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Durability of partially cured GFRP reinforcing bars in alkaline environments with or without sustained tensile load

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ABSTRACT

Corrosion of steel reinforcing bars (rebars) in reinforced concrete structures can be addressed by using corrosion-resistant materials like Fiber-Reinforced Polymers (FRP). However, in France, FRP rebars are primarily restricted to temporary applications due to lack of comprehensive durability data. While numerous studies have explored FRP durability in alkaline environments, understanding the combined effect of alkali ions and sustained loading remains limited. Furthermore, the impact of incomplete matrix curing on the long-term performance of FRP rebars is limited.

Accordingly, this paper investigates the durability of Glass FRP (GFRP) rebars exposed to alkali environments at varying temperatures. A batch of partially cured rebars was selected for this study, with part of them undergoing post-curing to investigate the effect of curing state on the ageing behaviour. Additionally, some partially cured specimens were subjected to combined sustained tensile loading at 20 % and 40 % of ultimate strength. Mechanical, physico-chemical and microstructural characterizations were conducted after various exposure durations. Results revealed that incomplete curing minimally impacted long-term tensile properties but significantly affected the interlaminar shear strength of aged rebars. Moreover, applying a combined load had little influence on residual properties at 20°C but led to rapid reduction in GFRP residual tensile strength at higher ageing temperatures (40 and 60°C).

1. Introduction

To address the issue of corrosion affecting steel reinforcements in concrete structures, an effective solution involves substituting steel with Fiber-Reinforced Polymer (FRP) reinforcing bars (rebars). Unlike steel, FRPs are resistant to corrosion and offer superior tensile strength. However, the adoption of FRP rebars in France remains limited, primarily due to a lack of comprehensive data regarding their long-term durability under service conditions [1]. While FRP bars utilized for internal reinforcement in concrete structures resist corrosion, they are still subjected to diverse environmental conditions during service, possibly impacting their long-term physical and mechanical properties, as well as

their interface behaviour with concrete. Among the potential detrimental factors are the influence of the alkaline cement environment, the effects of humidity or water cycles, and exposure to high temperatures or freeze/thaw cycles [2].

Concrete is an alkaline medium typically containing high concentrations of calcium, sodium, and potassium hydroxides within its interstitial pore solution. Consequently, the pH of concrete at early age can range between 11.5 and 13.5 [3]. This elevated pH is highly beneficial for the durability of steel reinforcements, as it promotes the formation of a passivation layer on the steel surface, resulting in low corrosion rates. In contrast, with Glass FRP (GFRP) rebars, these alkaline ions may initiate an alkaline attack on the glass fibres and lead to the

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accumulation of hydration products at the fibre/matrix interface [4–6]. However, owing to the limited mobility of alkalis within concrete, degradation kinetics progress very slowly under typical service conditions, making them challenging to assess through short-term studies. Consequently, laboratory investigations often rely on accelerated ageing protocols, involving immersion of the FRPs in an alkaline solution at pH ~ 12 –13, preferably at elevated temperature to accelerate alkali diffusion. However, it is generally recommended not to exceed an exposure temperature of 60°C, to prevent the activation of polymer matrix side reactions that do not occur under actual service conditions [7].

In various studies, vinylester matrix GFRPs (the most commonly encountered FRP rebars) have been exposed to synthetic concrete pore solutions at temperatures of up to 60°C and for relatively short periods (ranging from few months to 1 or 2 years), followed by determination of their residual mechanical properties through tensile or interlaminar shear tests [4–6] [8–12]. These studies have yielded variable and sometimes contradictory results, which can be attributed to significant variability in component materials and experimental protocols. For instance, some authors have reported tensile strength losses of up to -70% after 2 years of direct immersion in an alkaline solution at 60°C [8,9]. However, this ageing condition is unanimously regarded as excessively severe and not representative of actual service conditions [13–15]. Other studies recommend employing much less aggressive conditions, such as exposure to wet concrete, which induces significantly lower loss of properties for GFRPs (approximately -16% decrease in tensile strength after 8 months of ageing in wet concrete at 50°C [13]).

Overall, the polymer matrix is widely acknowledged to play a crucial role in the mechanical performances of composite materials [16] and their durability FRPs [14,15]. Nevertheless, only few authors have explored the effects of partial curing of the matrix on the mechanical and physical properties of pultruded composite rebars. Nazair et al. [17] found that vinylester GFRP rebars with a curing ratio of 96% (close to the 95% minimum requirement specified by CSA S807 for high durability bars of class D1 [18]) exhibited a lower Tg compared to fully cured specimens, with an average Tg value of 107°C compared to 124°C. The matrix-dominated properties, such as interlaminar shear strength, were also significantly affected by the curing ratio, with values being 8% lower in the case of partially cured specimens. Conversely, tensile properties and a few other physical properties (such as water absorption) were minimally impacted. However, there is limited documentation regarding the impact of the matrix crosslinking degree on the long-term performance of FRP rebars.

Additionally, only a limited number of authors have investigated the ageing behaviour of GFRP rebars under combined environmental and mechanical loading (simultaneous exposure to hygrothermal ageing conditions and constant loads) [19–24]. Their findings suggest that applied loads accelerate the degradation kinetics of FRP rebars, especially in alkaline solution [25], owing to synergistic effects between environmental and mechanical factors, but that current design guidelines still remain too conservative. In [22], the authors correlate various degradation phenomena with the level of applied load: ions diffusion at low load level (up to 25–30% of the ultimate strength), crack propagation at medium load level (between 30% and 50% of the ultimate strength) and stress-dominated damage at higher load level. However, this assertion relies on limited available literature, emphasizing the necessity for further studies to fully comprehend this complex degradation behaviour.

In this context, the research project outlined in this article had distinct key objectives:

- A first goal was to investigate the influence of incomplete matrix curing on the environmental durability of GFRP rebars (without load). To accomplish this, series of partially cured and post-cured rebars were directly immersed in an alkaline solution at 60°C for various durations up to 90 days. Subsequently, mechanical tests were

conducted to assess and compare their residual properties, enabling conclusions regarding the effects of the crosslink state of the vinyl-ester network on the ageing kinetics.

- The second objective was to evaluate the durability of partially cured specimens over longer periods (up to 540 days) and under more realistic alkaline environments, where the mobility of alkali ions is more comparable to that in the concrete medium. In this phase, the specimens were initially embedded in a thin concrete cylinder before immersion in alkaline solutions at different temperatures - a condition referred to as “indirect ageing”. Once again, the specimens were characterized after various exposure durations to evaluate their residual mechanical properties.
- Finally, the last focus was to provide deeper insights into the effects of combined sustained load on durability outcomes. Specific test devices were employed to subject GFRP rebars to both indirect alkaline ageing and creep load. Comparing the residual properties of GFRPs aged under these combined factors with those of specimens aged under indirect ageing without load enabled discussions regarding the influence of the load on degradation kinetics.

The initial section of the article introduces the FRP rebars under study, along with the characterization methods, ageing protocols, and the experimental program. The subsequent sections present the main findings relevant to the aforementioned investigations.

2. Materials and experimental methods

2.1. FRP rebars

This study examines the durability of commercially available FRP rebars, which consist of a vinylester matrix reinforced with glass fibers (GFRP). This type of rebar is widely utilized and is manufactured through a pultrusion process. The rebars feature a nominal diameter of 10mm and a sand-coated surface finish combined with helical wrap, as depicted in Fig. 1. Table 1 outlines the key properties of these FRP rebars as provided by the manufacturer. The name of the manufacturer and product brand are not released for confidentiality reasons.

To explore the impact of the crosslinking state of the vinylester matrix on the mechanical performance and durability of these GFRPs, specimens from a batch of partially cured rebars were initially chosen. Some of these specimens underwent a post-curing treatment for 3 hours at 130°C in an oven to enhance the crosslinking degree. Thus, both partially cured and post-cured specimens were included in the experimental testing program. The characteristic values of the glass transition temperatures (Tg) for both types of rebars will be detailed in Section 3.

2.2. Characterization methods

As part of this durability study, the rebars were characterized both in their initial state and after exposure to specific ageing conditions, to evaluate any changes in their mechanical and physico-chemical properties over time. This section offers comprehensive details on the experimental techniques and protocols employed.

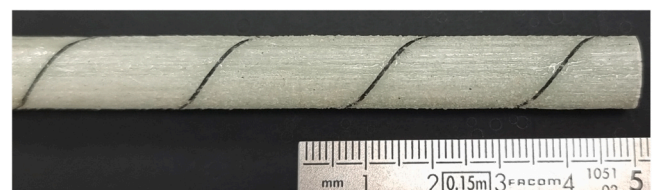


Fig. 1. Image of the GFRP rebars under study, showing the aspect of the surface finish.

Table 1
Properties of the GFRP rebars specified by the manufacturer.

| Rebars | Nominal diameter (mm) | Effective cross-section area (mm ²) | Guaranteed tensile strength (MPa) | Tensile modulus of elasticity (GPa) |
|--------|-----------------------|---|-----------------------------------|-------------------------------------|
| GFRP | 10 | 71 | 900 | 46 |

2.2.1. Physico-chemical characterizations

Various physico-chemical characterizations were conducted on both initial and aged FRP rebars. These characterizations mainly comprised Differential Scanning Calorimetry (DSC) analysis to evaluate the glass transition temperature (T_g) of the polymer matrix, Fourier Transform InfraRed (FTIR) spectroscopy to monitor any potential evolution of the chemical structure of the FRP material during ageing, as well as Scanning Electron Microscopy (SEM) observations.

The DSC experiments were performed using a TA Instruments DSC 250 calorimeter operating in the temperature modulation mode (MDSC). T_g of the FRP samples (~20 mg taken from the core of the rebars) was measured during a heating ramp from -10°C to 250°C, at a rate of 3 °C/min and with a superimposed temperature modulation of amplitude of 1.5°C and a period of 60 s. Two ramps were applied: the first to determine the T_g value for the state as is, and the second for the fully cured state. T_g was determined within the region of the sharp endothermic step change observed on the reversing heat flow thermogram, employing the midpoint temperature method (at half of the step height). Values were averaged from three repeated tests.

FTIR investigations were carried out using a Nicolet iS50 spectrometer from Fischer Scientific company, equipped with a diamond Attenuated Total Reflection (ATR) device. Spectra were collected over the 4000–400 cm⁻¹ wavenumber range, with a resolution of 4 cm⁻¹ and an accumulation of 64 scans.

SEM observations were performed using a SU5000 microscope equipped with an EDX probe (Energy Dispersive X-Ray). Prior to analysis, the samples underwent metallization with a 4 μm platinum coating.

Additional experiments were also carried out by Thermo-Gravimetric Analysis (TGA) to assess the mass ratio of glass fibers present in the GFRP rebars. Samples extracted from the core of the bars were analyzed using an STA 409 apparatus from NETZSCH Company. The analysis involved monitoring the mass loss of the samples during a temperature ramp ranging from ambient to 550°C, at a rate of 10 °C/min and under a nitrogen atmosphere. The ratio between the final and initial mass yielded the mass fiber content within the rebars. This value was derived from an average of three repeated tests.

