Ageing of pultruded glass fibre reinforced polymer composites exposed to combined environmental agents

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1. Introduction

Fibre Reinforced Polymer (FRP) composites are currently used in several civil engineering applications ranging from reinforcing rods and tendons, wraps for seismic retrofit of columns and externally bonded reinforcement for strengthening of walls, beams, and slabs, composite bridge decks, hybrid and all-composites structural systems. Their advantages in these applications are: low weight, high stiffness-to-weight and strength-to-weight ratios, ease of installation and potentially high overall durability. Properties of FRP are relatively known to scientists and engineers, but there are still many concerns about their durability and their performance under severe environmental conditions [1].

Several environmental agents have been identified as the cause of the ageing mechanisms that occur within FRPs. The currently available investigations in literature are mainly limited to the combination of moisture [2–4] or water absorption [5–7] with one of the environmental agents: low temperature (freeze–thaw cycles [8,9]), elevated temperature [10,11], ultraviolet (UV) radiation [12,13]. Frequently, harsh environments have been simulated with immersion in water solutions, such as alkali [14] or salt [15]. On the contrary, the combination of different environmental agents (as in natural exposition) is not in-deep investigated [16], in the authors' knowledge. This is the main objective of the present investigation.

The research detailed in this paper proposes an artificial ageing procedure and provides an understanding of the combined effect of relevant environmental agents on some mechanical and aesthetical properties of GFRP materials produced with different resins. Materials have been exposed to a combination of elevated and sub-zero temperatures, moisture (high levels of relative humidity close to the saturation grade) and UV radiation. Three commercially available GFRP pultruded profiles have been tested: isophthalic polyester/E-glass, orthophthalic polyester/E-glass and vinylester/E-glass.

Variations of the mechanical and aesthetical performance have been measured over 24 consecutive weeks (six months) of accelerated ageing in a climatic chamber and tested every four weeks (monthly interval). Tensile and flexural modulus and strength, interlaminar shear strength (ILSS) and change in lightness and colour have been measured and compared to those of the un-aged materials.

Moreover, the experimental study compares the values collected with the artificial ageing with those from specimens exposed to natural ageing in external atmosphere in the city of Milan (Italy). The artificial ageing has been defined with the aim of creating similar effects, both for type and magnitude, to those

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obtained in a natural environment of a semi-continental large city. The specimens, exposed to the natural climate, have been tested after twelve months.

2. Materials

Pultruded plates have been produced using E-glass fibres and three different compositions of matrices: isophthalic polyester, orthophthalic polyester and vinylester. Heat deflection temperature (HDT) for the resins, measured by the producer, is 90 °C for isophthalic polyester, 80 °C for orthophthalic polyester and 105 °C for vinylester. Fibre reinforcement consists of unidirectional direct roving of 4800 tex and continuous filament mat of 300 g/m^2.

Organic peroxide has been used as catalyser in each resin to have the same reactivity and the same process speed for all formulations (50 cm/min). In the heated moulds the temperature was set to get ≈95% of the complete polymerization inside the mould (according to the Differential Scanning Calorimeter tests of the producer).

The fibre volume fraction is equal to ≈60% for the three compositions, of which ≈48% is unidirectional roving and ≈12% is mat. Test specimens have been extracted from pultruded plates having average thickness of ≈2.85 mm and width of ≈310 mm. The plates stacking sequence has two external layers of continuous filament mat and one internal layer of unidirectional (UD) roving. A polyester veil is applied on the top (Fig. 1). It represents the 0.7% of the weight. The producer adopts the veil as protection of the internal glass fibres from atmospheric agents. The side of the specimens with the polyester veil has been exposed to the artificial UV radiation within the climatic chamber and to the sunlight during the process of natural ageing.

3. Experimental details

3.1. Artificial and natural ageing

Artificial ageing has been performed using a Binder MKF-720 climatic chamber (Fig. 2) for 24 consecutive weeks (six months). The ageing consists of 504 cycles divided in 252 'cold' and 252 'warm'. In the 'cold' cycle the temperatures range from −20 °C to +20 °C and the level of relative humidity (RH) is set to 90%, as detailed in Fig. 3a. In the 'warm' cycle the temperatures range from +20 °C to +60 °C and the level of relative humidity is set to 80% (see Fig. 3b). The adopted artificial cycles intend to simulate the extreme weather conditions of a semi-continental climate, characterized by a cold/wet winter and a hot/wet summer.

