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Long-term bending performance and service life prediction of pultruded Glass Fibre Reinforced Polymer composites

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ABSTRACT

This paper presents an experimental investigation on the long-term mechanical performance, focusing on bending, of pultruded composite plates adopted as cladding in building constructions. Three matrices have been used in combination with E-glass fibres: isophthalic polyester, orthophthalic polyester and vinylester.

The degradation of the mechanical performance has been monitored through a process of accelerated artificial ageing by using water baths set at different temperatures: 20 °C, 40 °C and 60 °C.

Flexural strength retention through time has been used as measurement of the imparted degradation and water absorption has been considered to define the diffusion coefficient and the activation energy for the three compositions. The Arrhenius methodology has been adopted to estimate the long-term degradation of the composites and to predict the expected service life of the materials. The outputs of the research show that isophthalic and vinylester systems have flexural strength retention of 65% for more than 75 and 100 years, respectively, while the orthophthalic composite has the same retention for more than 20 years.

1. Introduction

Fibre Reinforced Polymer (FRP) composites in recent years have known a widespread diffusion in several building construction applications [1]. This is due to the clear advantages they offer in terms of low weight, high stiffness-to-weight and strength-to-weight ratios, ease of installation and potentially high overall durability. Their diffusion has as counterpart the lack of available data on the behaviour in service conditions [2]. Methodologies are still not generally shared to account for the effects of long-term exposition to environmental agents and, most importantly, to estimate their expected service life.

The effects of environmental agents on the mechanical behaviour of composites adopted in building construction were not extensively detailed in the literature as in other industrial applications. The variation of shear-axial stress interaction in CFRP tendons for prestressed concrete as a result of exposure to aggressive solution environments (water, salt water and concrete pore solution) was experimentally investigated in [3]. In [4,5], the moisture absorption behaviour and associated mechanical degradation of unidirectional hybrid (carbon/glass) composite rods for overhead conductors was evaluated. The researches in [6,7] studied the moisture diffusion process in GFRP composites for a bridge deck and in pultruded profiles for structural applications, respectively. The effects of seawater and warm environment was investigated e.g. in [8], while extreme temperatures were assessed in [9], as well as the consequences of exposure to acid solutions were considered for GFRP bars in [10].

Those investigations, and others in the literature, provide a limited knowledge of the degradation of composites in building construction due to environmental effects and the prediction of the expected service life of the materials is still a challenge.

The scope of the present research is to give a contribution in predicting the service life of GFRP and in understanding the behaviour of different matrices. An Arrhenius related methodology [11] has been used to predict the long-term behaviour of three commercially available pultruded GFRP plates adopted as cladding in building constructions: Isophthalic polyester/E-glass, Orthophthalic polyester/E-glass and Vinylester/E-glass. Variations of the flexural strength for the materials, after artificial ageing in water baths at both ambient and warm temperatures (20 °C, 40 °C and 60 °C), have been used to predict long-term mechanical degradation. Variations of the strength retention through time

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have been considered as measurement of the materials ageing and, by defining a performance related threshold, it has been possible to estimate the expected service life of the three materials composition. A comparison of the mechanical performance of the composites immersed in water, with data collected through natural ageing [12] has been also proposed to compare the magnitude of the imparted degrade in water with that in a semi-continental natural environment. The natural ageing was imparted in the environment of a semi-continental city (Milan – Italy) placing the pultruded plates of the three compositions on a dark horizontal roof, 10 m above the ground, for twelve consecutive months [12].

2. Materials

The plates have been produced by pultrusion technique using E-glass fibres and three different compositions of matrices: isophthalic polyester, orthophthalic polyester and vinylester. The Heat Deflection Temperature (HDT) of the resins, measured by the producer, is 90 °C for isophthalic polyester, 85 °C for orthophthalic polyester and 105 °C for vinylester. Fibre reinforcement consists of unidirectional roving of 4800 tex and continuous filament mat of 300 g/m^2. The materials are the same adopted in the experimental investigation detailed in [12].

Organic peroxide has been used as catalyser in each resin to obtain the same reactivity and the same manufacturing speed for all formulations (50 cm/min). The temperatures of the linear mould have been set to get ≈95% of the complete polymerization during the pultrusion process (according to the Differential Scanning Calorimeter tests of the producer).

The fibre volume fraction within the profiles was almost 60% for the three compositions, of which ≈48% is unidirectional roving and ≈12% is mat. Test specimens have been cut from plates having average thickness of ≈2.85 mm and width of ≈310 mm. The plates stacking sequence had two external layers of continuous filament mat and one internal layer of unidirectional (UD) roving. A polyester veil, representing the 0.7% of the weight, has been applied on the top (Fig. 1), the external side of the cladding. The producer adopted the veil as a protection of the internal glass fibres from atmospheric agents.

3. Experimental details

3.1. Artificial ageing

The application of the Arrhenius model to the study of FRPs long-term performance is based on the exposure of the materials to the effect of different temperatures, ranging from room to warm, within an aqueous environment [11].

