinforced concrete (TRC). In this study, the bond strength between the cement-based matrix and the fabric was enhanced by decorating the polymer (an epoxy) coating the carbon fabric with hydrophilic micron-size particles (cement or silica) or nanocarbons (functionalized carbon nanotubes or graphene oxide). Cement powder decoration led to a 25% increase in the bond strength (measured by a pull-off test) and a 30% improvement in the mechanical properties of the composite. At the micron scale, the decoration resulted in the formation of a 100-μm thick interlayer between the decorated fabric and the cement-based matrix. Unexpectedly, exposure of the cement-decorated samples to a NaCl environment (as in off-shore constructions) resulted in enhanced bond strength due to the growth of salt crystals at the fabric–matrix interface.

Keywords Carbon fiber · Adhesion · Surface treatments · Textile-reinforce-concrete

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Abbreviations

TRC Textile-reinforced concrete
NE Neat epoxy
CEM Cement powder
SF Silica fume
GO Graphene oxide
f-CNT Functionalized carbon nanotubes
SP Superplasticizer
f Fresh
g Gelated
RH Relative humidity
SEM Scanning electron microscopy
EDS Energy dispersive spectroscopy
1 Introduction

In most structural elements, the low tensile strength of the brittle cement-based matrix (e.g., concrete) is enhanced by steel reinforcement bars (known as rebars) [1–3] with the aim of sustaining the developed tensile forces in the element [1, 3]. However, rebars tend to corrode upon exposure to moisture and air, particularly in a chloride environment [1, 3], leading to the deterioration of the mechanical properties, or even failure of the concrete element [1, 3]. A possible solution to this problem lies in replacing the steel rebars with carbon-based fabrics to produce textile reinforced concrete (TRC) [1, 4–8]. Since carbon fabrics are not sensitive to corrosion and have a higher tensile strength than steel rebars (~ 4 vs 0.4 GPa) [1, 5, 6], the TRC approach offers both higher strength and improved durability upon exposure to chlorides or to high temperatures [9]. An additional advantage is that the textile fabric is malleable and can be arranged in 2D or 3D arrays [5, 6], facilitating the production of elements with complex shapes that are much more difficult—or even impossible—to produce with steel-rebar reinforcement. Moreover, in the TRC approach, there is no need for a thick concrete cover layer to protect the reinforcing element against corrosion, and therefore at similar load-bearing capacity much less concrete is needed per structural element [6].

The above notwithstanding, the TRC approach is not problem free, and we describe here some of the challenges—and their solutions—in integrating carbon fabrics as reinforcement for cement-based matrices. One of the most pressing problems derives from the fact that the carbon fabric is made up of yarns that are composed of hundreds of densely packed filaments [10, 11] in which the filament-to-filament gap is too small (< 3 μm) to allow the penetration of cement particles during the preparation of a TRC element [11, 12]. As a result, only the filaments at the perimeter of the yarn (termed the sleeve) come into direct contact with the cementitious matrix, while the inner filaments (the core) do not. Consequently, upon tensile loading, the unique microstructure of the TRC exhibits telescopic pull-out behavior, in which the sleeve filaments are ruptured while the core filaments slide against each other, thereby impairing the reinforcing efficiency of the yarn and hence the tensile properties of the composite [10, 13].

In addressing this problem, it has been suggested that the load-bearing capacity of the fabric and the tensile properties of the composite can both be improved by impregnating the fabric with a polymer, e.g., an epoxy [6, 7, 11], that would provide enhanced stress transfer between the sleeve and core filaments during loading [10, 13], i.e., the yarn would then behave as a single unit during loading. However, it has been shown that due to the hydrophobic nature of epoxy-impregnated fabrics, weak bonding is developed between the fabric and the hydrophilic cementitious matrix (containing water and oxides) [6]. Epoxy is also known to have poor resistance to high temperature while its coating may also lead to lower flexibility of the fabric. The low adhesion may lead to fabric-matrix separation, known as delamination, and hence to catastrophic failure of the entire element [6, 14]. Therefore, a major challenge in the application of the TRC approach is to enhance the adhesion between the fabric and the matrix.

A possible approach to preventing delamination is to decorate the surface of the epoxy-impregnated fabric with hydrophilic particles, e.g., silica fume. It was found, for example, that applying micro silica particles to the fabric surface improved the tensile properties of TRC [14, 15], with the improvement being attributed to a pozzolanic reaction between the silica and the cement [16, 17]. In a different study, it was shown that enhanced adhesion between carbon yarn and cement-based matrix was facilitated by the presence of silica fume particles at the yarn surface, as evaluated by applying a pull-out test [11]. However, this test does not directly measure the fabric-matrix adhesion energy but rather its friction-based bonding and relates mainly to mechanical anchoring.