2.2.2. Mechanical testing

The tensile strength and longitudinal modulus of elasticity of the GFRP rebars were determined through tensile tests conducted in accordance with the American standard ASTM D7205 [26], the ISO standard 10406-1 [27] and the recommendations of the French Civil Engineering Association (AFGC) [1]. To prevent excessive damage to the ends of the FRP rebars during testing and to avoid premature rupture, a specific anchoring system was employed at each end (steel tube filled with an expansive mortar). Tensile tests were conducted on an Instron hydraulic universal testing machine (UTM) equipped with a load cell of capacity 250 kN, at a displacement rate of 1 mm/min (refer to Fig. 2.a). To measure the strain of the rebar during loading, a non-contact video extensometer was employed, ensuring accurate and reliable data collection during the experiments. The longitudinal modulus of elasticity of the rebars was determined using Eq. 1.

$$E_L = \frac{\sigma_{40\%} - \sigma_{20\%}}{\varepsilon_{40\%} - \varepsilon_{20\%}} \quad (1)$$

Where E_L is the longitudinal modulus of elasticity, $\sigma_{20\%}$ and $\sigma_{40\%}$ are the tensile stresses at 20 and 40% of the ultimate tensile strength, respectively, and $\varepsilon_{20\%}$ and $\varepsilon_{40\%}$ the corresponding tensile strains.

Additionally, interlaminar shear tests, also known as short-beam tests, were performed on the FRP rebars in accordance with ASTM D4475 standard [28] and AFGC recommendations [1]. This method involves subjecting the rebar to a three-point bending test with closely spaced supports (Fig. 2b), enabling the assessment of the fiber/matrix bond strength. The distance between the supports was set at five times the diameter of the FRP rebar. Short-beam tests were conducted using an electromechanical UTM with a capacity of 100 kN, at a displacement rate of 1 mm/min. To ensure more accurate force measurement within the strength range of the study, the UTM was equipped with a 50 kN load cell. The interlaminar shear strength (ILSS) was determined using the formula specified in the ASTM test standard [28].

2.3. Description of the various accelerated ageing conditions

2.3.1. Accelerated ageing in alkaline environments without mechanical loading

As previously outlined, a first step involved subjecting a series of GFRP rebars to accelerated ageing in alkaline environments without applying any load. This particular ageing condition is denoted as ANL (Ageing with No Load) in subsequent sections.

Two types of sample conditioning were considered in this phase of the study: direct and indirect immersions.

In the direct immersion method, rebars were immersed directly in an

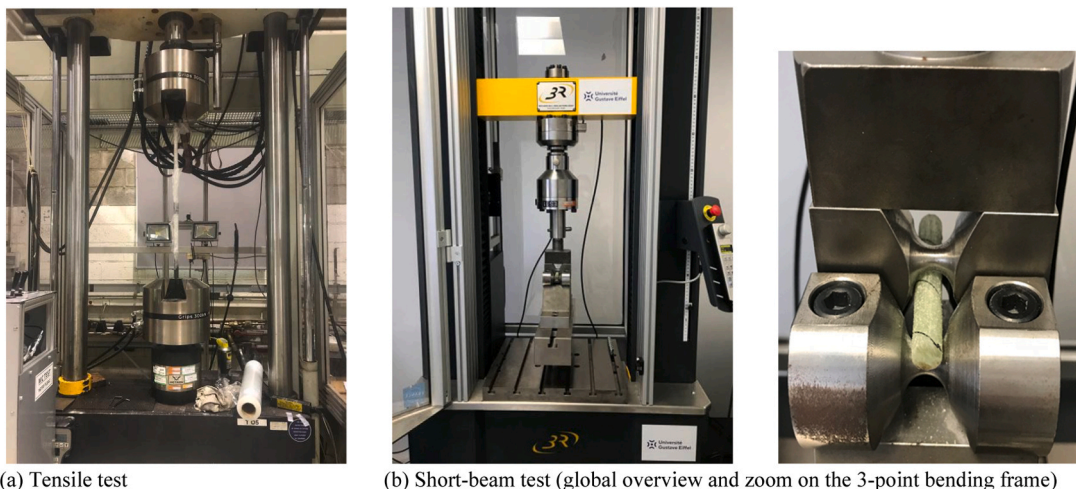


Fig. 2. Experimental setups for mechanical testing of GFRPs.

alkaline solution comprising sodium hydroxide (NaOH) at a concentration of 0.1 mol/L and potassium hydroxide (KOH) at a concentration of 0.5 mol/L, resulting in a pH of approximately 13.5 (at ambient temperature). This solution has been widely employed in previous durability studies [11,29] for simulating the concrete environment in its early stages. In practice, the extremities of the GFRP rebars were sealed with epoxy adhesive before immersion, to prevent any capillary absorption through the cut end sections. The rebars were then conditioned in large tanks filled with the alkaline solution, the temperature of which was carefully regulated using heating resistance. This direct ANL ageing condition was deliberately chosen to compare the ageing behaviour of partially cured and post-cured GFRP specimens under a consistent temperature of 60°C, aligning with the maximum recommended temperature level in the literature.

In contrast, the indirect immersion protocol aimed to replicate a more realistic service environment, reflecting the limited mobility of alkali ions. In this case, the rebars were first embedded in a thin concrete cylinder with an external diameter of 50 mm (resulting in a concrete cover of 20 mm, twice the diameter of the rebar), as shown in Fig. 3a and b. The length of the concrete cylinder was the same as that of the GFRP bar (1 m) and the ends were sealed with epoxy resin to prevent solution diffusion from the edges. The concrete mixture per cubic meter comprised 375 kg of CEM III-A cement, 810 kg of 0/4 sand, 1130 kg of 4/14 gravel, and 206.25 kg of water. Before being submitted to ageing, the specimens with concrete cover were cured in water at 20°C for one month, and then cured in air at 20°C for an additional month. Subsequently, these embedded GFRP specimens were also conditioned in thermoregulated tanks filled with the alkaline solution (Fig. 3c).

This indirect ANL condition was specifically implemented to evaluate the durability of a series of partially cured GFRP rebars over extended exposure durations of up to 540 days, and across three distinct temperatures: 20°C (room temperature), 40°C, and 60°C. Following various exposure periods (E1 = 30 days, E2 = 60 days, E3 = 90 days, E4 = 180 days, E5 = 360 days, E6 = 540 days), the specimens were extracted from the baths, and stored for one month in the laboratory to allow for drying at room temperature. Finally, the concrete cover was removed through a Brazilian splitting test, and the retrieved aged GFRP rebars underwent characterization using the same mechanical and physico-chemical methods outlined in the preceding sections.

2.3.2. Accelerated ageing in alkaline environments under sustained load

An additional set of partially cured GFRP rebars underwent exposure

to an alkaline environment under sustained load conditions, designated as ASL (Ageing under Sustained Load). In this setup, the exposure to the alkaline solution remained indirect, as the exposed portion of the rebars was embedded in an intermediate concrete cylinder of 50 mm diameter (Fig. 4a).

A sustained tensile load was applied to the specimens using two types of metallic frames, as illustrated in Fig. 4b. A total of six frames were employed, allowing six specimens to be loaded simultaneously. These devices facilitated loading via a hydraulic jack, with resulting longitudinal strain being secured by anchors fixed using specific threaded parts. The load was applied to the GFRP rebar using adapted wedges and sleeves. No failure was observed in the rebar indicating that this test setup was appropriate. Maintaining a constant force was a critical point in these investigations, especially considering the significant relaxation that can occur at early stages, particularly at elevated temperatures. Therefore, to address these relaxation phenomena and ensure long-term force constancy, a reloading procedure was implemented. This procedure was calibrated using preliminary specimens monitored by a load cell, with a maximum allowable load loss of 5 % throughout the entire test. In the test program, two load levels were examined, corresponding to 20 % and 40 % of the average tensile strength of unaged GFRP rebars (as determined at 20°C). These load levels are in line with those documented in the literature, although the highest level exceeds the current stress limits recommended by existing standards or guidelines (for instance a stress limit of 20 % is specified by ACI 440.1R-15 regarding creep rupture for GFRP rebars [30]).

Furthermore, each metallic frame was equipped with a double cell system: one cell housed the alkaline solution, while the other circulated water at a constant temperature, facilitating thermal regulation of the ageing solution. The length of the double cell (400 mm) determined the embedment length of the GFRP bars in concrete (refer to Fig. 4a and c). The specimens underwent ageing under ASL conditions at three distinct ageing temperatures (20°C, 40°C, and 60°C) and for durations of up to 90 days. At the end of the ageing test, the specimens were stored in the laboratory at room temperature for one month to facilitate drying. Subsequently, the concrete cover was removed through a Brazilian splitting test, and the retrieved aged GFRP rebars were then subjected to the various characterizations.

2.3.3. Summary of the experimental program

The global test matrix (exposure conditions and mechanical tests) is outlined in Table 2. It is worth noting that the physico-chemical analyses

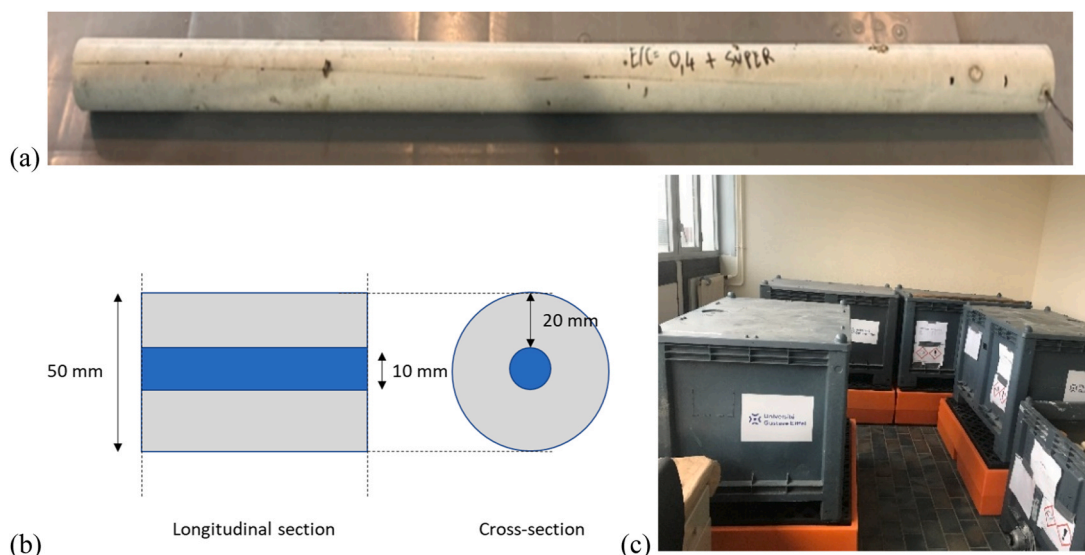


Fig. 3. ANL ageing protocol (no load) - (a) GFRP specimen with a concrete cover used for indirect immersion conditioning (b) schemes of the specimens, and (c) tanks containing the alkaline solution at different temperatures.