The single cycle had a period of 8 h and the 'cold' and 'warm' cycles have been alternated every 21 cycles (one week). Temperature and humidity have been controlled by the sensors equipment within the climatic chamber. The relative humidity, due to technical limits of the climatic chamber, has not been recorded below 10 °C.

The climatic chamber has been equipped with a set of four fluorescent UV lamps (Fig. 2). According to the standard [17], UV lamps emit on a wavelength in the range of 340–400 nm with a peak for 350 nm, and produce a UV irradiance of 40 W/m^2 on the specimen surface.

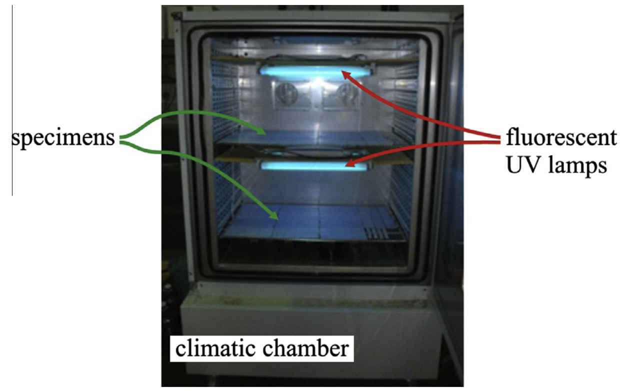

Fig. 2. Inside of the Binder MKF-720 climatic chamber equipped with fluorescent UV lamps.

The lamps were Philips Actinic BL 40 W Reflector, two for each layer of specimens (Fig. 2). The lamps have been used for 4 h in each cycle to simulate the daylight and substituted every 500 h to prevent an excessive decay of the UV flux emitted in the specimen direction.

Other specimens have been exposed to natural ageing in the external environment of Milan. Pultruded plates of the three compositions have been placed on a dark horizontal roof, 10 m above the ground, for twelve consecutive months (Fig. 4). Highest and lowest temperatures and relative humidity have been collected from a weather data centre close to the site of exposition and detailed in Fig. 5 for two years, including the natural exposition period. Weather data of the last decade have been used to properly define the artificial ageing set-up. Maximum temperature of 60 °C in the warm cycle has been selected both considering literature studies [18] and the temperature on a surface exposed to the direct sunlight during summer. Minimum temperature of −20 °C has been chosen considering the extreme sub-zero temperatures recorded in Milan in the last decade.

3.2. Mechanical tests

GFRP specimens have been used to measure the influence of both artificial and natural ageing on some of the main mechanical properties: tensile, flexural and interlaminar shear strength. Tests have been performed both in the roving direction (0°) and perpendicularly to it (90°).

Specimens used for tensile test were according to the standard [19]. Five specimens have been tested both for 0° and 90° direction. A MTS 647 hydraulic machine with a load cell of 100 kN has been used. The strain in load direction has been recorded by an extensometer MTS 634.25F-24, base length of 50 mm. The test speed was set to 2 mm/min according to [19].

Specimens for the flexural test have been prepared according to the Class III of the standard [20]. Five specimens have been tested both for 0° and 90° direction. A MTS 358 hydraulic machine with a load cell of 2.5 kN has been used. The three-point loading arrangement had span of 40 mm. A test speed of 2 mm/min was selected according to [20].

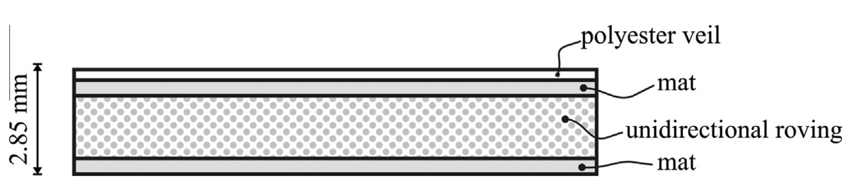

Fig. 1. Layup of the pultruded plates.