The current work aims to explore the direct adhesion bonding between the mortar and a range of epoxy-impregnated fabrics, each being decorated with hydrophilic particles of a different material, namely, plain cement powder (designated CEM), silica fume (SF), carboxyl-functionalized carbon nanotubes (f-CNT) and graphene oxide (GO). These materials were chosen on the basis of our premise that the CEM and SF particles would react with the matrix via hydration and pozzolanic reactions, respectively [16–19], and that the oxidized nano-carbon materials (f-CNT and GO [20, 21]), being partly hydrophilic [22–26], would interact with both the epoxy and the cement. Two other considerations influenced our choice of the latter two...
First, they possess a relatively high intrinsic mechanical strength [27–29], which will serve to mechanically anchor the coated fabric in the matrix and hence to significantly enhance the mechanical properties of cementitious matrices [22, 25, 26, 30–37], and second, they differ in dimensionality (1D and 2D for f-CNT and GO, respectively) and oxygen content (< 10% and 40%, respectively) [22, 38, 39].

The decoration techniques of the different particles were aimed at placing them as close as possible to the fabric-matrix interface to allow proper bonding. For CEM and SF, we used simple and straightforward powder coating. To avoid hazardous dispersion of nanoparticles in the air, f-CNT and GO were sprayed on the epoxy-impregnated fabric surface. The mechanical properties of nano-reinforced epoxy composites are well known and studied [40–43], however, their effect (when sprayed) on the fabric-matrix adhesion is studied here for the first time.

In the current study, we thus explored the effect of various surface decoration approaches on the adhesion bond strength between the fabric and the cementitious matrix by conducting pull-off tests (ASTM D7234) and compared the results to pull-out test. In the pull-off technique, a normal force applied on the sample leads to complete separation of the mortar bulk and the carbon fabric. The maximal force applied divided by the detached cross section is defined as the bond strength between the mortar matrix and the fabric [44].

2 Materials and methods

2.1 Materials

The reinforcing fabric (warp knitted fabric with stitch length of 4.2 mm along the warp direction, Fig. S1, supplementary information [45]) made of carbon yarns (1600 tex, 4 GPa tensile strength) was obtained from ITA Germany. The following materials were used as received for preparation of the matrix: CEM (Portland cement I 52.5 N; Nesher Israel Cement Enterprises, Ltd), dune sand 130 (0.6 mm, Yehud Clays Inc., Israel), and a polycarboxylate ether (PCE)-based superplasticizer (designated SP; LP-530, Larsplast, Ltd, Israel). The following materials were used for decorating the fabric: Epoxy resin (Part A EP 520 and Part B EPC 520, Polymere G); SF (1 μm diameter, Elkem silica fume Grade 920, Elkem, Germany); GO (10 μm lateral size, GO-V30, Standard Graphene Inc., South Korea); and carboxyl-f-CNT (10–20 nm in diameter, Cheaptubes Ltd, USA). The characteristic length of the decorating materials are of great importance in the analysis of the results and are therefore summarized in Table 1.

2.2 Preparation of the mortar

For preparation of the mortar, 0.5 wt% SP relative to cement powder and 20 wt% of the total quantity of required water were mechanically mixed (120 W RK-2200 overhead mixer, MRC) for 2 min at 500 rpm. Simultaneously, cement powder and sand (cement:sand = 1:1.3 w/w) were added to the remaining 80 wt% of the total required water and mixed mechanically (370 W mixer, 65-L0502, CONTROLS) for 2 min at 140 rpm. The SP solution was then poured into the mortar, and the entire mixture was mixed mechanically (370 W mixer, 65-L0502, CONTROLS) for 2 min at 140 rpm, followed by 4 min of mixing at 285 rpm. The water/cement ratio was 0.45 (w/w).

| Decoration material                        | Designation | Characteristic size |
|-------------------------------------------|-------------|---------------------|
| Silica fume                               | SF          | 1 μm diameter        |
| Cement                                    | CEM         | 17 μm diameter       |
| Carboxyl-functionalized carbon nanotubes  | f-CNT       | 10–20 nm diameter    |
| Graphene oxide                            | GO          | 10 μm lateral size   |
The fabric–matrix bond strength was enhanced by optimizing the viscosity of the mortar (140 flow value, according to ASTM C1437) to allow full penetration of the mortar into the spacings between the fabric yarns ($\sim 0.8 \times 0.7\text{cm}^2$) during casting. After 14 days of hydration in lime saturated solution, the mortar matrix demonstrated compressive and flexural strengths (6 specimens for each test, ASTM C1679 and C293, respectively) of $61.4 \pm 0.2$ and $5.4 \pm 0.7$ MPa, respectively.

2.3 Decoration of the fabric

For preparation of the impregnated carbon fabric, the fabric was first uniformly and gently brushed and impregnated with the epoxy (resin:hardener ratio of 10:3, Fig. 1a). Thereafter, the epoxy was decorated with fillers either by powder coating (CEM and SF; Fig. 1b) or by spray coating (f-CNT and GO; Fig. 1c). The decoration was performed either immediately after the epoxy impregnation (fresh state; designated with the suffix ‘f’) or after 90 min of epoxy aging (gelated state in which the epoxy crosslinking process is already in progress; designated with the suffix ‘g’), to confirm that the decorating particles are partially embedded in and not completely covered by the epoxy coating. Since powder coating with the fluffy f-CNT and GO powders may result in hazardous dispersion of particles in the air, these decorations were applied by spraying of an ethanol-based dispersion. CEM and SF particles were not applied by spray coating, since powder coating is much simpler and does not require dispersion and optimization.