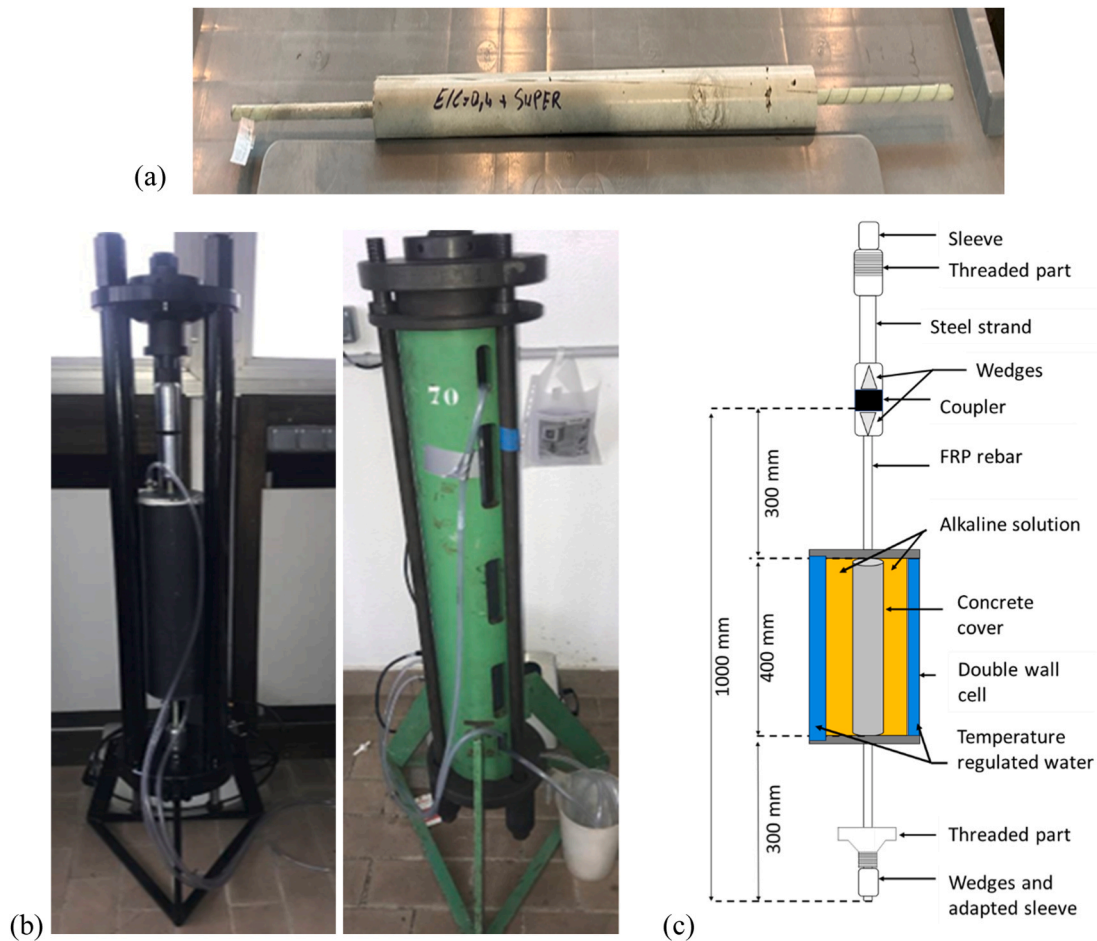


Fig. 4. ASL ageing protocol (with sustained load) - (a) GFRP specimen partially embedded in concrete; (b) Two models of metallic frames used for applying sustained load and (c) Schematic representation of the double cell enabling exposure to alkaline solution at various controlled temperatures.

(DSC, FTIR, SEM-EDX) were systematically conducted after all ANL exposures, and for some ASL specimens as well.

The nomenclature used for the samples is specified in Fig. 5. The first term indicates the presence (ASL) or absence (ANL) of sustained load during ageing. The second term specifies whether the sample was post-cured (PC) or not (NPC). The third and fourth terms provide information on the ageing environment, respectively, direct (D) or indirect immersion (I) and the temperature level. The fifth term denotes the level of sustained load (20 or 40 %), and the last term indicates the ageing duration in days.

3. Experimental results

3.1. Characterizations of unaged GFRP rebars

3.1.1. Physico-chemical properties

Several physico-chemical characterizations were carried out on the initial lot of GFRP rebars provided by the manufacturer.

SEM observations of polished cross-sections revealed a uniformly distributed arrangement of glass fibers within the polymer matrix (refer to Fig. 6a). The average diameter of the glass fibers was measured to be around 24 μm . Notably, no significant voids or prominent defects were detected in the examined samples.

TGA characterizations yielded a mass ratio of glass fibers of (84.5 ± 0.4) % in the GFRP rebars, which is well above the minimum requirement of 70 % as recommended by AFGC guidelines [1].

DSC analyses performed on samples of this initial lot confirm that the GFRP rebars are partially cured. Specifically, the glass transition

temperature (T_g) determined during the initial ramp exhibited an average value of (71 ± 1) $^{\circ}\text{C}$, whereas the T_g measured during the subsequent run for the fully cured composite was notably higher at (114 ± 1) $^{\circ}\text{C}$. Additionally, an exothermic peak is evident during the initial run on the non-reversing heat flow signal, indicating incomplete polymerization of the vinyl ester matrix (see Fig. 6b). Unfortunately, quantitative determination of the curing ratio from the enthalpy of the residual exothermic peak was not possible due to missing information regarding the total heat of polymerization of the matrix (the manufacturer did not disclose the commercial name and characteristics of the matrix system). Finally, since the initial T_g falls below the recommended minimum value of 100 $^{\circ}\text{C}$ outlined by AFGC and CSA S807 guidelines [1, 18], this batch of partially cured specimens does not meet the criteria for high durability GFRP bars (class D1).

As previously mentioned in the introduction, one objective of the study is to explore the influence of the crosslinking state of the vinyl ester matrix on the durability of the GFRP rebars. In line with this objective, a series of partially cured samples was subjected to a thermal post-curing treatment at 130 $^{\circ}\text{C}$ for 3 hours for comparison with the initial lot of rebars. DSC characterizations provided a T_g value (first run) of (102 ± 1) $^{\circ}\text{C}$ for these post-cured samples, indicating a substantial increase in the crosslinking degree compared to the initial specimens. Although this T_g value remains lower than that of the fully cured system (as determined by DSC during the second run), no residual exothermic peak was detected on the thermogram, suggesting a high curing ratio. Furthermore, the T_g value of these post-cured samples now exceeds the threshold of 100 $^{\circ}\text{C}$ required for durability class D1.

In the following sections, the post-cured rebars will be denoted as PC,

Table 2
Experimental investigation test matrix.

| Application of sustained load or not | Application of post-cure or not | Direct or indirect ageing | Ageing temperature (°C) | Sustained load (% of ultimate load) | Ageing duration (days) | Number of samples -tensile tests | Number of samples - ILSS | | |
|--------------------------------------|---------------------------------|---------------------------|-------------------------|-------------------------------------|------------------------|----------------------------------|--------------------------|---|---|
| ANL | PC | — | — | — | — | 3 | 3 | | |
| | | D | 60 | — | 45d | 3 | 3 | | |
| | NPC | D | 60 | — | 90d | 3 | 3 | | |
| | | — | — | — | — | 3 | 3 | | |
| | | D | 60 | — | 45d | 3 | 3 | | |
| | | D | 60 | — | 90d | 3 | 3 | | |
| | | I | 20 | — | 30d | 3 | 3 | | |
| | | I | 20 | — | 60d | 3 | 3 | | |
| | | I | 20 | — | 90d | 3 | 3 | | |
| | | I | 20 | — | 180d | 3 | 3 | | |
| | | I | 20 | — | 360d | 3 | 3 | | |
| | | I | 20 | — | 540d | 3 | 3 | | |
| | | I | 40 | — | 30d | 3 | 3 | | |
| | | I | 40 | — | 60d | 3 | 3 | | |
| | | I | 40 | — | 90d | 3 | 3 | | |
| | | I | 40 | — | 180d | 3 | 3 | | |
| | | I | 40 | — | 360d | 3 | 3 | | |
| | | I | 40 | — | 540d | 3 | 3 | | |
| | | ASL | NPC | I | 20 | 20 % | 28d | 3 | 0 |
| | | | | I | 20 | 40 % | 28d | 3 | 0 |
| I | 20 | | | 40 % | 90d | 3 | 0 | | |
| I | 40 | | | 40 % | 16d | 3 | 0 | | |
| I | 40 | | | 40 % | 42d | 3 | 0 | | |
| I | 40 | | | 40 % | 70d | 0 | 9 | | |
| I | 60 | | | 40 % | 12d | 0 | 9 | | |
| | | | | | TOTAL | 87 | 90 | | |

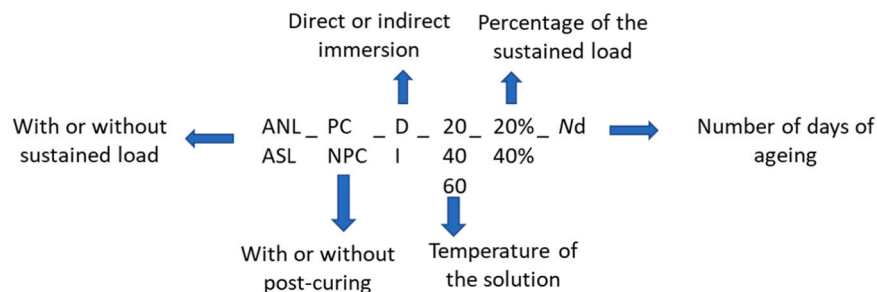


Fig. 5. Nomenclature of the experimental investigations.

while the initial sample lot will be noted as NPC (for Non Post-Cured).

3.1.2. Mechanical properties

Tensile and short-beam tests were subsequently conducted on both unaged NPC and PC GFRP specimens. The mechanical properties, including tensile strength (f_t) and longitudinal modulus (E_L), as well as Interlaminar Shear Strength (ILSS), are presented in Table 3. Values represent averages from three repeated tests, with standard deviations calculated using Pearson’s method in accordance with the AFGC recommendations [1].

A comparison of the tensile properties between NPC and PC specimens reveals that the curing state does not significantly influence the longitudinal strength and elastic modulus of the rebars, which are primarily determined by the fiber characteristics. In both cases, a brittle and explosive failure of the specimens was observed, as illustrated in Fig. 7a.

In contrast, post-curing of the rebars was found to significantly enhance the ILSS by nearly 30 %. This finding aligns with the research of Nazair et al., who demonstrated that matrix-dominated properties such

as ILSS are influenced by the curing ratio [17]. The typical failure mode observed in the specimens involved interfacial decohesion as depicted in Fig. 7b.

Furthermore, it’s worth noting that both the tensile properties of the unaged NPC and PC rebars exceed the minimum threshold values specified by AFGC [1]. As a reminder, it is recommended that the tensile strength exceeds 850 MPa for GFRPs with a nominal diameter of 10 mm, while the longitudinal modulus should be over 45 GPa. Similarly, the measured ILSS properties surpass the threshold value of 40 MPa specified by the CSA S807 code for GFRP rebars [18].

