Hydration and Microstructure of Paste Cement and Textile Reinforced Concrete Composite (A Review paper)

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Abstract

Recently, many types of concrete that are superior to ordinary concrete have appeared, including textile reinforced concrete (TRC). To improve the mechanical behavior of cementitious composites, however, using continuous fibers instead of short fibers in the primary direction of tension is a far more successful strategy. Lately, a solution to the issue of how to make reinforced concrete more sustainable was offered by the non-corrosive textile material that is used as reinforcement to the finely-grained concrete matrix. Furthermore, it has various benefits including corrosion resistance and a thin, light structure. The high strength-to-weight ratio, durability, speed of installation, and thickness decrease beyond 50 mm are further advantages of textile composites. This research aims to compare the microstructures of cement matrix and textile reinforcement concrete from the effect of hydration products on microstructure and the interface transition zone (ITZ), and understand the bonding characteristics between cement matrix and multi-filament yarn structures. The interfacial transition zone (ITZ) of the TRC and cement based matrix was characterized using scanning electron microscopy (SEM) and energy-dispersive X-ray (EDX) spectroscopy. The connection between the texture of the textile and cement mortar is inadequate in terms of the filament's surface properties and sizing material. According to this research, the structure and sizing impact of AR glass textile are more complicated than that of rebar reinforcement.

Keywords: Textile; Cementitious Matrix; Microstructure; Interface Transition Zone ITZ; Hydration Products.

الترطيب والبنية التحتية الدقيقة لمونة الأسمنت ومركبات الخرسانة المسلحة بالنسيج (ورقة مرجعية)

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الملخص

في الآونة الأخيرة، ظهرت أنواع كثيرة من الخرسانة التي تتفوق على الخرسانة العادية، بما في ذلك الخرسانة المسلحة بالنسيج، أو ما يسمي بالانجليزية Textile Reinforced Concrete (TRC) ومع ذلك، لتحسين السلوك الميكانيكي للمركبات الأسمنتية، فإن استخدام الألياف المستمرة او النسيج بدلاً من الألياف القصيرة في للشد، يعد استراتيجيا أكثر نجاحًا. في الآونة الأخيرة، تم الوصول الي كيفية جعل الخرسانة المسلحة بالنسيج أكثر استدامة من خلال استخدام مواد غير مسببة للتآكل والتي يتم استخدامها كتعزيز للمونة الإسمنتية. علاوة على ذلك، هذه الخرسانة تتمتع بفوائد مختلفة بما في ذلك مقاومة التآكل، هياكل ضعيفة السمك، خفيفة الوزن. تعتبر نسبة المقاومة/الوزن العالية، المتانة، سرعة التركيب، وانخفاض السمك بما يتجاوز 50 ملم للسقف من المزايا الإضافية لمركبات النسيج.

يهدف هذا البحث إلى مقارنة البنية التحتية المجهريه للمونة الأسمنتية والخرسانة الإسمنتية المسلحة بالنسيج، من ناحية تأثير منتجات الترطيب على البنية المجهريه والمنطقة الانتقالية البينية(ITZ) ، وفهم خصائص الترابط بين المونة الأسمنتية و النسيج متعدد الشعيرات عند هذه المنطقة. تم تمييز المنطقة الانتقالية باستخدام المجهر الإلكتروني الماسح (SEM) والتحليل الطيفي للأشعة السينية المشتتة من الطاقة(EDX), العلاقة بين النسيج والمونة الأسمنتية ليست سهلة الفهم لأنها تعتمد بالأساس علي خصائص سطح الشعيرة، و التركيب الكيميائي لمادة البولبمر المغلفة للسطح أثناء عملية التصنيع. وفقًا

لهذا البحث، فإن هيكل النسيج الزجاجي المقاوم للقلوية أكثر تعقيدًا من تأثير تقوية حديد التسليح الاعتيادي.

الكلمات المفتاحية: النسيج، المونة الإسمنتية، البنية المجهريه. المنطقة الانتقالية البينية (**ITZ**)، منتجات الترطيب.

Introduction

Concrete is a composite material that consists essentially of a binding medium within which are embedded particles or fragments of aggregate. In hydraulic cement concrete, the binder is formed from a mixture of hydraulic cement and water [1].