For the powder coating, the CEM or SF powder was spread on a clean tray. The epoxy-impregnated carbon fabric (fresh or gelated) was then gently pressed into the powder (both sides, in a manner similar to preparing schnitzel for frying) to fully decorate the impregnated epoxy (Fig. 1b) and then transferred to a clean tray for epoxy curing. The fabric was weighed before and after decoration for evaluation of the total amount of decorated particles [46]. For the spray coating, the f-CNT or GO was dispersed in 50 ml of ethanol (2 mg/ml, total amount of 100 mg of nano particles decoration) and mixed mechanically (120 W RK-2200 overhead mixer, MRC) for 15 min at 1000 rpm before being poured into the tank of a spray gun (0.8-mm nozzle, Star S106T/J, Queensland, Australia). The spray setup included a compressor (TC-AC 190/6/8 OF Einhell, Dragomiresti, Ilfov, Romania) that fed compressed air and liquid to the spray gun. The dispersion was sprayed onto the epoxy-brushed carbon fabric (fresh or gelated) for 1 min (50 ml/min, 4 bar, 20 cm spray distance from fabric; Fig. 1c). In addition, fabric impregnated solely with neat epoxy (without further decoration; designated NE) and plain fabric (not impregnated) were prepared as reference samples. A summary of the prepared systems is given in Table 2.

![Fig. 1](image)

**Fig. 1** Schematics of fabric impregnation and decoration. a The plain fabric was impregnated with fresh epoxy, followed by either b powder coating (SF or CEM) or c spray (GO or f-CNT) coating. The decoration was carried out either immediately after epoxy impregnation (fresh) or after 90 min of epoxy hardening (gelated). d The impregnated fabric was then integrated into a TRC structure
2.4 Preparation of TRC samples

A TRC sample for the pull-off tests was prepared by pouring mortar into the mold shown in Fig. 2a such that its eight holes (2 cm in diameter and 0.5 cm height) were completely filled. The epoxy-impregnated fabric was then placed on top of the 8 mortar-filled holes (Fig. 2b), and additional mortar was cast on top of the fabric until the entire mold was filled (1 cm thickness, Fig. 2c). At this stage, the fabric was covered on both sides with mortar. After 24 h of aging, the mold was removed, and the eight-sample setup was turned upside down (Fig. 2d), providing the eight protruding cylindrical specimens that would be used for the pull-off testing. Thereafter, the molded TRC sample was cured for 12 days either in air at 23 °C and 60% relative humidity (RH) or immersed in an aqueous 3 M NaCl solution, followed by 24 h of curing at 23 °C and 60% RH, so the total curing time was 14 d for all samples. All the samples were thus tested 14 days after casting. A number of specimens with different decorations of the epoxy-impregnated fabrics (Table 2) were produced in this way for the pull-off testing.

In addition, samples of TRC beams (20 × 20 × 80 mm³) were prepared for measuring the flexural properties of the different compositions. A single layer of fabric was positioned 5 mm above and parallel to the bottom of the mold into which mortar was then poured. The obtained TRC sample (Fig. 3) was removed from the mold 24 h after casting and then cured in a lime-saturated solution for 12 days, followed by 24 h of drying at 23 °C and 60% RH before the flexural measurements were performed.

Table 2  Detailed description of the TRC samples

| System abbreviation* | Decorating material     | Coating method       | Fresh/gelated state epoxy |
|----------------------|-------------------------|----------------------|---------------------------|
| Plain fabric         | –                       | –                    | –                         |
| NE                   | –                       | –                    | –                         |
| CEMf                 | Cement powder           | Powder               | Fresh                     |
| SFf                  | Silica fume             | Powder               | Fresh                     |
| f-CNTf               | Functionalized carbon nanotubes | Spray | Fresh               |
| GOf                  | Graphene oxide          | Spray                | Fresh                     |
| CEMg                 | Cement powder           | Powder               | Gelated                   |
| SFg                  | Silica fume             | Powder               | Gelated                   |
| f-CNTg               | Functionalized carbon nanotubes | Spray coating | Gelated               |
| GOg                  | Graphene oxide          | Spray coating        | Gelated                   |

All samples (except the plain fabric) were epoxy-impregnated

*NE = fabric impregnated solely with neat epoxy; the suffix ‘f’ at the end of an abbreviation indicates freshly prepared; the suffix ‘g’ at the end of an abbreviation indicates after 90 min of epoxy aging (gelated state)

Fig. 2  TRC sample preparation for the pull-off testing: a Mortar was poured into a mold with eight holes (empty and filled holes are indicated by red and yellow arrows, respectively). b After the holes were completely filled, the fabric was placed over the surface of the filled holes and c additional mortar was then added to completely fill the mold. d TRC sample after demolding (turned upside down)
During the flexural test, the fabric was located in the tensile zone (Fig. 3) of the specimen [47].