3.2. Durability of GFRP rebars under alkaline environment without load (ANL)

3.2.1. Short-term ageing of GFRPs under direct immersion: impact of curing state

To assess the influence of the curing state on the durability of GFRP rebars, both partially cured (NPC) and post-cured (PC) rebars were subjected to direct immersion (designated by the letter D) in the alkaline

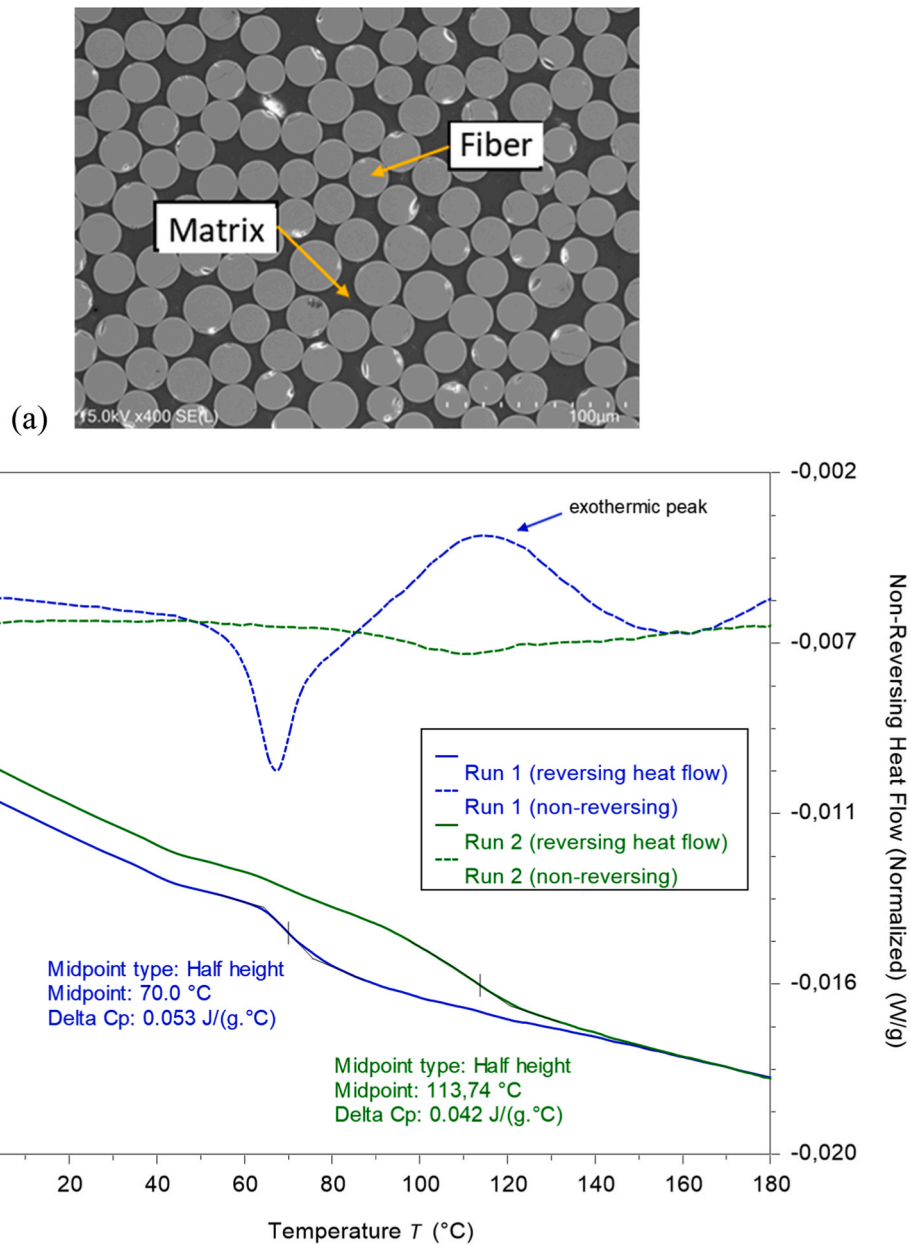


Fig. 6. Physico-chemical characterizations of the initial lot of GFRP rebars – (a) SEM observation of the microstructure and (b) typical thermograms obtained by modulated DSC.

Table 3
Initial mechanical properties determined on NPC and PC GFRP rebars.

| Type of GFRP rebar | Tensile strength, f_u (MPa) | | Tensile elastic modulus, E_t (GPa) | | Interlaminar shear strength, ILSS (MPa) | |
|--------------------|-------------------------------|--------------------|--------------------------------------|--------------------|---|--------------------|
| | Mean value | Standard deviation | Mean value | Standard deviation | Mean value | Standard deviation |
| NPC_GFRP | 1128.3 | 42.8 | 58.0 | 1.6 | 45.6 | 1.9 |
| PC_GFRP | 1156.0 | 23.2 | 56.7 | 0.7 | 58.9 | 0.5 |

solution at 60°C, representing the most severe conditions examined in this study. The rebars underwent ageing for two durations (45 and 90 days) before undergoing various characterizations.

DSC analyses were conducted on the aged specimens to evaluate changes in Tg during ageing, providing insights into the curing state evolution of the polymer matrix. Fig. 8 depicts the Tg evolution over

exposure time for both NPC and PC series of GFRP rebars. The results indicate that undercured NPC specimens undergo a significant cross-linking process during exposure, evidenced by a rapid increase in Tg to approximately 101°C after 90 days. In contrast, the Tg of PC specimens displays minimal variation throughout the ageing process in the 60°C solution, possibly even showing a slight decrease. Notably, after 90 days of exposure, both specimen types exhibit closely aligned Tg values, approximately 101°C.

Mechanical characterizations were also conducted on the aged NPC and PC rebars. The residual properties obtained from these tests are reported in Table 4 and illustrated in Fig. 9. In Table 4, the percentage of variation with respect to the unaged specimen is also presented.

The results indicate that the tensile properties of both rebar series are minimally affected by direct alkaline exposure for up to 90 days. No significant change was observed in the tensile elastic modulus, aligning with findings in existing literature [11,17]. As for tensile strength, a marginal decrease may be observed after 90 days, particularly



Fig. 7. Representative failure modes of NPC GFRP Rebars in (a) Tensile and (b) Short-Beam Tests.

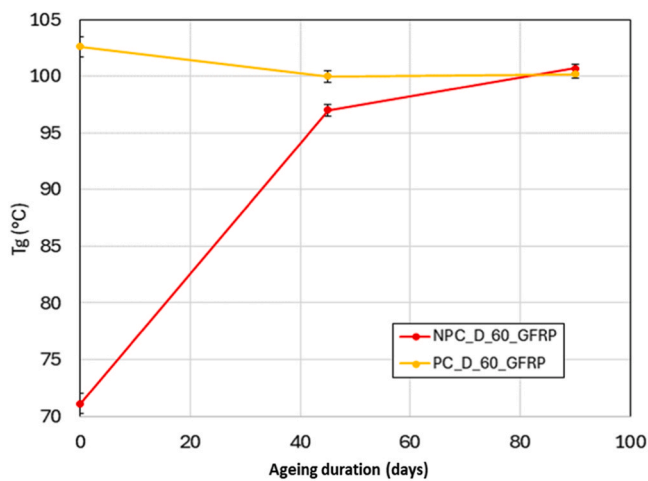


Fig. 8. Evolution of Tg for NPC and PC GFRP specimens during direct immersion (D) in alkaline solution at 60°C and without load (ANL).

noticeable in NPC samples. However, this kinetic evolution is relatively minor compared to findings reported by other researchers studying direct alkaline immersion [10,25]. For example, Chen et al. [10] reported a drop of 50 % of the initial tensile strength after only 70 days of direct alkaline exposure, while in our study a decrease of only 3 % was observed for NPC GFRP rebars after 90 days of alkaline attack at 60°C.

In terms of interlaminar shear strength (ILSS), PC samples exhibit minimal change with ageing, whereas NPC samples demonstrate a notable increase in ILSS values over the course of 90 days. This phenomenon correlates well with the changes in Tg highlighted by DSC analysis for both types of specimens. ILSS serves as a key indicator of fiber/matrix bond strength and is significantly influenced by matrix properties [17]. In NPC rebars, the observed rise in Tg signifies a

crosslinking process during ageing, thereby explaining the ILSS increase. Conversely, the curing state of PC specimens remains relatively stable throughout ageing, resulting in negligible fluctuations in ILSS values. It's noteworthy that both specimen types converge to similar ILSS values after 90 days of exposure, consistent with their comparable Tg values (101°C). This suggests that any initial differences in curing states between NPC and PC specimens are compensated during ageing, leading to closely aligned residual properties after 90 days of direct immersion.

In conclusion, it appears that the incomplete cure does not significantly impact the durability of the NPC GFRP rebar series when compared to the PC specimens under direct immersion, at least within the 90-day ageing period investigated. However, comparison over extended ageing periods should be conducted before generalising this result.

3.2.2. Long-term ageing of partially cured GFRPs under indirect immersion

In this section, the long-term ageing behavior of a series of NPC GFRP specimens subjected to indirect immersion in the alkaline solution (without load) at different temperatures is examined. In this configuration, the mobility of alkali ions is restricted compared to direct immersion, due to the presence of the concrete cover. This can be considered more representative of actual field conditions.

3.2.2.1. Impact on physico-chemical properties and microstructure

3.2.2.1.1. Changes in glass transition temperature. Fig. 10 displays the evolution of Tg (determined by DSC during the first heating ramp) for NPC GFRP specimens exposed to indirect ANL conditions (which involves embedment in concrete cylinders and immersion in the alkaline solution at 20, 40 or 60°C), over periods of up to 18 months (540 days).

The kinetics of Tg evolution in GFRP rebars exhibits a strong sensitivity to the temperature of the ageing environment. At 60°C, a swift rise in Tg is initially observed, suggesting a post-curing phenomenon consistent with previous findings under direct immersion conditions at the same temperature (Cf. Section 3.2.1). However, after approximately

Table 4

Residual mechanical properties of NPC and PC GFRP rebars after direct immersion (D) in the alkaline solution at 60°C for various durations.

| Sample designation | f _u (MPa) | | | E _t (GPa) | | | ILSS (MPa) | | |
|--------------------|----------------------|--------------------|------------------|----------------------|--------------------|------------------|------------|--------------------|------------------|
| | Mean | Standard deviation | Variation (in %) | Mean | Standard deviation | Variation (in %) | Mean | Standard deviation | Variation (in %) |
| NPC_GFRP (unaged) | 1128.3 | 42.8 | — | 58.0 | 1.6 | — | 45.6 | 1.9 | — |
| NPC_D_60_GFRP_45d | 1171.3 | 25.0 | 3.8 | 56.3 | 1.4 | -2.9 | 53.0 | 1.7 | 16.2 |
| NPC_D_60_GFRP_90d | 1095.9 | 27.2 | -2.9 | 56.8 | 0.7 | -2.1 | 59.3 | 3.8 | 30.0 |
| PC_GFRP (unaged) | 1156.0 | 23.2 | — | 56.7 | 0.7 | — | 58.9 | 0.5 | — |
| PC_D_60_GFRP_45d | 1164.6 | 29.2 | 0.7 | 56.9 | 0.9 | 0.4 | 56.0 | 4.3 | -4.9 |
| PC_D_60_GFRP_90d | 1136.0 | 36.9 | -1.7 | 56.3 | 1.0 | -0.7 | 57.2 | 4.0 | -2.9 |