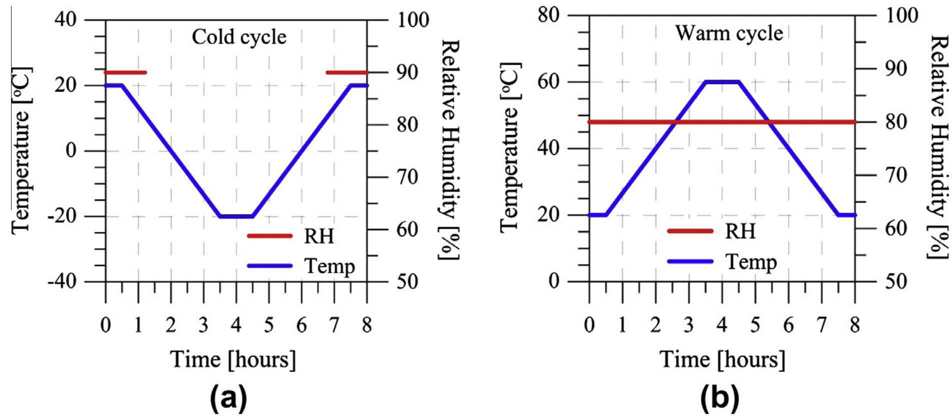

Fig. 3. Temperature and relative humidity for (a) 'cold' and (b) 'warm' cycles.

Fig. 4. Specimens exposed to natural weathering, on a horizontal roof (a) during a summer day and (b) a winter day.

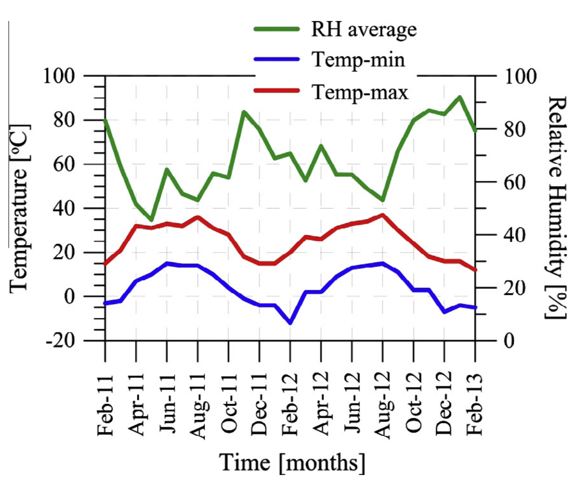

Fig. 5. Weather data recorded close to the site of exposition in two years, February 2011–February 2013.

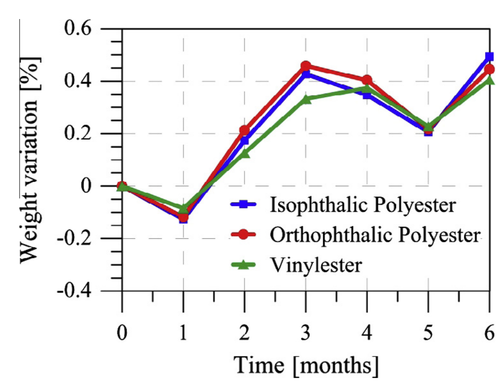

Fig. 6. Weight variation during artificial ageing.

Five specimens for each material have been cut along the roving direction (0°) for ILSS test, according to the standard [21]. The MTS 358 hydraulic machine, above mentioned, has been adopted. The short-beam had supports distance of 15 mm. The test speed was set to 1 mm/min according to [21].

The present investigation involved 600 mechanical tests including those for the un-aged materials.

3.3. Spectrophotometry

Over the past years several examples both in buildings and bridges construction showed GFRP cladding panels or structural profiles without painting or coating the external surface. The atmospheric ageing could produce variation of colour and hence an aesthetical degradation. Therefore, the modifications of the surface lightness and colour have been evaluated using a Minolta CM-2600d spectrophotometer. Results have been expressed in terms of the CIE colour space $L^*/a^*/b^*$ according to the standard [22]. In this system, L^* refers to the measurement of lightness, while a^* and b^* give the colour components in the green-red and blue-yellow range, respectively.

4. Results

The influence of the ageing is summarized considering the materials un-aged (as produced) and conditioned in the artificial environment. Each month of the six of artificial ageing the