2.5 Characterization methods

2.5.1 Pull-off tests

The adhesion (chemical bond strength) between the fabric and the mortar matrix was determined by pull-off tests (ASTM D7234), as shown in Figs. 4 and 5. In this test method, a dolly (2 cm in diameter) was glued to the top surface of each of the eight protruding mortar cylinders (that were attached to the reinforced mortar plate, Figs. 4a, b and 5) with a strong adhesive (EP11HT Resinlab adhesive, parts A:B ratio of 1:1). After 24 h of adhesive curing, the sample was ready for pull-off test, which was performed as follows: The dolly was pulled-off (DeFelsko PosiTest AT-A adhesion apparatus, rate of 0.2 MPa/s, arrow in Fig. 4d) from the mortar cylinder until clear failure in the vicinity of the fabric–matrix interface was obtained [4, 8, 22, 37, 38], revealing the imprint of the fabric on the matrix (Fig. 4e, f). The force at this point divided by the detached cross section was defined as the bond strength between the mortar and the fabric. Each data point was an average of eight measurements (Sect. 2.4).

2.5.2 Scanning electron microscopy

SEM imaging was performed before (on the decorated fabric–mortar interface cross section) and after the pull-off test (on the fractured TRC surface) with a high-resolution cold-field emission gun SEM (JSM-7400F, JEOL) with the dual aim to explore the bonding morphology and failure mechanisms of the various decorated fabrics, and to evaluate the uniformity of the decoration. A thin cross-section (< 1 cm) was sliced–using an industrial saw (TS 351, Shatal)–out of the pull-off sample before the pull-off test.
(Fig. 5, red rectangle) to examine the microstructure of the interfacial area. After the pull-off test, the fractured surface (Fig. 4f) was cut from the TRC plate for imaging and energy dispersive spectroscopy (EDS) analysis. The EDS (installed on a Various 460L SEM, Oxford EDS setup) was performed on both the interfacial cross-section samples and the fractured composite surface for elemental analysis at the fabric-matrix interface.

2.5.3 Flexural properties of TRC beams

The flexural properties of the TRC beams were determined on $20 \times 20 \times 80$ mm$^3$ TRC specimens by a three-point flexural test (ASTM C293) using a displacement control mode (LVDT adaptor, 0.5 mm/min) on an Instron 5982 testing system. Three specimens for each sample were measured (Sect. 2.4).

3 Results and discussion

SEM imaging and elemental analysis of the decorated fabric are presented first. Thereafter, the results of the pull-off test, investigating the effect of the different decorations (CEM, SF, GO and f-CNT) on the fabric–matrix bond strength are described. The results include the effects of the gelation time (or epoxy viscosity) on the decorated epoxy and of the curing environment (ambient conditions vs NaCl solution) on the properties of the TRC samples. The microstructure of the TRC samples was then explored by SEM imaging of the interfacial cross section (before the pull-off test) and the fractured surface (after the pull-off test). Plain fabric (without any decoration) and fabric impregnated with NE were used as reference systems. Finally, the flexural strength of the TRC beams was examined in order to evaluate the influence of the decoration of the fabric on the overall mechanical properties.

3.1 Materials and fabric characterization

3.1.1 Microscopy

SEM micrographs of the fresh epoxy-impregnated fabric decorated with various particles showed that in the CEMf system (Table 2) a dense and uniform cement powder decoration had formed over the surface of the fabric (which was therefore completely hidden; Fig. 6a, upper panel). In all the other decorated systems, the fabric was only partially decorated with the particles, and naked filaments were clearly visible (Fig. 6b-d). In the SFf and f-CNTf systems, the particles were aggregated (Fig. 6b, d). The separation of the filaments in the yarn and the formation of voids were clearly observed in the GOf system (Fig. 6c), most probably due to the spray coating approach used in the decoration (Sect. 2.3). The same coating approach was also used for the f-CNTf system; however, due to the relatively smaller mean lateral dimensions of the nanotubes compared to GO (Table 1), the separation of the filaments was less pronounced (Fig. 6d).
Examination of the CEM-decorated epoxy in the fresh and gelated states revealed that the cement particles were partially embedded in and not completely covered by the epoxy coating in both cases, allowing a clear particle surface to react with the mortar matrix during the hydration process. The fresh epoxy systems (CEMf) were densely coated with cement particles (Fig. 6a, upper panel), while only a few scattered particles were found in the gelated epoxy systems (CEMg, Fig. 6a, lower panel). The reason for this is the relatively high crosslinking of the epoxy, which led to increased viscosity. In addition, when powder coating was used, the gaps between the filaments were completely filled with epoxy (Fig. 6a, lower panel and Fig. 6b) for both fresh and gelated samples, indicating strong contact between the filaments within the yarn, which would then act as a single reinforcing unit.

The density of the decorating particles is expected to substantially affect the bond strength in the TRC element. It is clearly shown that the density of particle decoration over the epoxy-coated fabric was higher for the micron-scale powder-coated particles (CEM and SF) than for the nano-scale spray-coated particles (GO and f-CNT, Fig. 6). Moreover, it was found that the gelation time of the impregnated epoxy also affected the density of the decorating CEM particles where lower viscosity of the epoxy (CEMf, Fig. 6a upper panel) led to higher decoration density compared to higher viscosity epoxy (CEMg, Fig. 6a lower panel).