Microstructural characteristics such as the interfacial transition zone (ITZ) and cracking patterns from compressive strength testing are main features that characterize concrete behavior. Certain materials such as blast furnace slag or fly ash introduced in the concrete mix aid in improving its strength and durability. While others, such nanosilica particles, might only have a little impact on the paste's microstructure, they wouldn't significantly strengthen the paste-aggregate link or ITZ [2]. According to Durta, 2020 [3], the first and most important stage in controlling the microstructure and its effect on performance is to comprehend the behavior of cement concrete at these minuscule scale levels. Furthermore, studying the microstructures of fresh cement paste in situ can significantly advance our understanding of how concrete develops early on (such as setting and hydration), which can assist to improve the quality of concrete [3]. Common technologies such as Scanning Electron Microscope (SEM) are currently employed in petrographic analysis of cementitious materials and concrete microstructure. In microstructural terms, concrete is an extremely complex system of solid phases, pores, and water, with a high degree of heterogeneity [4]. The microstructure that forms throughout the concrete's mixing, setting, and hardening processes will have an immediate impact on its final characteristics and functionality. In addition to cement hydration, the homogeneous dispersion of aggregates, mineral

admixtures, and cement particles controls microstructural development [5].

ITZ, which surrounds large aggregates in normal concrete as a thin shell typically 80 to 510 μm thick, and it occupies about 30 to 50% of total volume of cement paste in normal concrete; is much more influential on the mechanical behavior of concrete. ITZ is typically weaker than either of the two main components of concrete, namely the aggregate and the bulk hydrated cement paste [6]. Each aggregate particle may have microcracks, voids, and several minerals. In same way, the interfacial transition zone and bulk hydrated cement paste often have a heterogeneous distribution of various kinds and quantities of solid phases, pores, and microcracks [7]. Concrete's microstructure is not an intrinsic property of the material since its two constituent parts; the hydrated cement paste and the interfacial transition zone are subject to temperature, humidity, and environmental changes throughout time.This work aims to study the effects of hydration compounds on the microstructure and interface transition zone (ITZ) and analyze the bonding characteristics between multi-filament yarn structures and cement matrix using energy-dispersive X-ray (EDX) spectroscopy and scanning electron microscopy (SEM).

Composite Material
a. Normal mortar

Mortar is a mixture of sand, cement, and water [8].Usually usedhydraulic cement and called cement is called hydraulic when the hydration products are stable in an aqueous environment. The most used hydraulic cement for making concrete is *Portland cement*, which consists essentially of reactive calcium silicates; the calcium silicate hydrates formed during the hydration of Portland cement are primarily responsible for its adhesive characteristic. Cement is a finely pulverized, dry material that by itself is not a binder but develops the binding property as a result of hydration (i.e., from chemical reactions between cement minerals and water) [7]. Fine aggregate refers to aggregate particles smaller than 4.75 mm. The term sand is commonly used for fine aggregate resulting from natural disintegration and abrasion of rock or processing of

friable sandstone [9]. Distilled, or deionized, water must be used for validation tests. For other tests, drinking water can be used. In case of dispute, distilled or deionized water should be used. Chemical admixtures can alter the cement paste's setting and hardening properties by affecting the cement hydration rate. Admixtures that reduce water tension can plasticize newly mixed concrete mixtures, can lessen thermal cracking in mass concrete [10].In the first 24 hours, mortar usually cures to 60% of its ultimate compressive strength. After then, it will take around 28 days for the treatment to reach its maximal strength.

b. Material Properties of TRC

The AR glass textile technique is adhered to by the composition of the matrix employed. Primarily, pozzolanic additives and blast furnace cement are utilized as binding agents. Within the context of earlier research programs, extensive experience with these fine-grained concrete matrices has already been obtained [11]. Typically, the composite material used in regular mortar may be utilized in TRC as long as the fine aggregate size is limited to 2 mm. This will prevent the filaments from being harmed during the casting process and make it easier to fill the space between the filaments.