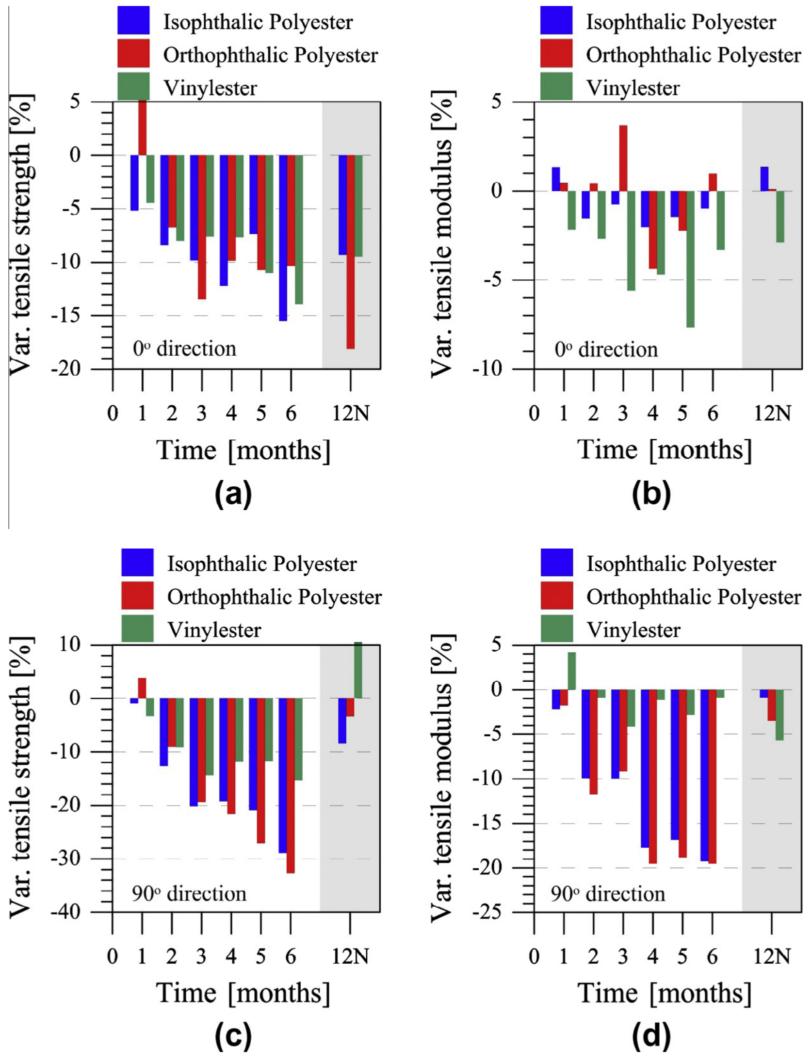

Fig. 7. Results of the tensile tests. Variation of the average strength (a and c) and of the average tensile modulus (b and d); in 0° (a and b) and 90° (c and d) direction. '12 N' means 12 months of natural ageing.

mechanical and aesthetical properties have been measured. Moreover, the imparted modifications have been compared to those of the materials exposed to the natural environment for 12 months.

4.1. Weight variation

One of the main responsible of the mechanical properties degradation, presented in literature [2–7,10,11,14,15], is the coupling of water (moisture) absorption and elevated temperature. The moisture uptake, during environmental exposition, is considered an indication of the possible damage imparted, e.g. matrix cracks and debonding at the interface between fibres and matrix [23,24]. It has been also observed that the rate of moisture absorption at elevated temperatures is higher than at ambient temperature [25,26]. Therefore, a preliminary evaluation of the effects of the relative humidity and temperature within the climatic chamber has been performed weighting five specimens after each month of artificial ageing. Specimens have been carefully wiped to remove any superficial condensation and eventual residual dust. Weight variations with respect to the un-aged material have been calculated as: $100 \times (m(t) - m(0))/m(0)$, being $m(0)$ and $m(t)$ specimen mass at the initial state and at the time t [7].

The average measurements detailed in Fig. 6 show a reduction (≈0.1%) in the first month. This initial behaviour can be explained as a consequence of loss of styrene, as observed in [5]. In the second and third month, the increase of the moisture absorption generates a progressive weight gain (0.4%). This weight increment reflects an increase of the water absorption capacity as consequence of the growth of crack density allowing water permeation [5]. In the last three months, the specimens weight has an oscillation, it decreases in the fifth and recovers in the sixth month. Probably, for a longer period, the weight becomes stationary about to the increment of 0.4%.

All three compositions behave similarly to the moisture absorption, even though vinylester matrix absorbs less moisture if compared to polyester based materials [23].

It must be underlined that the mentioned results available in literature are consequence of constant conditions (humidity and/or temperature), while present measurements represent the effect of cyclic variation of humidity and temperature.

4.2. Mechanical tests

4.2.1. Tensile properties

Results of tensile test have been summarized in Fig. 7. Diagrams show the percentage variations after artificial ageing of the average tensile strength and tensile modulus with respect to the un-aged materials (Table 1). Moreover, Fig. 7 collects

Table 1
Mechanical properties of un-aged composite materials.