### 3.1.2 Elemental analysis

The oxygen concentration for each decoration type (i.e., GO, f-CNT, CEM and SF) served as a measure of its hydrophilicity and compatibility in the mortar matrix. It has been shown that the carboxylic acid groups in GO [25] and f-CNT [36] interact with the calcium-silicate-hydrate or Ca(OH)₂ of the cement during the cement hydration reaction. Therefore, the oxygen concentration was measured by EDS for all decoration types as well as for the carbon fabric (Table 3; an example of an EDS spectrum is provided in Fig. S2, supplementary information) in an attempt to correlate it with the fabric–matrix bond strength. The EDS measurements of the powders indicated that f-CNT exhibited the lowest oxygen content (5%), followed by CEM (25%) and GO (34%), while SF displayed the highest oxygen content (55%).

| Element | f-CNT | CEM | GO | SF | Carbon fabric |
|---------|-------|-----|----|----|---------------|
| C       | 95    | 0   | 66 | 0  | 100           |
| Si      | 0     | 18  | 0  | 45 | 0             |
| O       | 5     | 25  | 34 | 55 | 0             |
| Ca      | 0     | 57  | 0  | 0  | 0             |

Values in the Table are given as %

Another parameter that may affect the fabric–matrix bond strength is the type of reaction between the decoration and the matrix. The decorated CEM particles are expected to react with the cementitious matrix via the hydration process [19], while the SF particles (active silica) will likely interact with the hydrated calcium hydroxide products (Ca²⁺ ions) via a pozzolanic reaction [14, 16].

### 3.2 Pull-off behavior

The fabric–mortar bond strength (adhesion) was measured by a pull-off test (Figs. 4 and 5) and compared to the NE-impregnated fabric as a reference (Fig. 7). The NE–mortar bond strength was 1.3 MPa, in keeping with the literature [4, 9]. Note that the plain fabric (without epoxy impregnation) exhibited slightly higher (by 4%) bond strength than that of the NE system (Fig. 7). These results do not contradict the principle of coating the fabric with epoxy to obtain one-unit yarn. It is known that coating the yarn with epoxy prevents telescopic pull-out [48]; however, in the pull-off test, the force was applied perpendicular to the fabric and hence the higher roughness of the uncoated fabric (naked filaments) compared to the epoxy-coated fabric could have produced some anchoring in the cementitious matrix.

It is clearly demonstrated below that all the parameters tested in this study, namely, the type and size of the decorating material (CEM, SF, GO or f-CNT), the viscosity of the epoxy (fresh vs gelated) and the decoration application approach (spray or powder coating) have a crucial effect on the macroscopic properties of the TRC element, i.e., the bond strength (Fig. 7).
Decoration with cement particles yielded higher relative bond strength (compared to NE) in both fresh and gelated epoxy states, with enhancements of 25% and 8%, respectively (Fig. 7b; CEMf and CEMg). A similar trend was found for the SF decoration (13% and 2% enhancement for SFf and SFg, respectively). The enhancement in these cases was likely due to a chemical reaction of the decorating materials with the cement-based matrix products through hydration and pozzolanic reactions in the CEM and SF systems, respectively, as elaborated in Sect. 3.1.

The SF decoration reacted with the hydration calcium hydroxide products that are frequently formed at the fabric–matrix interface due to a bleeding process [19]. The CEM decoration yielded a stronger bond strength than the SF decoration for both fresh and gelated epoxy-impregnated fabric systems, suggesting that the contribution of the hydration reaction to the bond strength was greater than that of the pozzolanic reaction. We note that the differences between the findings for the CEM and SF systems could stem not only from the different nature of the bonding reactions but also from the higher decoration density in the CEM system compared to the SF system (e.g., in the fresh state; Fig. 6a top panel vs Fig. 6b).

The curing state of the epoxy (fresh vs gelated) affected the stickiness of the decoration, where higher bond strength values and consequently higher decoration density were obtained for the fresh epoxy systems (CEMf > CEMg and SFf > SFg; Fig. 7). The decorating particles were strongly attached to both the fabric (through the epoxy) and the cement matrix (through chemical reaction), resulting in high bond strength. Note that for the CEM and SF systems, decorating gelated (rather than fresh) epoxy did not offer any significant enhancement in the relative bond strengths for both CEMg and SFg systems. Therefore, it could be concluded that powder decoration should be conducted on fresh epoxy to obtain higher decoration density and consequently higher bond strength.