By adopting a small maximum grain size of $d_{max} = 0.6$ mm, high binder concentrations, and the addition of various pozzolanic additives and plasticizers, the unique features of the fine-grained concrete matrices, such as extremely flowable consistencies, are produced [12]. Furthermore, the matrices are constructed with an enhanced stability of AR-glass rovings in mind. This improvement in stability is achieved by using the right binder systems, which minimize the amount of OPC clinker content by using natural or synthetic pozzolanic materials, including fly ash and silica fume, in place of blast furnace slag. Silica fume can be used to significantly lower the calcium hydroxide content and the alkali ion concentration in the pore solution [13]. Lieboldt, et al., 2006 [14] concluded that the allowed ingredients for the fine-grained concrete mix are 1 MP (mass parts) of water, 3 MP of binding agent, and 3 MP of fine sand with a maximum grain size of 2 mm. 1 MP of pozzolanic additive and 2 MP of cement CEM III/B 32.5 N make up the binding agent.

The pozzolanic share consists of 90 % fly ash and 10 % microsilica (solid matter). This exemplary composition has to be modified using different additions to fit the requirements of the hardened concrete qualities and the manufacturing process. As a result, the fresh concrete's plasticity will offer excellent stability. Brameshuber et al. 2006 [15] provide into depth of essential mechanical characteristics, whether they are fresh or hardened.

Mechanism of Hydration

Heat is released when water is added to Portland cement; how much and at what pace depends on the cement's composition. The four main Portland cement compounds—$3CaO.SiO_2$, $2CaO.SiO_2$, $3CaO.Al_2O_3$, and $4CaO.Al_2O_3.Fe_2O_3$—have their temperatures of full hydration calculated [16].

The hydrated product's amount, quality, stability, continuity, and formation rate are all crucial. Mill grinding can break up the calcium silicates and crystals in the interstitial phase, which changes the microstructure of the cement. Depending on the situation, it can also change the type of calcium sulfate that is supplied at this point to regulate the cement setting [17]. The parameters of cement hydration are subsequently influenced by these aspects. There are two distinct techniques to classify cement hydration, "through solution" and "solid-state," may operate concurrently when cement and water react.23% of the water is chemically combined with cement, so it is called bound water [18].A certain amount of water is sucked into the gel pores. This water is called gel water. It can be said that bound water and gel water are complementary. If the amount of water is not enough to fill the gel pores, the formation of the gel itself will stop, and if the formation of the gel stops, there is no problem with the gel pores. It is further estimated that about 15% by weight of cement is required to fill the gel pores.

a.   In normal mortar

Hydraulic properties of the cement constituents, that they form compounds by reaction with water stable hydrated with very little solubility in water; pozzolanic properties, have the ability to form at ordinary temperature, in the presence of water, by combination with

lime, stable, hydrated compounds; physical properties that improve certain qualities of cement (increase in workability and compactness, reduction of bleeding, etc.). The water/cement ratio also has a great influence on the porosity of the matrix. For a lower W/C the capillary porosity is in fact made up of a finer and more discontinuous pore network [19].Hearn et al [20] studied the effect of water to cement (W/C) ratio on the advancement of hydration. The results showed that low values of the W/C ratio lead to a slowing of hydration reactions at a young age. A high concentration of C-S-H is obtained more quickly when the quantity of pore water is low. Beyond this period, a drop in W/C produces the opposite effect. According to Bouny 1994 [7], the size of the pores is all the smaller as the W/C ratio is low.

### b. Mineralogy of the cement paste matrix and hydrate morphology

Several study focused mainly on the anhydrate phases, on the hydrates $Ca(OH)_2$ and C-S-H and to a lesser extent on ettringite and $CaCO_3$. The distinction of materials with scanning electronic microscopy (SEM) is based on the differences in reflective power, (see Figure 1 and 2). In this technique, a slight attack by HNO_3, after polishing these samples, causes the coloring of C_3S, C_2S, $Ca(OH)_2$ and to a lesser extent that of C-S-H and makes it possible to clearly differentiate on these images [7]. Moreover, the anhydrous phases (residual clinker): the C_3S grains (alite) appear yellow and often hexagonal C_2S grains (belite) appear brown, rounded and streaked (twins) and $C_3A + C_4AF$ appears as a whitish area possibly spotted with black. Hydrates consisting essentially of C-S-H constitute the predominantly beige background which appears in the images. In ordinary materials, Portlandite $Ca(OH)_2$ appears in the form of very jagged green or brown spots (archipelagos). It appears in the form of bluish beams in the materials and the voids (cracks, air bubbles or pores in the paste) appear as black areas. The cement paste and concrete are almost hydrated, we only see C_3S grains under the microscope in exceptional cases, and, instead find a few crystals of C_2S and C_4AF which are slower to hydrate these materials have a very complex structure (numerous hydrates and