Matrix	Direction	Mechanical property	Average	Stan. Dev.
Isophthalic polyester	0°	Tensile strength (MPa)	489.15	13.82
		Tensile modulus (GPa)	31.11	0.67
		Flexural strength (MPa)	472.62	17.26
		Flexural modulus (GPa)	13.33	0.15
		ILSS (MPa)	28.06	1.12
	90°	Tensile strength (MPa)	47.20	1.55
		Tensile modulus (GPa)	8.63	0.36
		Flexural strength (MPa)	131.29	10.34
		Flexural modulus (GPa)	7.64	0.44
Orthophthalic polyester	0°	Tensile strength (MPa)	493.55	25.93
		Tensile modulus (GPa)	31.36	0.74
		Flexural strength (MPa)	485.13	15.03
		Flexural modulus (GPa)	13.51	1.25
		ILSS (MPa)	28.15	1.18
	90°	Tensile strength (MPa)	43.74	3.94
		Tensile modulus (GPa)	7.61	0.32
		Flexural strength (MPa)	116.24	18.73
		Flexural modulus (GPa)	6.71	0.30
Vinylester	0°	Tensile strength (MPa)	503.61	11.84
		Tensile modulus (GPa)	32.72	0.47
		Flexural strength (MPa)	534.78	35.79
		Flexural modulus (GPa)	15.82	0.53
		ILSS (MPa)	46.46	0.60
	90°	Tensile strength (MPa)	49.69	3.64
		Tensile modulus (GPa)	10.91	0.23
		Flexural strength (MPa)	127.27	15.03
		Flexural modulus (GPa)	8.34	0.22

the tensile properties after twelve months of natural ageing (12 N).

Tensile strength in roving direction (0°) (Fig. 7a) shows the larger part of the reduction in the initial three months of the ageing, it has slight variation in the fourth and fifth month and a further reduction in the remaining ageing time, for any of the three matrix. After six months, the composite with isophthalic polyester matrix has a strength loss of 15.5%, orthophthalic polyester of 10.4% and vinylester of 13.9%. Twelve months of natural ageing produces reduction of strength in roving direction lower than the artificial ageing both for isophthalic polyester and vinylester (≈9.5%), while for the orthophthalic polyester is about 18.1%.

Tensile modulus in roving direction (0°) (Fig. 7b) does not vary significantly; the differences are in the range of the experimental data scatter band (standard deviations are in the range 0.4–2.3 GPa). A comparable behaviour is observed after twelve months of natural ageing. Therefore, in the roving direction only the tensile strength varies, mainly in the initial three months of artificial ageing, and the effect is similar to that of the natural ageing for a double time of exposition.

Tests in the matrix dominant direction (90°) (Fig. 7c) show a continuous reduction of the tensile strength, faster in the first half and in the last month of the artificial ageing. After six months, the reduction for composite with isophthalic polyester is 28.9%, for orthophthalic is 32.7% and the vinylester has a degradation of 15.3% of the initial value. Twelve months of natural ageing do not show a relevant impact on the transverse strength.

In the transverse to the roving direction, it is quite important the effect of the artificial ageing on the tensile modulus (Fig. 7d). Both polyester matrices based composites have a considerable decrease of stiffness, close to 20% of the un-aged value. The glass/vinylester composite shows a slightly variable stiffness, within the experimental standard deviations (0.2–0.9 GPa). This result is expected being vinylester hydrolytically more stable than polyesters [27]. The twelve months natural ageing has not relevant effect on the 90° direction tensile modulus.

In the matrix dominant direction, the artificial ageing produces a considerable degradation of the tensile properties. This effect is not observed after twelve months of natural ageing.

4.2.2. Flexural properties

Results of the bending tests are detailed in Fig. 8 in term of variations with respect to the un-aged materials (Table 1). The flexural strength in roving direction (0°) has an initial increase for the isophthalic polyester, a reduction in the second and third month of artificial ageing and then remains stable. After six months, a reduction of 14.8% and 10.5% is recorded for the orthophthalic polyester and the vinylester based composites, respectively. The same trend is observed after twelve months of natural ageing with slight lower amount of strength loss (Fig. 8a).

The flexural modulus in roving direction (0°) is unaffected by the ageing; the differences are in the range of the experimental data scatter band (standard deviations are in the range 0.1–1.2 GPa), as observed in tensile tests (Fig. 8b). After twelve months of natural ageing, the flexural modulus shows a negative trend with comparable reductions respect to the artificial ageing. As for the tensile properties, the natural and artificial ageing demonstrate similar influence on the bending behaviour in roving direction.