Decoration with GO and f-CNT

The GO and f-CNT systems showed the opposite behavior to that of the CEM and SF systems in that decoration with GO and f-CNT on fresh-state epoxy (GOf and f-CNTf systems) caused the relative bond strength to deteriorate by 15–20%, while decorating the gelated-state epoxy enhanced the relative bond strength.
strength of the f-CNTg system by 12% (Fig. 7). The deterioration in bond strengths for the fresh state systems (GOf and f-CNTf) may be related to the aggressive spraying approach used during composite preparation, in which it was found that the pressure (∼ 4 bars) applied to the surface of the fabric probably damaged the epoxy filling between the filaments of the bundle yarn. Moreover, the ethanol solvent used in the spray coating would have interacted with the fresh epoxy [49], causing its properties and hence the bond strength to deteriorate. The spray-coated fabrics exhibited voids and gaps inside the bundle, as clearly shown in Figs. 6c and d. This damage was more pronounced for the GOf system, most probably due to the larger lateral size of the GO particles compared to the size of the f-CNT particles. In the GOf system, most of the pores (∼ 50 μm) were blocked either by the GO particles or epoxy (Fig. 6c), which impaired the bonding with the mortar matrix. The reduction in bond strength was somewhat higher for the f-CNTf (20%) than for the GOf (15%) system, despite the demonstration of fewer defects (Fig. 6d compared to Fig. 6c). A plausible explanation lies in the higher hydrophilicity of GO compared to that of f-CNT (34% vs 5% oxygen concentration, as measured by EDS; Table 3), which led to stronger chemical affinity of the GO with the mortar matrix, thereby compensating for the damaged fabric effect. The sprayed nanoparticles on fresh state epoxy deteriorated the bond strength compared to NE system due to agglomerated particle decoration and formation of voids between the carbon fabric filaments.

On the other hand, when gelated epoxy (crosslinked for 90 min) was sprayed, the applied high pressure did not damage the partly hardened epoxy (not shown), and therefore fewer voids were found, resulting in higher bond strength of the spray-coated gelated epoxy (f-CNTg and GOg) compared to the fresh epoxy (f-CNTf and GOf) systems (Fig. 7). The f-CNTg system showed the highest relative bond strength enhancement among the epoxy-gelated systems (12%) due to the anisotropic morphology of the nanotubes and the bridging they induced [18] at the fabric-matrix interface. Such a configuration would be less likely for the GO particles, due to their higher lateral dimensions (Fig. 6c). Indeed, it was shown that incorporation of anisotropic decoration into TRC enhanced its mechanical properties [50] through bridging at the fabric-matrix interface, thereby offering more contact points and hence facilitating stronger bonding. Moreover, it was shown that CNT incorporation on the surface of fibers healed surface defects and enhanced their mechanical properties [51], as may have occurred in the present systems.

To summarize, the decoration in the CEMf and SFf systems via powder coating was less aggressive than spray coating (Sect. 2.3). Consequently, fewer voids were formed in the fabric coating (Fig. 6a, b) leading to superior bonding strength of the CEMf and SFf over the f-CNTf and GOf systems (Fig. 7). The CEM and SF decorations underwent hydration and pozzolanic reactions, respectively, with the mortar matrix (Sect. 3.1), while the carbon-based particles did not. These findings insinuate that a simple powder coating approach yields high bond strength (25% enhancement of the CEMf over NE, Fig. 7) and could be more attractive to civil engineering applications than spray coating. Since the CEM-based systems yielded the best performance, we further explored their microstructure at the fabric–matrix interface.

3.2.3 Pull-off vs pull-out

Pull-out tests are commonly used in the cement reinforcement field to measure fabric-matrix friction-based bonding, which are mainly related to mechanical anchoring [52, 53]. In this study, we aim at enhancing the fabric-matrix chemical adhesion that prevents delamination—a highly problematic process in structural applications. This value of the chemical adhesion is accurately and directly provided by pull-off test, while pull-out yields only an estimation based on the chemical debonding energy. A comparison between the chemical adhesion (measured by pull-off, Fig. 7a) and chemical debonding energy (calculated from our pull-out results, a detailed description of the pull-out test is provided in section S1.1, supplementary information) [46] suggests that they are indeed correlated for all tested systems (Fig. 8). For both pull-off and pull-out tests, the best adhesion is obtained for the CEMf system, validating the pull-off approach for the measurement of adhesion in TRC systems.

3.3 Microstructure of the CEM systems at the fabric–matrix interface

We studied samples of the CEMf and CEMg systems by SEM before (interfacial cross-section) and after
(fractured surface) the pull-off test with the aim to explore the effects of decoration with cement particles and of gelation time on the micro-structure of the TRC systems. An analysis of the micrographs provided the correlation between the bond strength (Fig. 7) and the micro-structure of the TRC element.

3.3.1 Micro-structure of the TRC element before pull-off

The adhesion mechanisms of the CEMf and CEMg (compared to NE) systems were explored by imaging the interfacial cross-section of the TRC samples by SEM as depicted in Fig. 5 (red rectangle), in which the contrast between the TRC components was very distinct, namely, bright for the cement-based matrix and dark for the epoxy-impregnated carbon fabric (Fig. 9).