very diverse morphologies needles of ettringite, monosulfo aluminates clusters of platelets of Ca(OH)$_2$ various morphologies of C-S-H.
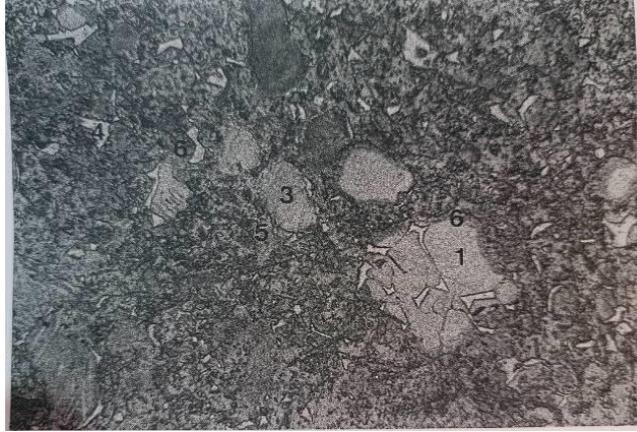
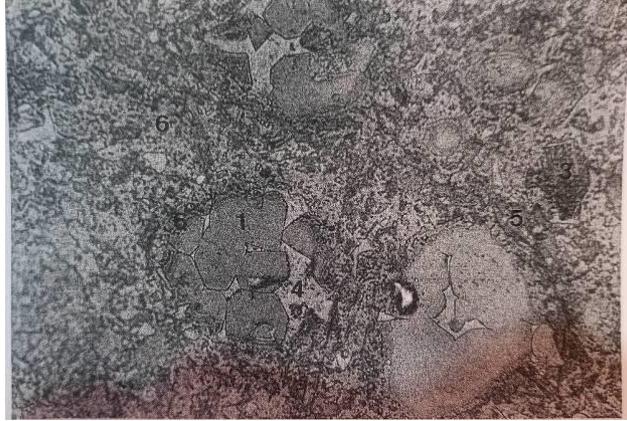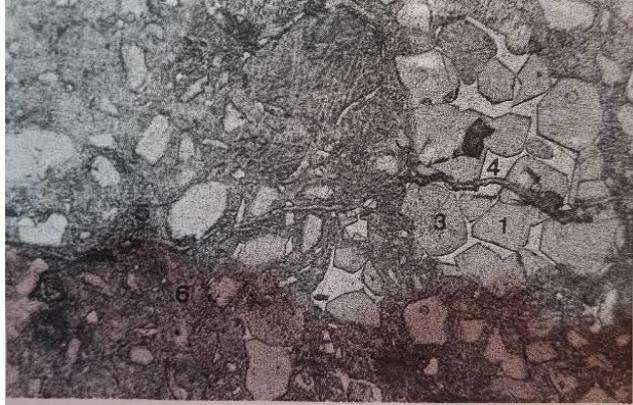Cement Paste (Pcd), (CO), W/C = 0.348
1. Tricalcium silicate ; 3. Dicalcium silicate; 4. Tricalcium Aluminate + Tricalcium Aluminoferrite ; 5. Portlandite; 6. CSH, calcium silicate hydrates.
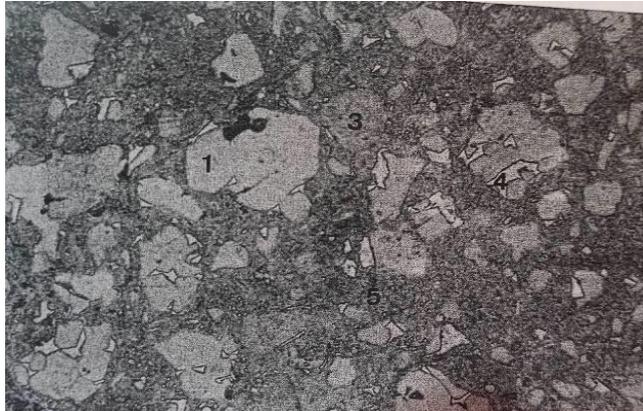
Cement Paste (Pcd), (CH), W/C = 0.196
1. Tricalcium silicate ; 3. Dicalcium silicate; 4. Tricalcium Aluminate + Tricalcium Aluminoferrite ; 5. Portlandite; 6. CSH, calcium silicate hydrates.