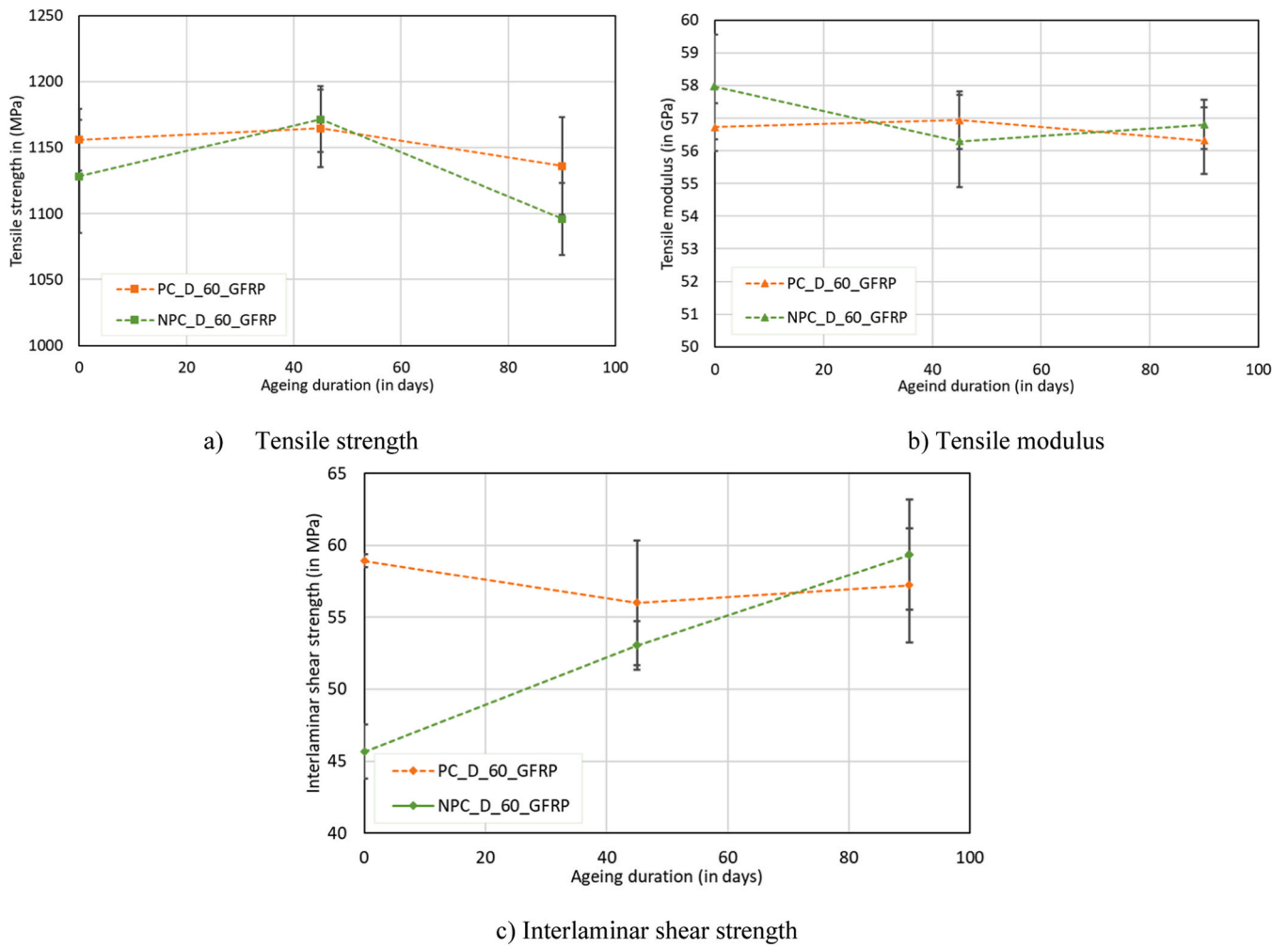


Fig. 9. Changes in mechanical properties of NPC and PC GFRP rebars during direct immersion (D) in alkaline solution at 60°C and without load (ANL).

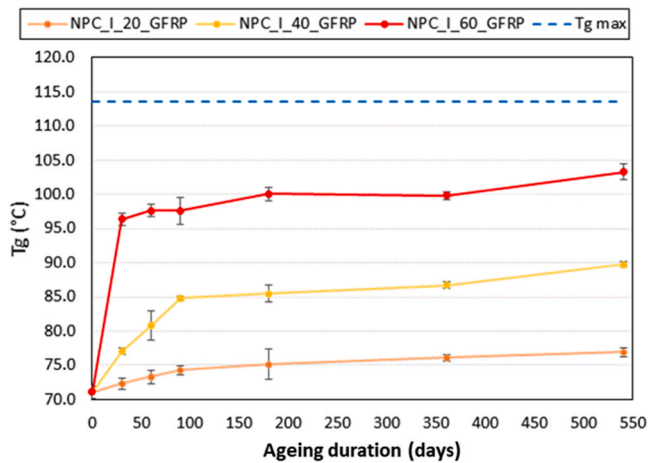


Fig. 10. Evolution of Tg for NPC GFRP specimens during indirect immersion (I) in alkaline solution at various temperatures and without load (ANL).

40–50 days at 60°C, the kinetics decelerate significantly, resulting in a limited further increase in Tg, with a value reaching about 103°C after 540 days. At 40°C, a comparable post-curing process is observed, although with slower kinetics and a lower Tg value reached after 540 days. At 20°C, the evolution of Tg is notably slower, and even after one and a half years of exposure, the Tg value barely exceeds 78°C,

indicating that the matrix remains substantially undercured.

Overall, the Tg values of the aged specimens remain below that of the fully cured system (approximately 114°C, as measured by DSC during the second run), highlighting that the ageing temperatures investigated in the study are insufficient to achieve complete polymerization of the rebars from the NPC lot.

3.2.2.1.2. Microstructural changes. SEM analysis of cross-sections revealed that NPC GFRP rebars subjected to indirect immersion at 60°C displayed signs of localized damage, which became apparent after 60 days of exposure (Fig. 11) and increased with prolonged exposure time. These damages encompassed fiber breakages and localized failures in regions characterized by substantial porosity. This finding suggests the initiation of a degradation process within the composite rebars at elevated temperature. Furthermore, evidence of diffuse fiber damage was also detected in specimens aged at 20°C and 40°C, especially for exposure durations exceeding 360 days, indicating a much slower degradation kinetics at these temperatures.

3.2.2.1.3. Effect on chemical structure. Complementary analyses were performed by SEM-EDX to detect any possible migration of the alkali ions from the ageing solution or the intermediate concrete cover towards the core of the NPC GFRP rebars. EDX mapping of potassium of sodium elements within the aged samples did not reveal any significant difference compared to the unaged specimens. Therefore, the onset of degradation previously observed at 60°C could not be correlated to a diffusion of the alkali ions in the rebars.

Similarly, FTIR analyses of the aged specimens revealed no significant alteration in the chemical structure of the vinyl ester matrix of

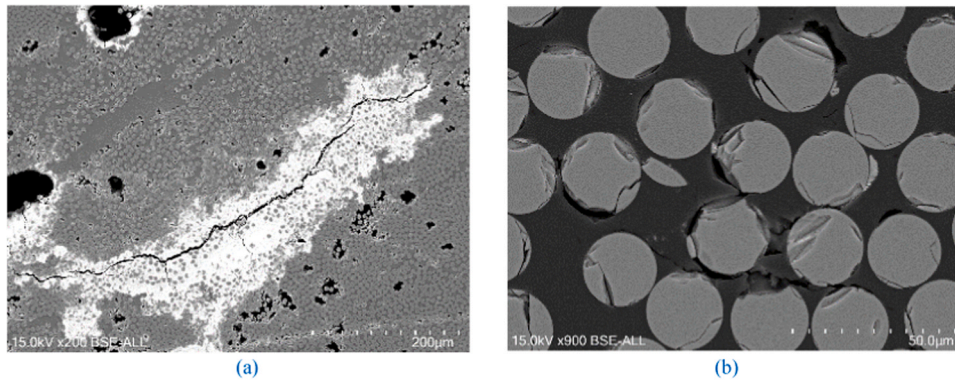


Fig. 11. SEM examination of cross-sections of NPC GFRP specimens after 60 days of indirect immersion in the alkaline solution at 60°C, highlighting localized damages at (a) low magnification, and (b) high magnification.

GFRPs following indirect alkaline exposure, regardless of the temperature. Hydrolytic degradation typically results in a significant increase in the intensity of absorption bands attributed to hydroxyl species around 3400 cm^{-1} , due to the conversion of ester groups (R-COO-R') into alcohol (R-OH) and carboxylic (R-COOH) species. Fig. 12 illustrates that the spectra of the samples remained unchanged after conditioning (here after 360 days), thereby confirming the vinyl ester matrix's good resistance to alkaline degradation.

Overall, physico-chemical analyses did not enable precise identification of the mechanism responsible for the diffuse microstructural damage detected by SEM observations.

3.2.2.2. Impact on mechanical properties

3.2.2.2.1. Changes in residual tensile properties. Tensile tests were conducted on NPC GFRP specimens aged for up to 540 days under indirect alkaline immersion. All aged specimens exhibited brittle fracture mode, very similar to unaged NPC samples (refer to Fig. 6a). The residual tensile properties are outlined in Table 5, while the changes in tensile strength and elastic modulus over the ageing period are illustrated in Fig. 13.

The experimental findings indicate limited changes in the tensile properties over the 540-day exposure period for specimens conditioned under indirect immersion at 20°C and 40°C, with reductions of 4 % and 5 % respectively. Conversely, a significant decrease in strength was noted at 60°C for exposure durations exceeding 180 days (with a

Table 5

Residual tensile properties of NPC GFRP rebars after different durations of indirect immersion (I) in alkaline solution at various temperatures.

| Sample designation | Ultimate load (kN) | f_u (MPa) | | E_L (GPa) | |
|--------------------|--------------------|-------------|--------------------|-------------|--------------------|
| | | Mean | Standard deviation | Mean | Standard deviation |
| NPC_GFRP (unaged) | 88.6 | 1128.3 | 42.8 | 58.0 | 1.6 |
| NPC_I_20_GFRP_30d | 85.8 | 1092.3 | 75.3 | 56.6 | 0.8 |
| NPC_I_20_GFRP_60d | 90.8 | 1156.2 | 27.1 | 56.0 | 1.5 |
| NPC_I_20_GFRP_90d | 84.9 | 1080.6 | 12.9 | 56.7 | 1.3 |
| NPC_I_20_GFRP_180d | 93.2 | 1187.1 | 13.5 | 55.9 | 0.4 |
| NPC_I_20_GFRP_360d | 91.7 | 1168.1 | 19.2 | 56.9 | 1.0 |
| NPC_I_20_GFRP_540d | 85.3 | 1085.6 | 23.5 | 57.0 | 1.9 |
| NPC_I_40_GFRP_30d | 91.9 | 1169.6 | 27.0 | 57.9 | 1.0 |
| NPC_I_40_GFRP_60d | 86.4 | 1100.3 | 83.6 | 56.4 | 1.3 |
| NPC_I_40_GFRP_90d | 86.2 | 1097.1 | 52.7 | 57.2 | 1.2 |
| NPC_I_40_GFRP_180d | 91.6 | 1166.4 | 34.3 | 57.0 | 1.1 |
| NPC_I_40_GFRP_360d | 87.0 | 1107.6 | 5.7 | 57.1 | 1.3 |
| NPC_I_40_GFRP_540d | 84.2 | 1072.1 | 14.7 | 57.0 | 0.9 |
| NPC_I_60_GFRP_30d | 88.9 | 1132.0 | 11.6 | 57.4 | 1.7 |
| NPC_I_60_GFRP_60d | 87.5 | 1114.2 | 14.4 | 57.1 | 0.2 |
| NPC_I_60_GFRP_90d | 80.0 | 1018.4 | 27.7 | 56.0 | 1.2 |
| NPC_I_60_GFRP_180d | 84.5 | 1076.4 | 24.9 | 57.7 | 0.9 |
| NPC_I_60_GFRP_360d | 74.3 | 946.6 | 10.7 | 56.0 | 1.6 |
| NPC_I_60_GFRP_540d | 65.2 | 829.7 | 6.8 | 58.6 | 2.4 |