In the cross-section SEM micrograph of the reference system (NE-impregnated fabric), the layers of the mortar matrix and the carbon fabric were clearly discernible (Fig. 9a). A closer examination of the impregnated yarn revealed a uniform epoxy filling in between the filaments comprising the yarn (Fig. 9a, circle). Interestingly, a different arrangement of layers was observed for the CEM system, in which an interlayer (~ 100 μm in thickness, Fig. 9b) had formed at the interface between the epoxy-impregnated fabric and the mortar layers. Elemental analysis of the interlayer (EDS, Table S1 and Fig. S4) showed mainly carbon atoms, which were driven from the epoxy coating around the yarn perimeter (applied during impregnation), as well as calcium, silicon, alumina and iron, indicating the presence of unhydrated CEM particles in the interlayer. These findings attest to the strong attachment of unhydrated CEM particles–located at interlayer surface near the mortar matrix–to the epoxy coating. These unhydrated cement particles reacted with the water in the mortar matrix during the hydration process forming a layer of dense hydration products and thereby a tight, void-free connection between the interlayer and the mortar matrix (Fig. 9b).

The above-described three-layer arrangement at the microscopic level resulted in higher adhesion bonding between the epoxy-impregnated fabric and the cement-based matrix for the CEMf system vs the other decorated systems (Fig. 7). An interlayer was not found in the cross section of the CEMg system (Fig. 9c), in which the decoration density of the cement particles was lower (Fig. 6a, lower panel), and consequently a weaker bond strength than that of the CEMf system was measured (Fig. 7). The absence of the interlayer in the CEMg sample (Fig. 9c) might stem from the low-density coating of the cement particles (Fig. 6a, lower panel) compared to that of the fresh epoxy (CEMf, Fig. 6a, 8).
and provides a microstructural evidence that the gelation time (i.e. epoxy viscosity) plays a key role in the bond strength enhancement.

### 3.3.2 Micro-structure of the TRC element after pull-off

Visual examination of the fractured surface after the pull-off test (Fig. 4f) suggested that the failure occurred at or very close to the fabric-matrix interface for all the samples. SEM imaging of the NE system showed a thin layer of cementitious matrix that partly covered the fabric (Fig. 10a, b), leaving the majority of the filaments exposed. This indicated that in the NE system the failure had occurred mostly at the fabric-matrix interface, i.e., suggesting relatively weak interfacial bond strength. The CEMf system demonstrated dense, uniform and almost complete fabric coverage by the cementitious matrix, with hardly any filaments being visible (Fig. 10c, d), thereby implying that the failure had occurred far from the fabric-matrix interface, where bonding was strong. For the CEMg system, the fraction of fabric covered by the cementitious matrix was lower than that of the CEMf system (Fig. 10e, f), suggesting a weaker fabric-matrix bond. Both weft and warp yarns of the fabric in the CEMf system were uniformly coated (Fig. 10c), while in the CEMg system, most warp yarns are exposed (Fig. 10e), highlighting again the importance of performing the cement particle decoration in fresh state of the epoxy-impregnated fabric.

The CEMf system demonstrated higher coverage of the cementitious matrix after pull-off than the CEMg system (Fig. 10), in keeping with the higher decoration density of the cement particles on the fabric surface before casting (Fig. 6a, upper panel). These SEM micrographs suggest that the presence of this dense CEM decoration led to a massive hydration with the cement matrix, on the one hand, and strong bond strength with the epoxy, on the other. These observations were consistent with the measured bond strength trend: CEMf > CEMg > NE (Fig. 7).

### 3.4 Effect of chlorides on the fabric–matrix bond strength

Since steel bar reinforcements are usually corroded at high salt concentrations [54], it was important to evaluate the influence of an aggressive high-salt aqueous environment (3 M NaCl) on the fabric–matrix bond strength. To this end, CEMf and NE systems were submerged in an aqueous 3 M NaCl solution (at 23 °C) for 12 days before the pull-off tests were performed. Unexpectedly, in both TRC systems, the fabric-matrix bonding strength was significantly enhanced after exposure to the NaCl solution compared to similar systems exposed to ambient conditions (23 °C and 60% RH, Fig. 11), with an average bond strength enhancement of 80% and 100% for NE and CEMf, respectively. This enhancement was attributed to the growth of Na-based salt crystals at the fabric–matrix interface (Fig. 12, arrow), as supported by EDS measurements (Table S2, supplementary information). These crystals filled the gaps at the fabric–matrix interface, which led to a more compact and intimate interface endowing improved bonding. Our findings thus revealed that a NaCl environment was favorable for TRC applications, since it strengthens the fabric–matrix bonding, in contrast to steel-reinforced concrete, which tends to corrode.
The CEMf system yielded a higher bond strength than the NE system, by \( \sim 0.3 \) MPa, for both ambient and NaCl environments, with the CEMf exposed to the aqueous NaCl environment yielding the highest adhesion between the matrix and the fabric compared to all other examined systems, thereby indicating that the NaCl environment does not affect or damage the fabric surface treatment (Fig. 11).

It should be mentioned, that unlike the test conditions, construction elements in coastal areas are exposed not only to a chloride environment but also to other factors, such as rain, that may reduce or dissolve the salt crystals formed during the exposure to chlorides. However, our results suggest that the integration of carbon fabric reinforcement in elements submerged in salt water (e.g., floating pontoons) could be highly effective.

### 3.5 Effect of the decoration on the flexural strength of TRC elements

The global effect of the fabric decoration on the macroscopic properties of a TRC element (e.g., flexural properties) loaded with CEMf (superior bonding properties) was studied. NE and plain matrix (REF, without fabric) were used as reference systems.