Figure 1.Optical microscope (x500 magnification), samples aged 2 years and stored at RH = 100% until the processing date [7].

Cement Paste (Pcd), (C0), W/C = 0.196
1. Tricalcium silicate ; 3. Dicalcium silicate; 4. Tricalcium Aluminate +
Tricalcium Aluminoferrite ; 5. Portlandite; 6. CSH, calcium silicate hydrates.

Cement Paste (Pcd), (CH), W/C = 0..196
3. Tricalcium silicate ; 4. Tricalcium Aluminate + Tricalcium Aluminoferrite ; 5.
Portlandite; 6. CSH, calcium silicate hydrates.

Figure 2. Optical microscope (x600 magnification), samples aged 2
years and stored at RH = 100% until the processing date [7].

From previous Figures, Portlandite crystals often take the shape
of considerably finer platelets that more closely resemble lamellae
and develop in platelets in porous regions where they are likely to
find space to crystallize. According to [7], the mass of $Ca(OH)_2$ per
anhydrous cement unit mass (%) in ordinarycement or ordinary

concrete is 24 and 25, respectively aged 6 months and stored at RH = 100%, T = 21°C. Since ordinary cement has low initial water content, ionic diffusion within the material is really highly slowed down, resulting in the hardened cement paste being incompletely hydrated throughout time. Because of this, ordinary cement actually ages very slowly. It is evident from the data that standard cement paste or concrete materials have similar and traditional $Ca(OH)_2$ concentrations.

Porous distribution in cement paste that has hardened it's crucial to keep in mind that, despite the network's intricacy and the irregularity of the pores' shapes, the sizes determined by mercury porosimetry correspond to openings (pore access diameter).

When using an additive, such as silica fume, in a mortar where W/C = 0.42 when a portion of the cement is substituted with silica fume. Pore dispersion on these mortars after 28 days, kept in water, the intergranular areas where the external hydrates develop and where the liquid phase is first found. Then, the paste becomes densified and access to porosity reduces due to the pozzolanic reaction between $Ca(OH)_2$ and silica fume, which produces more C-S-H.The porosity as determined by the mercury test declined with age (or the degree of hydration) in the two situations that were described as shown in figure 3 and 4, yet these hydrates, which are porous products, formed.
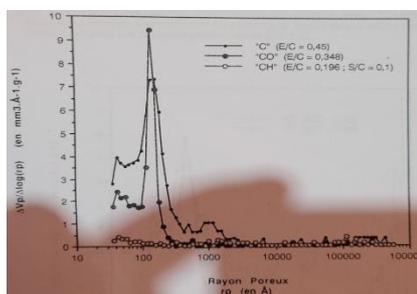

Figure 3. Porosity distribution for hardened cement paste aged 1.5 years, W/C = 0.42 determined using mercury porosimetry [7].
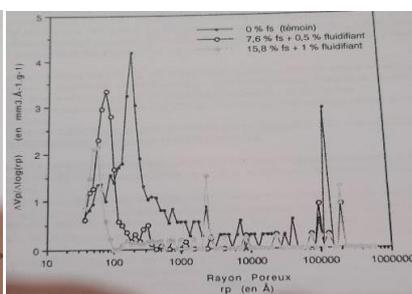

Figure 4.Porosity distribution for hardened cement paste aged 28 days, W/ (C+S) = 0.45 determined using mercury porosimetry [7].