Analogous comments arise for the bending properties in the matrix dominant direction (90°). Considerable reduction of the flexural strength is observed in the first half of the artificial ageing (Fig. 8c), even though in the first month an evident improvement is recorded. This is probably consequence of a further polymerization [28] and/or a hardening effect [8] of the matrices due to the high and low temperature imposed in the climate chamber. A minor influence of these factors is evident in the tensile properties. The flexural modulus in 90° direction does not show an evident effect of ageing (Fig. 8d). The materials have a final reduction below 6%, in the experimental scatter band (standard deviations are in the range 0.1–0.9 GPa). The twelve months of natural ageing produces a similar effect.

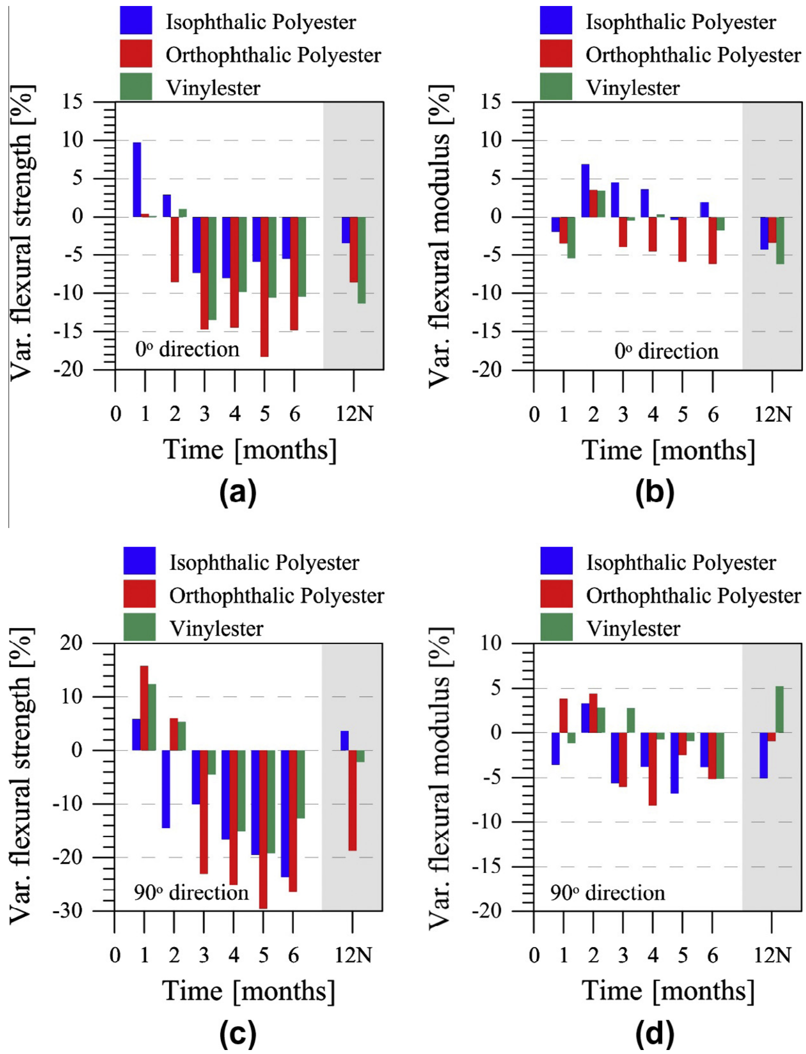

Fig. 8. Results of the flexural tests. Variation of the average strength (a and c) and of the average modulus (b and d); in 0° (a and b) and 90° (c and d) direction. '12 N' means 12 months of natural ageing.

4.2.3. Interlaminar shear strength

The interlaminar shear tests, in the roving direction, provide the strength variations detailed in Fig. 9, with respect to the un-aged materials (Table 1). The variation of the ILSS reveals different behaviour for the considered matrices. The polyester based specimens have an increase higher than 10% after the first month of artificial ageing. As mentioned above this could be the effect of a further polymerization and/or hardening of the matrices. In the following five months a continuous decrease of ILSS is observed, with a residual positive variation lower than 3%. The vinylester based composite has an initial slight improvement of the strength and a fast degradation until the third month, than maintaining a reduction close to 8%. The natural ageing produces a similar trend for orthophthalic polyester and vinylester matrices, while the isophthalic polyester has an enhancement of 13%.