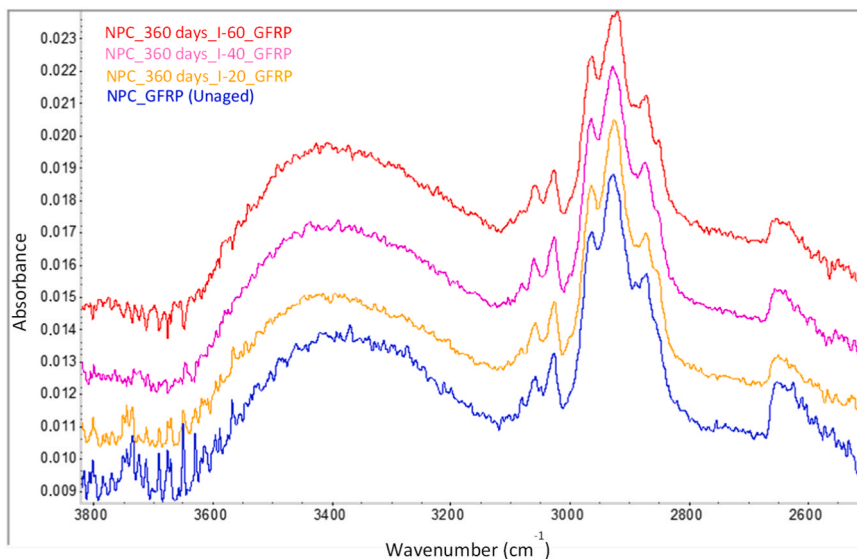


Fig. 12. FTIR spectra of NPC GFRP specimens in initial unaged state, and after 360 days of exposure to alkaline solution at various temperatures.

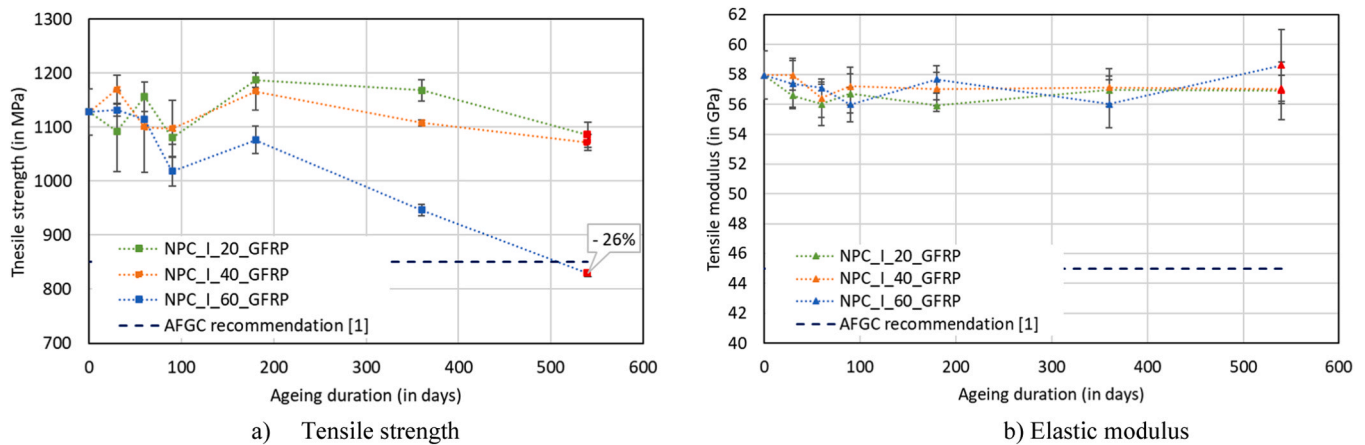


Fig. 13. Changes in tensile mechanical properties of NPC GFRP rebars during indirect immersion (I) in alkaline solution at various temperatures and without load (ANL).

reduction of approximately 26 % after 540 days), highlighting substantial degradation process within the aged GFRP rebars in accordance with previous SEM observations of fiber damage. Notably, for these specimens, the residual tensile strength falls below the minimum requirement recommended by AFGC (850 MPa) [1].

Interestingly, the elastic tensile modulus remains unaffected by the ageing conditions at any of the considered temperatures, which is consistent with existing literature [11].

3.2.2.3. Impact on residual ILSS. The residual interlaminar properties of the NPC GFRP specimens were also assessed via short-beam tests following exposure to indirect alkaline immersion. The observed failure mode of all aged specimens closely resembled that of the initial specimens, characterized by interlaminar fracture at the “neutral fiber” (see Fig. 6b).

The residual ILSS of the rebars measured after various ageing periods are presented in Table 6. The observed variations over time are illustrated in Fig. 14.

The results demonstrate an increase in ILSS for NPC GFRP rebars after 200 days of ageing, showing improvements of up to 31 % for specimens aged at 60°C for 540 days. This trend aligns with the post-curing process of the matrix observed during ageing at various temperatures as evidenced by DSC, although the kinetics of evolution do not

Table 6

Residual Interlaminar shear strength (ILSS) of NPC GFRP rebars after different durations of indirect immersion (I) in alkaline solution at various temperatures.

| Specimen designation | Ultimate load (kN) | ILSS (MPa) | |
|----------------------|--------------------|------------|--------------------|
| | | Mean value | Standard deviation |
| NPC_GFRP (unaged) | 5.4 | 45.6 | 1.9 |
| NPC_I_20_GFRP_30d | 5.4 | 45.9 | 2.1 |
| NPC_I_20_GFRP_60d | 5.4 | 46.2 | 2.9 |
| NPC_I_20_GFRP_90d | 5.3 | 44.7 | 0.5 |
| NPC_I_20_GFRP_180d | 5.1 | 43.5 | 0.3 |
| NPC_I_20_GFRP_360d | 6.5 | 54.9 | 2.2 |
| NPC_I_20_GFRP_540d | 6.7 | 56.9 | 1.2 |
| NPC_I_40_GFRP_30d | 5.9 | 49.9 | 3.5 |
| NPC_I_40_GFRP_60d | 5.4 | 46.2 | 1.9 |
| NPC_I_40_GFRP_90d | 5.9 | 49.7 | 1.5 |
| NPC_I_40_GFRP_180d | 5.5 | 46.6 | 2.5 |
| NPC_I_40_GFRP-360d | 7.1 | 60.7 | 1.1 |
| NPC_I_40_GFRP_540d | 6.5 | 55.5 | 1.3 |
| NPC_I-60_GFRP_30d | 5.6 | 47.1 | 1.1 |
| NPC_I_60_GFRP_60d | 5.8 | 48.9 | 2.5 |
| NPC_I_60_GFRP_90d | 5.9 | 50.4 | 0.9 |
| NPC_I_60_GFRP_180d | 5.8 | 49.3 | 1.1 |
| NPC_I_60_GFRP_360d | 6.0 | 51.2 | 4.9 |
| NPC_I_60_GFRP_540d | 7.0 | 59.9 | 0.9 |

correlate precisely with those observed for Tg (refer to Fig. 9). Generally, it is established that a higher curing ratio leads to enhancements in matrix-dominated properties, particularly in ILSS [17]. Overall, the residual ILSS values obtained for all aged samples significantly exceed the minimum requirement of 40 MPa specified by AFGC for GFRP rebars [1].

3.2.3. Comparative effects of Direct and Indirect alkaline immersion without load (ANL)

Since some of the NPC GFRP specimens underwent direct and indirect alkaline immersion for comparable durations (up to 90 days), it allows for a comparison of the effects of these exposure conditions on various properties of the partially cured GFRP rebars. Fig. 15 illustrates the comparative evolutions of Tg, tensile strength, and ILSS for specimens subjected to direct and indirect ageing over 90 days.

Different trends can be drawn from this Figure:

- The increase in Tg attributed to the post-curing effect is notably more pronounced with direct immersion in the alkaline solution at 60°C compared to indirect ageing, especially noticeable between 40 and 90 days of exposure (Fig. 15a). Direct immersion likely promotes the absorption of water molecules into the rebars, thereby enhancing the plasticization of the polymer network during ageing. This increased molecular mobility may facilitate the diffusion of residual monomers and, consequently, the post-curing process in these rebars compared to specimens embedded in concrete.
- The decrease in tensile strength over exposure time appears to happen at a higher rate for specimens subjected to indirect ageing through the intermediate concrete cover, compared to direct immersion (Fig. 15b). This trend was unexpected, as the opposite phenomenon is generally reported in the literature, based on the restriction of diffusion of deleterious species within the intermediate concrete medium [13]. This discrepancy may be attributed to the utilization of partially cured rebars in this study. Further investigation is required to elucidate the underlying mechanism.
- The increase in ILSS attributed to the post-curing process over the ageing period is more pronounced for specimens subjected to direct immersion (Fig. 15c). This could be attributed to the influence of the ageing condition on the post-curing process, as evidenced by the discrepancies in Tg values observed previously.