c. In TRC

Characterizations of the penetration using the scanning electron micrographs of cross sections of AR-glass rovings embedded into cementitious matrix with different linear mass density expressed in tex : cementitious matrix with different linear mass density expressed in tex : 320 tex; 640 tex; 1200 tex and 2400 tex [21]. In homogenous interface interspersed with ettringite, structural conditions in the interface are also governed by yarn sizing (composition scarcely may be modified by the user), as shown in figure 5, 6. Protecting the yarn during the looming process is the primary purpose of sizing. Only when the size material has high adhesion qualities that is, when there is a significant force of attraction between the fiber and the size material can that occur. These forces are often hydrogen bonds formed by Van Der Waals. Since hydrogen bonds are known to be far stronger than van deer Waals forces, it follows that materials with favourable adhesion qualities will be small enough to form hydrogen bonds [22].
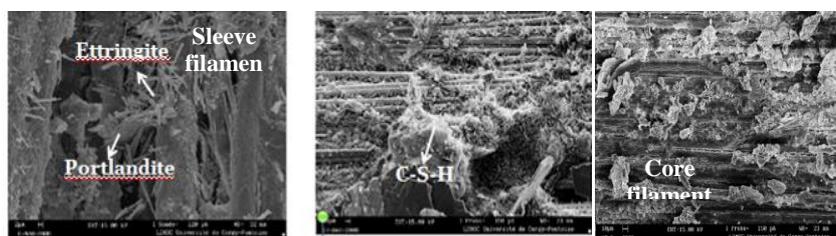

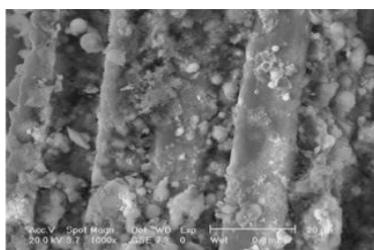Figure 5.Hydration products into filaments of yarn aged 28 days [23].

Figure 6.SEM image of hydration products between filaments in matrix (alkaline); age of specimen 360d [24].

Impregnation investigation

Textile position

In the applied deterministic and stochastic bond layer models the scatter in the interface layer and the disorder in the filament bundle are reflected by distributions: (1) of the bond quality, (2) of the bond free length and (3) of the delayed activation of filaments within the bond free length. The failure process in the bond layer next to the crack bridge involves both the damage of filaments and of the bond between the filaments and the matrix. The filaments of a roving are by no means evenly dense across the cross section distributed [25]. There are cavities of different sizes between the filaments, see figure7 and 8.
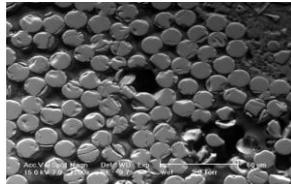

Figure 7.A roving: The effect of the filaments is visible [25].

There are two distinct regions in the yarn's of textile cross-section: the outer filaments are in direct contact with the matrix, and the inner filaments, where the mechanism of stress transfer is due to friction between individual filaments.
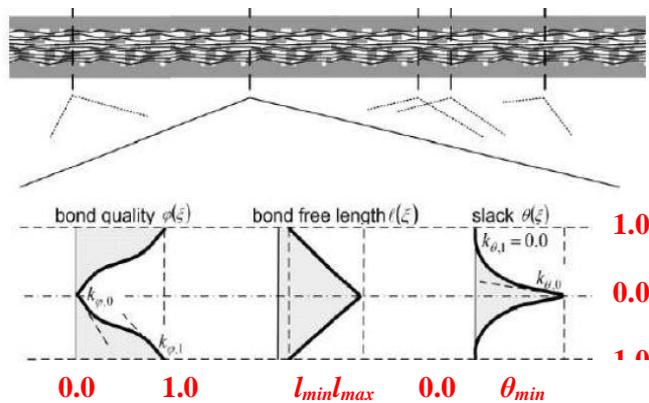

Figure 8. Profiles describing the heterogeneity in the material structure; main parameters related to textile position in concrete [26].

This is because the bond characteristics of multifilament yarn are not uniform due to the limited impregnation of a matrix into the yarn. Even though, the multifilament yarn in a matrix can be simplified as a monolithic reinforcement, and the obtained bond stress slip relationship (BSR) can be regarded as a representative on according for the whole cross-section of the yarn, [27].