Fig. 10 shows microscopic images of damaging in two specimens of the orthophthalic polyester/E-glass composite. The loading introduces a longitudinal crack at the mid thickness both in the un-aged specimen and in the material artificially aged for two months. In the latter, beside the main crack, several small cracks are visible (Fig. 10b). These originate from the micro damage imparted during ageing and are responsible of the ILSS reduction discussed above. Similar damage patterns have been observed in the other two materials under consideration.

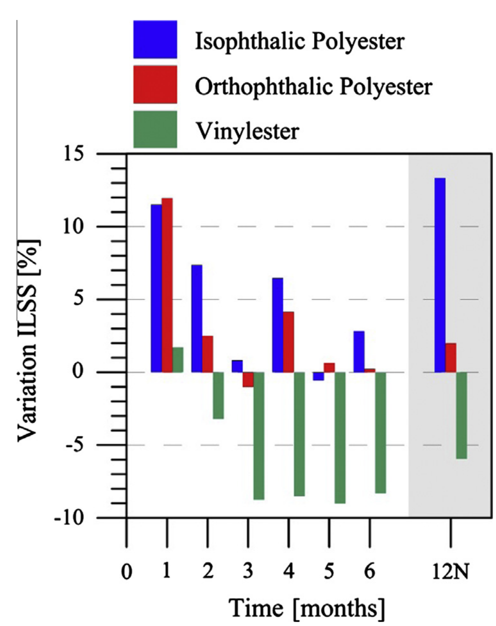

Fig. 9. Results of the inter-laminar shear tests. Variation of the average ILSS in 0° direction. '12 N' means 12 months of natural ageing.

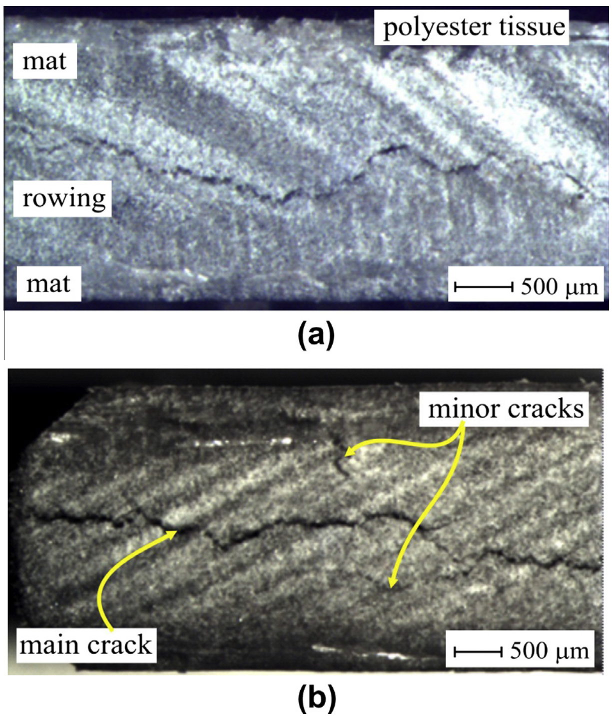

Fig. 10. Orthophthalic polyester/E-glass composite: failure mode after interlaminar shear test of (a) un-aged specimen and (b) two months artificial aged specimen.

4.3. Colour characterization

Exposure to UV light causes degradation of polymeric constituents through a mechanism of photo-degradation. This, amongst other effects, produces discoloration and surface oxidation [16].

Fig. 11a presents the lightness measurements (L^*) for each month of exposition to artificial ageing. The diagram reveals small variations for all the three GFRP compositions. The slight increase is limited to one unit for both polyester matrixes [29], while vinylester maintain the same lightness in the six months ageing. A loss of one unit of lightness (L^*) represents a visible variation for the human eye. The natural ageing produces an analogous lightness increase in the polyester matrix composites (≈2 units), while the vinylester one has a reduction of 4 units.

Measurements of the colours variation have been detailed in Fig. 11b and c. These show a slight degradation of the green part of the colour and a tendency to red for the three composites (a^*, Fig. 11b), during the artificial ageing. At the same time an increase of the yellow component has been observed (b^*, Fig. 11c). In this case, the variation of b^* is close to 3 units for both materials with polyester matrices. The composite with vinylester matrix exhibits a lower variation along both the red-green and blue-yellow axes. The natural ageing does not affect the green part of the colour in the polyester composites and shows the same trend for the vinylester (Fig. 11b). On the blue-yellow scale, the natural exposition generates 5 and 10 units of increment of the yellow component for the polyester and vinylester composites, respectively.