3.3. Durability of GFRP rebars subjected to indirect alkaline ageing with sustained load (ASL)

3.3.1. Impact of ASL conditions on residual tensile properties

To investigate the influence of sustained loading during ageing, some

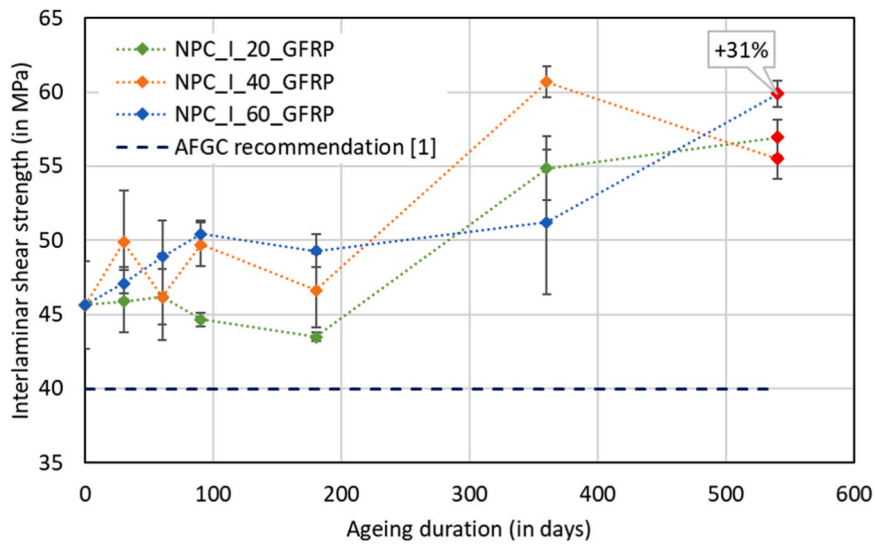
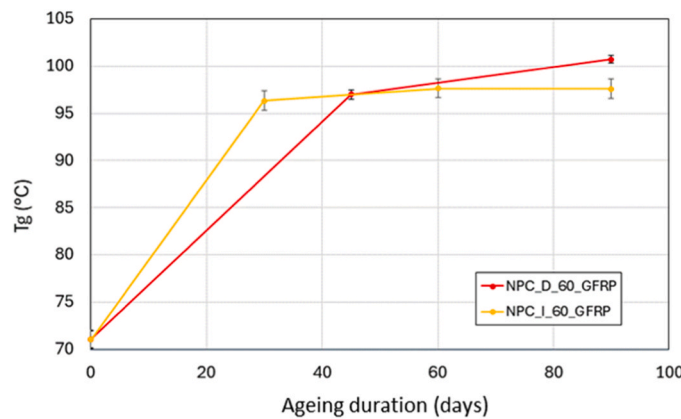
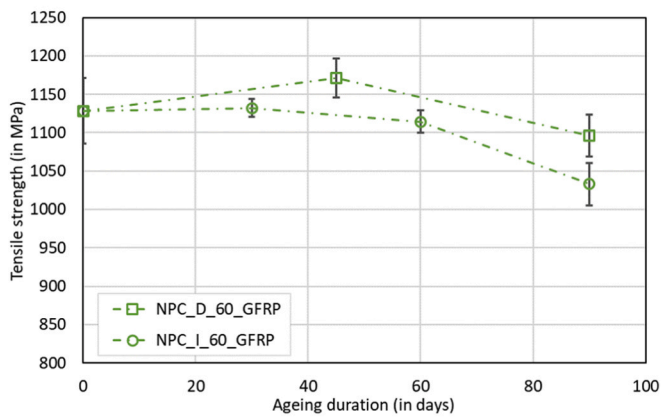


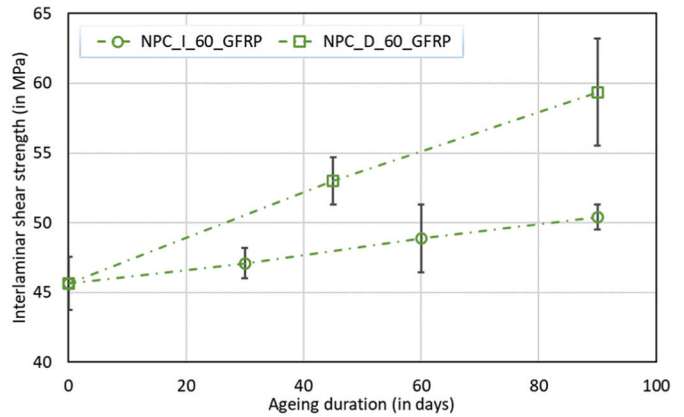
Fig. 14. Changes in residual ILSS of NPC GFRP rebars during indirect immersion (I) in alkaline solution at various temperatures and without load (ANL).



(a)



(b)



(c)

Fig. 15. Comparative effects of direct (D) and indirect (I) alkaline immersion at 60°C on Tg (a), residual tensile strength (b) and residual ILSS of NPC GFRP rebars exposed for duration up to 90 days (c).

NPC GFRP specimens were exposed to combined environmental and mechanical conditions (referred to as ASL). Ageing was conducted under indirect immersion in the alkaline solution at various temperatures, with a maximum duration of 90 days.

Noteworthy, several GFRP specimens exposed to the most severe conditions (40°C and 60°C with a sustained load equal to 40 % of the initial tensile strength) experienced premature failure (in creep) during

the ageing process after average durations of 70 and 12 days, respectively. As a result, these rebars were unable to undergo tensile testing afterward but were subsequently tested in short beam tests.

The average residual tensile properties of the GFRP rebars that did not fail during ASL ageing are summarized in Table 7. Additionally, Fig. 16 compares the residual tensile strengths of the GFRP specimens aged under ASL and ANL conditions (with and without combined load).

For specimens that experienced premature failure during ASL ageing, their residual strengths are also depicted, with their values considered to be the level of sustained load applied when they failed. These specific results are indicated as square markers on the graph.

Experimental results demonstrate that the application of sustained load during ageing significantly impacts the residual strength of the NPC GFRP rebars, with values obtained after ASL exposure being lower than those under ANL conditions. Moreover, this effect exhibits high temperature dependency, with the combined load exerting a greater influence on the residual strength of the GFRPs at higher ageing temperatures. Interestingly, this influence is minimal at room temperature (20°C) and becomes even negligible after 90 days of exposure.

Overall, the observed influence of the load is found to be more significant compared to most of the results reported in the literature [31]. For instance, in the present study, a decrease of 24 % in tensile strength was recorded at 40°C and 40 % of the ultimate load after 42 days, whereas in [20], a decrease of only 18 % was observed after 60 days at a similar load level combined with direct alkaline immersion. Hence, it is plausible that the incomplete curing of the GFRP rebar exacerbates the coupling effect between load and temperature in the alkaline environment. This may be due to the higher creep sensitivity of partially cured GFRPs at elevated temperatures. Further investigations are needed to confirm this hypothesis.

Furthermore, similar to the results under ANL conditions and in accordance with literature data, the elastic modulus does not appear to be affected by ASL ageing (see Table 7).

3.3.2. Impact of ASL on residual ILSS

This section focuses on NPC_GFRP specimens aged under the ASL condition (60°C/40 % of the initial tensile strength and 40°C/40 % of the initial tensile strength) that experienced premature failure after 12 days and 70 days of exposure during the ageing process. Although the failure did not occur near the anchorages, portions of the rebars remained intact and could be subjected to short beam tests to evaluate their interlaminar properties. For each sample, three interlaminar shear tests could be carried out.

The average ILSS values for this series of specimens are presented in Table 8, along with the corresponding calculated standard deviations. In Fig. 17, a comparison is made between these ILSS value and those obtained for specimens aged under ANL conditions at the same temperatures (indirect immersions at 40°C and 60°C).

The experimental results reveal that at 40°C, residual ILSS values exhibit similar trends under both ASL and ANL conditions. Conversely, at 60°C, the ILSS value of the ASL-aged specimen significantly exceeds that of the ANL-aged specimens, approaching the value previously obtained for the PC specimens in Section 3.1.2 (58.9 MPa). Such enhancement in ILSS may result from an indirect effect of GFRP creep occurring in the early period after load application, exacerbated by the

Table 7
Residual tensile properties of the NPC_GFRP rebars aged under ASL conditions.

| Sample designation | Ultimate load (kN) | f_u (MPa) | | E_L (GPa) | |
|--------------------------------|--------------------|-------------|--------------------|-------------|--------------------|
| | | Mean | Standard deviation | Mean | Standard deviation |
| NPC_GFRP (unaged) | 88.6 | 1128.3 | 42.8 | 58.0 | 1.6 |
| ASL_NPC_I_20_20 % _GFRP_28d | 79.0 | 1006.1 | 38.9 | 56.0 | 0.9 |
| ASL_NPC_I_20_40 % _GFRP_28d | 85.0 | 1082.9 | 21.1 | 55.8 | 0.1 |
| ASL_NPC_I_20_40 % _GFRP_90d | 85.8 | 1091.9 | 9.0 | 57.4 | 0.6 |
| ASL_NPC_I_40_40 % _GFRP_16d | 79.8 | 1016.5 | 13.3 | 56.4 | 1.0 |
| ASL_NPC_I_40_40 % _GFRP_42d | 67.5 | 859.1 | 50.6 | 56.2 | 1.1 |

low initial Tg value that barely exceeds the temperature of the ageing solution in this case. Such creep effect may promote local rearrangement of the fibers (enhanced alignment) and radial contraction of the bar leading to a confinement of the fiber/matrix interface. The reloading procedure employed to compensate the load loss induced by the initial relaxation stage, may also contribute to the phenomenon at this ageing temperature of 60°C. Nevertheless, the observed improvement in ILSS did not prevent creep failure from occurring during ageing. In light of the limited experimental results available on this matter, further investigations are definitely needed.

3.3.3. Complementary physico-chemical characterizations

Additional investigations were carried out by SEM-EDX and FTIR analyses on specimens subjected to combined environmental/mechanical ageing (ASL) and that failed in creep. Fig. 18 depicts images of a typical cross-section of a sample that experienced creep failure after 12 day-exposure to indirect alkaline ageing at 60°C combined with 40 % of the ultimate load. Although extensive cracks are observed, the precise underlying degradation mechanism could not be determined. EDX and FTIR characterizations did not reveal any evidence of alkali ion diffusion into the rebars or any signs of chemical modification of the vinyl ester matrix, which does not support the hypothesis of a chemical degradation process. It cannot be excluded that these cracks resulted from the release of elastic energy during creep failure

4. Conclusions

This article presents experimental investigations on the durability of partially cured GFRP rebars subjected to accelerated ageing in alkaline environments, or to the combination of alkaline environment and sustained load. Based on the obtained results, the following conclusions can be drawn. However, further research involving more in-depth statistical analysis is recommended to strengthen these findings.