Scanning Electronic Microscopy Observations

Using a scanning electron microscope (SEM) test after a mechanical test, a variety of micromechanical tests have been developed to determine the properties of the interfacial contact between filaments and matrix. Investigating the texture of porous materials can be done in part by using SEM. ASTM standard test method C 1356 [28] is used for quantitative phase abundance determination. The penetrability of mortar inside the yarn of textile is dependent on the water–cement ratio of the matrix and fiber surface properties as shown in figure 9, i.e., surface treatment and fiber type, especially when coated by epoxy, greatly increase the anchorage of the textile. Peled et al. 2008, [29] explained that higher water content and improved fiber surface properties resulted in improved compatibility leading to good cement penetrability and improved bonding.
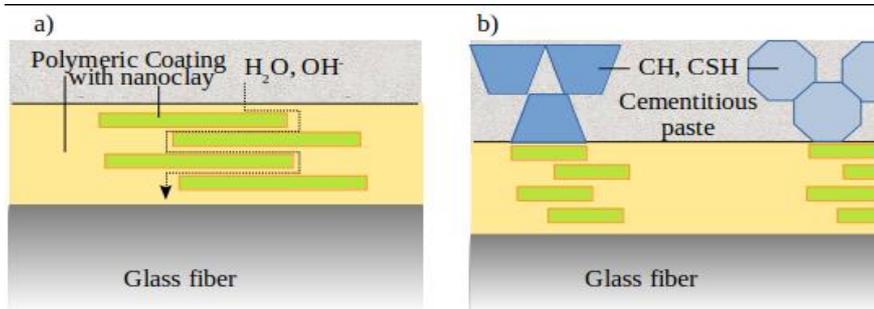

Figure 9. (a) Structure of a silicate layered coating on the surface of
glass fibers and its barrier properties according to [30];
(b) nanoclay particles dispersed in the coating promoting
the formation of hydration products at the fiber-matrix interface [31].

The hydration products in the inner filaments zone provide sticky bridges between the filaments rather than fully covering the filament surface (see figure 10). The end product is linkages between the outside filaments and the cement paste that are either linear or even pointy and span short distances. The capacity of the filaments to move in relation to one another is restricted by the production of adhesive bridges, even in cases where the filaments are not yet bonded over extended lengths with the concrete or each other.
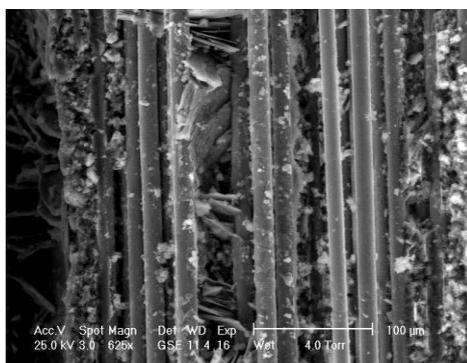

Figure 10. Filaments with a dhesive bridges [24].

The adhesion effect or bond lengths are significantly altered by the new hydration product forms and recrystallizations that occur throughout time. It is also possible for mechanical tension brought on by crystallization pressure to develop on the fiber, which in the positive case maintains the bonding bridge function in the negative case Unfavorable point loads are produced throughout the yarn in this instance.

Energy-dispersive X-ray (EDX) spectroscopy

Characterizing polymer-layered silicate (PLS) nanocomposites has been done extensively using X-ray diffraction. Bragg's law [Mayer 1007] really provides a method for determining the "interlayer" or "gallery," or the space between the silicate layers:

$$Sin\theta = n\lambda/2d' \qquad (1)$$

Where:

θ: is the diffraction angle measured:

λ: the wave length of the X-ray radiation used in the diffraction experiment.