During flexural loading of a TRC beam, the brittle cement matrix failed first (\( \sigma_1 \) and \( \varepsilon_1 \) indicate the flexural stress and strain at the matrix failure, respectively; Fig. 13), while the fabric continued to bear the applied load until it also failed (at \( \sigma_2 \) and \( \varepsilon_2 \), Fig. 13) [55]. We used these four parameters to analyze the flexural properties of the three systems, namely, CEMf, NE, and reference (REF), as summarized in Table 4.

The CEMf-decorated TRC beam exhibited the highest \( \sigma_1 \), with an enhancement of 30\% compared to the REF system, while the \( \sigma_1 \) of the NE TRC system was similar to that of the reference system (Table 4).

![Fig. 10 SEM micrographs of the fractured surface samples after pull-off (Fig. 4f):](image-url)
These findings indicate that the fabric–matrix bond strength affected the macroscopic properties, such as the stress at the first crack \( [14, 15] \): Stronger bond strength (as indicated by the pull-off tests, Fig. 7) indeed yielded higher \( r_1 \) values. The value of \( r_2 \) in the CEMf system was higher than that of the NE system, which can also be related to the high adhesion bond strength of the former. The highest \( e_2 \) obtained for the CEMf-decorated TRC, as shown in Table 4, again revealed the importance of decoration. For both TRC beams (CEMf and NE), the value of \( e_2 \) was threefold higher than that of \( e_1 \), indicating that the reinforced fabric improved the ductility of the TRC, as expected.

These results showed the benefits of decorating the epoxy-impregnated fabric with CEM particles, which yielded the strongest adhesion and consequently improved macroscopic mechanical properties of the TRC.

In summary, this study clearly revealed significant differences between the adhesion properties as a function of the type of decoration, the gelation time, and the coating method, thereby highlighting the micro-structure differences between the different decorations and the high potential of carbon fabric reinforcement in coastal regions. In TRC, the reinforcing fabric is located very close to the surface of the element (a few millimeters), as opposed to much thicker protection layer (25–80 mm \([56]\)) for steel rebars, again demonstrating the utility—in terms of the quantity of concrete required—of replacing steel rebars with carbon fabric.

**Table 4** A summary of the flexural properties and their standard deviations (\( r_2 \) and \( e_2 \) cannot be calculated for the REF system, since it is fabric free)

|           | REF | NE  | CEMf |
|-----------|-----|-----|------|
| \( \sigma_1 \) [MPa] | 5.4 ± 0.7 | 4.7 ± 0.5 | 7 ± 2 |
| \( \sigma_2 \) [MPa] | – | 14 ± 2 | 17 ± 3 |
| \( e_1 \) | 0.011 ± 0.002 | 0.007 ± 0.001 | 0.011 ± 0.003 |
| \( e_2 \) | – | 0.021 ± 0.004 | 0.028 ± 0.003 |

**Fig. 11** Bond strength of NE and CEMf systems upon exposure to ambient environment and to a 3 M NaCl solution

**Fig. 12** SEM micrograph of a cross section of a TRC (NE) sample that was submerged in an aqueous 3 M NaCl solution. Arrow points to Na-based salt crystals formed between the epoxy impregnated carbon fabric and the cement-based matrix. EDS analysis at points 1 and 2 are provided in Table S2

**Fig. 13** Representative flexural curve of TRC (NE). The brittle matrix failed first (\( \sigma_1 \) and \( e_1 \) indicate the flexural stress and strain at the matrix failure, respectively), while the fabric continued to bear the load applied until element failure (at \( \sigma_2 \) and \( e_2 \)).
4 Summary and conclusions

Fabric decoration of cement powder markedly enhanced the interfacial chemical bonding between the fabric and the cementitious matrix in TRC systems. The decoration with cement powder over fresh epoxy-impregnated fabric (CEMf) yielded a 25% enhancement in the adhesion bond strength compared to a non-decorated system. The efficacy of the various tested decorations (CEM, SF, f-CNT, GO) depended on: (i) the decoration density on the epoxy-impregnated fabric, which depended on the viscosity of the epoxy, (ii) the reaction type with the cementitious matrix—pozzolanic vs. hydration—and particle decoration hydrophilicity, and (iii) void formation in the fabric during the decoration process reliant on the application methods. The morphology of the fabric–matrix interface in the CEM-decorated system that exhibited the strongest bond strength (CEMf) included an interlayer, consisting of epoxy and un-hydrated cement particles, between the matrix and the fabric. These un-hydrated cement particles reacted with the cement-based matrix during the hydration process. The interlayer (which was absent in the other studied systems) enhanced the fabric–matrix bond strength, as manifested in superior values of pull-off and flexural strength of the resulting TRC.

Exposure to a high salt concentration (typical of coastal areas) resulted in an unexpected enhancement of the fabric–matrix bond strength relative to ambient conditions (80% and 100% increase for the NE and CEMf systems, respectively) due to crystallization of salt at the fabric–matrix interface and the consequent sealing of voids.

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