- The initial incomplete curing of the GFRP rebars did not compromise their initial tensile properties, as these latter were found to be very close to those of post-cured specimens. However, the curing state significantly affected the initial ILSS of the rebars, which is a matrix-dominated property (lower curing ratios resulted in lower ILSS values, consistent with findings in the literature). Nevertheless, all measured properties exceeded the minimum requirements specified by the French AFGC guidelines for GFRP rebars [1].
- Durability tests conducted via direct immersion in an alkaline solution at 60°C, without load, revealed no detrimental effects on the tensile properties of undercured or post-cured GFRP rebars over a 90-day exposure period. Undercured specimens underwent significant crosslinking during ageing, evidenced by the rise in Tg observed via DSC, with their Tg value becoming comparable to that of the post-cured specimens by the end of the period. This phenomenon led to a significant improvement in ILSS, with its value converging toward that of the post-cured GFRPs. Additionally, no matrix degradation or diffusion of alkali ions was detected during ageing. Nevertheless, it would be interesting to complement the study with longer ageing durations.
- Further investigations were conducted by subjecting partially cured GFRPs to indirect alkaline immersion, with the rebars embedded in an intermediate concrete cylinder, at different temperatures and without load, for extended exposure periods. Results revealed a decrease in tensile strength of approximately 4 %, 5 %, and 26 % after 540 days of ageing at temperatures of 20°C, 40°C, and 60°C, respectively. Signs of diffuse fiber damage were detected by SEM observations, which could explain the observed effects after long term exposures. The observed reductions in residual tensile strength align with existing literature, emphasizing the importance of limiting the average service temperature for GFRP rebars. Additionally, an

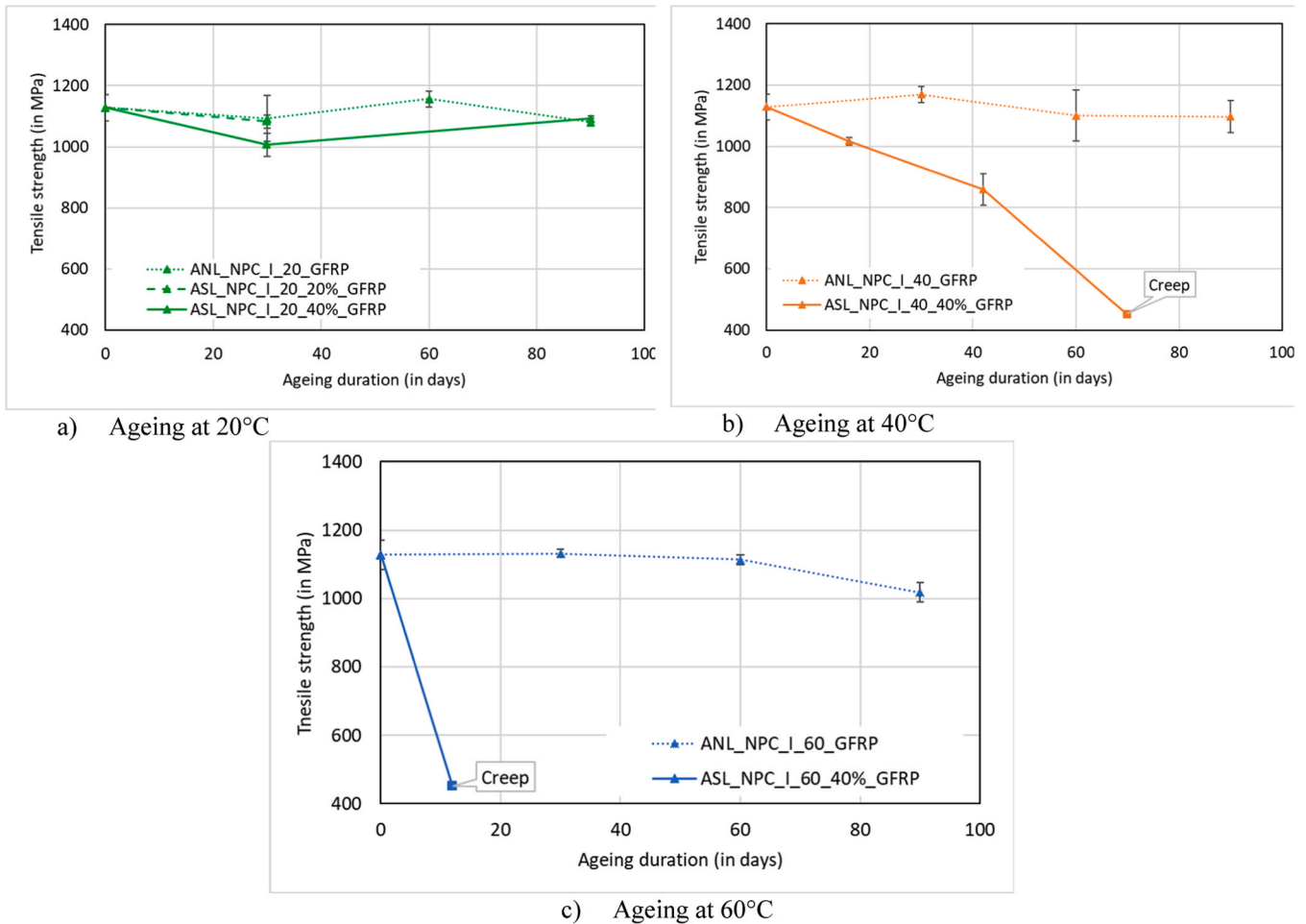


Fig. 16. Comparative evolutions of the residual tensile strength of NPC_GFRP rebars under ASL conditions (indirect alkaline ageing with combined load) and under ANL conditions (indirect immersion without load) at different temperatures.

Table 8
Residual interlaminar shear strength (ILSS) of NPC_GFRP rebars aged under ASL conditions.

| Specimen Designation | Ultimate load (kN) | ILSS (MPa) | |
|----------------------------|--------------------|------------|--------------------|
| | | Mean value | Standard deviation |
| NPC_GFRP (unaged) | 5.4 | 45.6 | 1.9 |
| ASL_NPC_I_60_40 %_GFRP_12d | 6.8 | 57.7 | 1.2 |
| ASL_NPC_I_40_40 %_GFRP_70d | 5.1 | 43.2 | 5.5 |

increase in ILSS was observed over ageing, once again related to a post-curing process as indicated by the evolutions of T_g.

- Finally, a last series of partially cured GFRP specimens was exposed to indirect alkaline immersion at various temperatures, combined with sustained load, for durations of up to 90 days. The results revealed significant effects of the applied load on the residual tensile strength, with a notable temperature dependency. Indeed, these effects were most pronounced at 60°C, where they even led to rapid creep failure, but remained minimal at 20°C. Overall, the influence of load appeared to be more pronounced than that observed in some studies from the literature on fully cured GFRP rebars, indicating that incomplete curing may exacerbate synergistic effects under these combined ageing conditions, possibly due to increased creep sensitivity at elevated temperature. The results confirm the necessity of

limiting permanent stress to prevent creep failure, as recommended in most guidelines. For instance, ACI 440.R1-15 recommended a limit equal to 20% of the design tensile strength [30], which has recently been updated to 30% in ACI CODE 440.11-22 [32]. Further investigations are needed to develop predictive approaches that can account for both environmental factors and applied stress.

Throughout this study, it is noteworthy that the elastic tensile modulus of the rebar remained unaffected by ageing. This consistency aligns with existing literature and current design guidelines, which do not consider degradation of the elastic modulus for durability concerns [1].

Additionally, it is important to note that changes in ILSS over ageing are typically inconsistent with the evolution of tensile properties. This discrepancy arises because ILSS reflects the fiber/matrix bond strength between and is strongly influenced by post-curing effects. Therefore, ILSS measurement should not be relied upon as the most reliable indicator when assessing the durability of GFRP rebars. Nevertheless, ILSS remains the most commonly used test method in field durability investigations due to its ease of implementation. Further research is necessary to identify parameters correlated with tensile strength that could be more readily measured in the field.

Finally, the conducted investigations underscore the need to improve quality control of FRP rebars, to increase confidence in the final materials, and promote their wider deployment on site. Specifically, emphasis should be placed on controlling T_g specifications, as this parameter is believed to strongly impact durability under combined

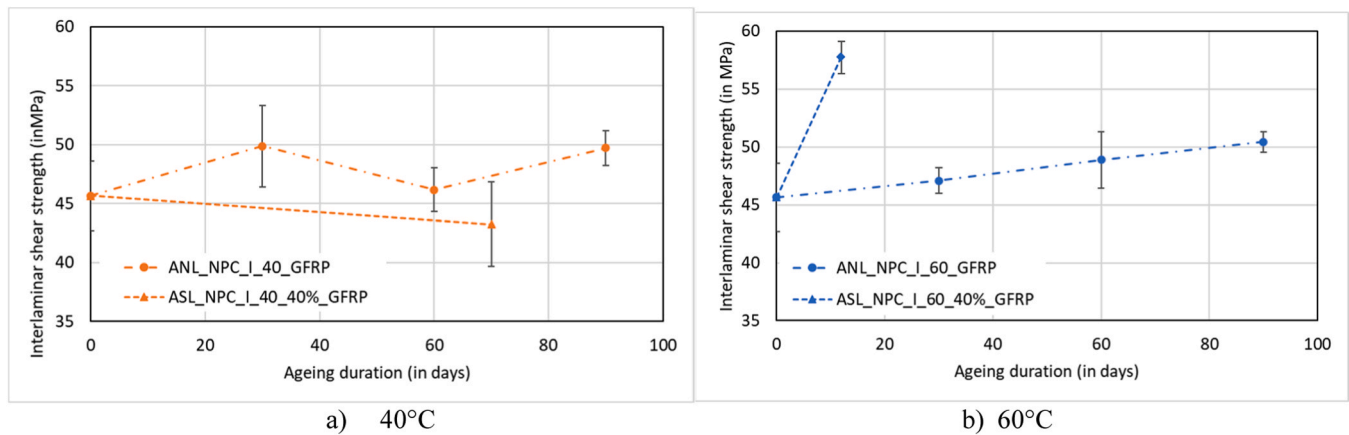


Fig. 17. Comparative evolution of the residual ILSS of NPC_GFRPs under ASL conditions (indirect immersion at 60° and 40°C with 40 % sustained load) and under ANL conditions (indirect immersion without load).

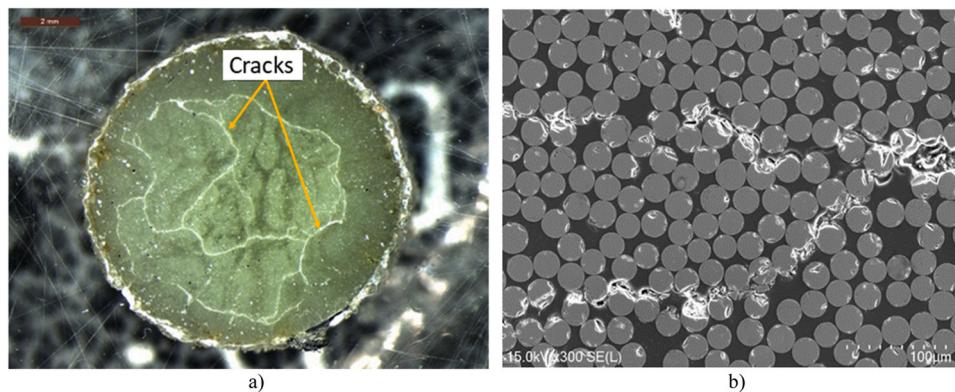


Fig. 18. Cross-sectional images of an NPC GFRP rebar post-creep failure (60°C, 40%), captured at a distance exceeding 3 cm from the failure point - optical microscopy (a) and SEM observation (b) revealing the presence of cracks.

alkaline attack and sustained load.

CRediT authorship contribution statement

Noémie Delaplanque: Writing – original draft, Methodology, Investigation, Formal analysis. **Maxime Tharreau:** Investigation, Formal analysis. **Laurent Gaillet:** Validation, Supervision. **Xavier Bourbon:** Writing – review & editing, Validation, Supervision, Project administration, Funding acquisition, Conceptualization. **Arnaud Rolland:** Validation, Supervision, Methodology. **Karim Benzarti:** Writing – review & editing, Validation, Supervision, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization. **Ludwig Batais:** Investigation. **Sylvain Chataigner:** Writing – review & editing, Visualization, Validation, Supervision, Project administration, Methodology, Investigation, Funding acquisition, Conceptualization. **Marc Quiertant:** Writing – review & editing, Validation, Supervision, Project administration, Methodology, Funding acquisition, Conceptualization.

Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: N. Delaplanque reports financial support and equipment, drugs, or supplies were provided by Andra. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability

Data will be made available on request.

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