In order to study the textile's attack microstructures, Arabi et al. (2018), [32] employed SEM equipped with an energy dispersive spectrometer (EDX), and they used X-ray diffraction to measure the Portlandite and other phase development. The effect of curing conditions on the longevity of alkali-resistant glass fibers in a cement matrix was investigated [32]. Zirconium oxide (AR)-coated alkali-resistant yarns showed similar degradation characteristics. The degree to which the yarns are protected is largely dependent on the type of cement used. They came to the conclusion that while using these materials in humid conditions still poses a risk, the addition of silica fumes decreases the degradation by reducing the pozzolanic impact, which lowers the quantity of Portlandite. These observations support the results derived from X-ray diffraction. First, the pozzolanic reaction that converts Portlandite into gel CSH and lowers the matrix alkalinity makes it possible to protect the textile with the addition of silica fume [33]. Second, the emergence of glass yarns is stable in a dry environment, due to consummation of Ca++ and Portlandite cannot grow in such an environment. The microanalysis reveals larger silica (Si) concentrations than those found on the yarn, and the silica fume additions do not cause the yarn body to degrade excessively. Figure 11 illustrates that the samples conserved in ambient air had greater silica (Si) contents according to energy dispersive X-ray (EDX) examination than the yarn of the samples stored in a damp chamber. This suggests that the alkali-resistant glass fibers employed in a cement matrix do not degrade quickly in their conservation habitat at room temperature. This indicates that the alkali-resistant glass fibers employed in a cement matrix do not degrade glass structure while stored in an ambient air environment. The diagrams of X-rays are shown in figure 12.
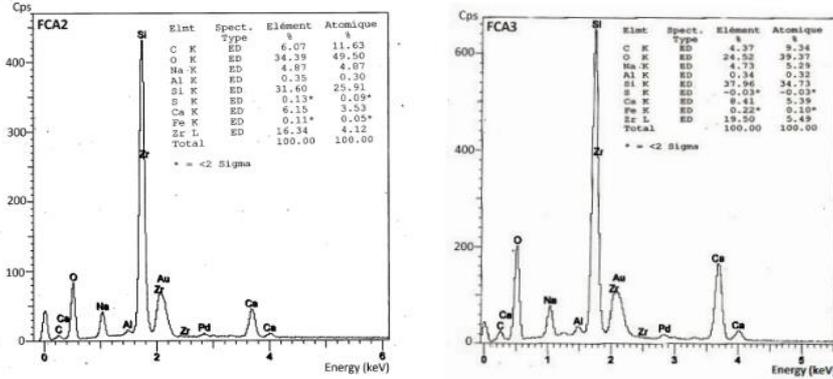
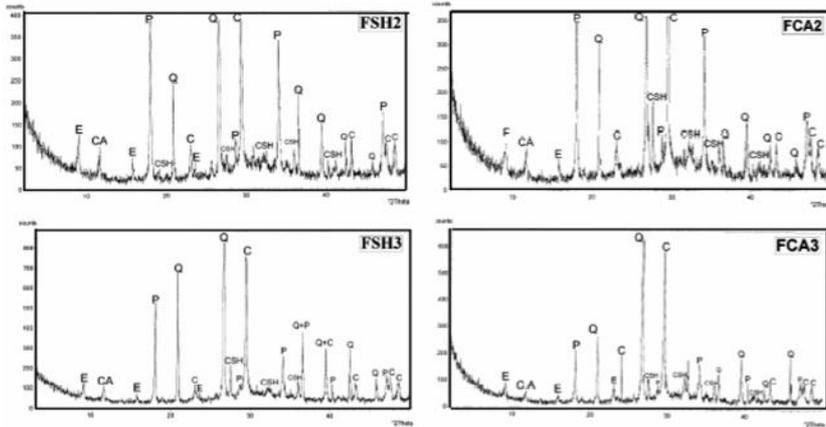Figure 11. Spectrometry X-ray analyses of the fibre body of GFRC cured at ambient air [32].

Figure 12. X-ray diffraction patterns (λ Kα Cu). Q, quartz; P, portlandite (Ca(OH)2); C, calcite (CaCO3); E, Ettringite; CSH, calcium silicate hydrates; and CA, calcium carboaluminate [35].

However, as Kwana, 2018 [34] demonstrated, the inclusion of AR glass fiber inhibits a rapid rise in permeability in both the tropical climate and cyclical wetting and drying. In order to determine the carbonation of the samples, the peak strength of calcium carbonate, or calcite, is also mentioned [35]. Comments pertinent to users of alkali resistance (AR) glass fiber present in ASTM standard C1666/C1666M – 08 [36].

Conclusion

It is more difficult to determine how certain processes affect the behavior of composites because TRC's characteristics cannot be arbitrarily changed in experimental studies (owing to the particular yarn structure and sizing role). It is evident that improving the interaction of matrix ductility and fabric-to-matrix adhesion is necessary for optimal performance. The ability to examine the impact of certain factors is provided by theoretical research in this regard as filaments position in the cementitious matrix. In order to explore the mechanisms, including the impacts of lower stiffness in the cracked state, yarn waviness, and tension softening of the cementitious matrix, experimental findings of tensile-loaded specimens are first presented. These results aid in the identification of various mechanisms in advance as SEM or X-ray. As confirmed by previous reviews, introduce pozzolanic to mortar lead to enhance the TRC structure and improve its resistance (Changes to the microstructure improve material properties).

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