The discrepancies in the lightness and colours variations between natural and artificial exposition, mainly for vinylester com-

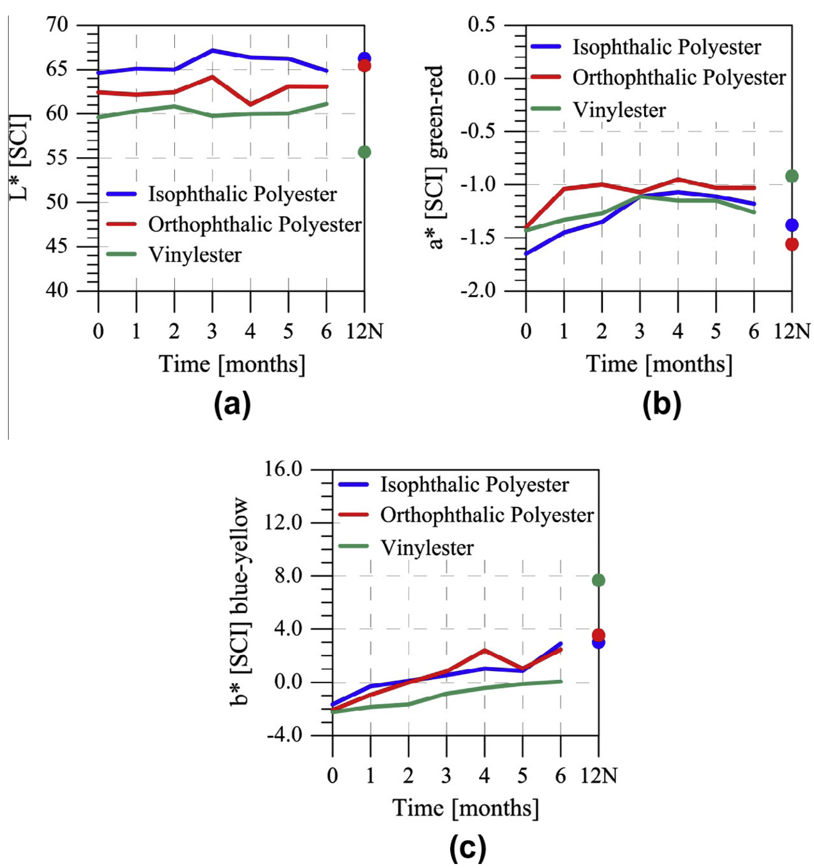

Fig. 11. (a) lightness (L^*); colour variation in the (b) green-red-range (a^*) and (c) blue-yellow range (b^*). '12 N' means 12 months of natural ageing. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

posite, can be motivated with the exposition to the UV wavelength that does not cover the complete spectrum of the sunlight.

5. Discussion and conclusions

An overview of the mechanical tests detailed in this paper allows getting some observations and conclusions. The mechanical features, mainly strengths, have slight variation in the first month of artificial ageing (Figs. 7–9). The diagrams show a faster degradation in the second and third month. In the following months, the composite materials have a slower reduction or a stabilization of the measured properties. Comparing this behaviour to the weight variation (humidity absorption) in Fig. 6, several links are evident. The loss of weight in the first month, explained as a loss of styrene [5] and further polymerization of the matrices, produces the small increase or negligible reduction of the mechanical properties in this ageing period. In the second and third months the materials have the larger increment of weight (Fig. 6) as consequence of the increase of cracks and moisture uptake. This generates the larger reduction of the strength mentioned above. The weight is stable in the fourth month of artificial ageing, while it has an oscillation over the same value in the last two months. This observation can be explained with a stable or slightly variable pattern of cracks already imparted. The counterpart in term of mechanical properties is a lower reduction or invariance.

The six month combination of high temperature, freeze–thaw cycles, relative humidity and UV radiation imposed in the climatic chamber has similar effect of the twelve months natural ageing in the roving direction. As expected, the artificial ageing accelerates the reduction of the mechanical properties in the matrix dominant direction of the GFRP profiles. A correlation between the artificial and the natural ageing is not straightforward. This can be the aim of a future theoretical modelling.

As main conclusion, the combination of weathering agents generates a greater influence on the mechanical performance of GFRP materials if compared to literature data obtained through the application of a single or a couple of environmental agents [8–13].

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