In the present work the specimens have been completely immersed within water baths containing de-ionised water and maintained for several months at three constant temperatures: +20 °C, +40 °C and +60 °C.

These temperatures, and in particular the highest, have been selected according to relevant literature studies [13–15] and considering as reference the values provided by the manufacturer for the Heat Deflection Temperature (HDT) of the materials. In fact the maximum testing temperature in the water baths should not exceed 80% of the transition temperature T_g of the composite materials [11]. The T_g for the three matrices has been evaluated by the producer to be about 10 °C lower than the HDT, therefore equal to 75 °C for the orthophthalic polyester, 80 °C for the isophthalic polyester and 95 °C for the vinylester.

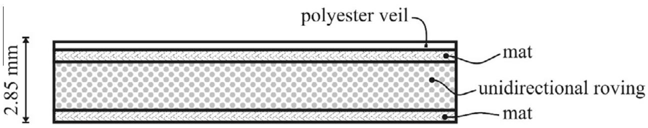

Fig. 1. Stack sequence for the GFRP plates.

Fig. 2. The FALC WB-M15 thermostatic bath used for conditioning of the specimens.

The specimens have been maintained at the described temperatures within a FALC WB-MD15 thermostatic water bath. This apparatus has 12 litres capacity and is able to operate in the range +20 °C to +120 °C with a tolerance of ±1 °C, Fig. 2. The specimens have been disposed on a supporting equipment to allow both an adequate exposure of the edges to water, and avoid direct contact of the materials with the metal base of the water bath. The temperature of the water has been additionally monitored with an external thermometer to ensure to be maintained constant at the prescribed values; moreover the container has been constantly covered with a polycarbonate top to avoid excessive evaporation of the water.

The conditioning periods of the specimens with the three different matrices are reported in Table 1, in detail: 12 months at 20 °C, 12 months at 40 °C and 6 months at 60 °C. Specimens at 60 °C have been conditioned for a shorter time frame being the materials degradation already significant after six months exposition.

3.2. Mechanical tests

GFRP specimens have been tested to measure the influence of ageing on flexural properties. Tests have been performed in roving direction (0°), with the polyester veil in the compression zone of the cross section. This test setup has been selected according to the main loading condition of the considered panels adopted as external cladding in constructions.

Table 1
Period of the immersion in water for the specimens at different temperatures.

Water temperature	GFRP composite	Period of immersion
20 °C	Isophthalic/E-glass	12 months
	Orthophthalic/E-glass	12 months
	Vinylester/E-glass	12 months
40 °C	Isophthalic/E-glass	12 months
	Orthophthalic/E-glass	12 months
	Vinylester/E-glass	12 months
60 °C	Isophthalic/E-glass	6 months
	Orthophthalic/E-glass	6 months
	Vinylester/E-glass	6 months

Table 2
Time intervals for the weight measurement and the mechanical testing of the three GFRP composites immersed in water at different temperatures.

Water temperature	GFRP composite	Interval of testing
20 °C	Isophthalic/E-glass Orthophthalic/E-glass Vinylester/E-glass	0 month 1 month 6 months 9 months 12 months
40 °C	Isophthalic/E-glass Orthophthalic/E-glass Vinylester/E-glass	0 month 1 month 5 months 8 months 11 months 12 months
60 °C	Isophthalic/E-glass Orthophthalic/E-glass Vinylester/E-glass	0 month 1 month 4 months 5 months 6 months

Specimens for the flexural test have been prepared according to the Class III of the standard [16]. Four specimens have been tested for each time interval, as detailed in Table 2. A MTS 358 hydraulic machine with a load cell of 2.5 kN has been used for the three-point loading arrangement with supports span of 40 mm. A test speed of 2 mm/min has been selected according to [16].

4. Testing results

The influence of the ageing has been monitored by comparing the materials un-aged (as produced) with those conditioned in the water baths. The specimens have been weighed and then tested, as extracted from the bath, according to the time intervals in Table 2.

4.1. Weight variation

As extensively discussed in the literature [5,17,18] one of the main causes of the reduction of the mechanical performance in FRPs is the coupling effect of water (or moisture) absorption and elevated temperatures. Hence the assessment of the weight variation is essential to better understand the behaviour of the material ageing.

Four specimens for each GFRP material have been weighed according to the time intervals in Table 2. These have been carefully wiped prior to weighing to remove any superficial water and eventual residual dust.

Weight variations with respect to the un-aged materials have been calculated as (see e.g. [19]):

$$\Delta_m = 100 \times (m(t) - m(0))/m(0) \tag{1}$$

where $m(0)$ and $m(t)$ are respectively the specimen mass at the initial state and at the time t.

The average weights for the specimens with the three matrices immersed at 20 °C are detailed in Fig. 3a, those related to the specimens immersed at 40 °C are reported in Fig. 3b, while for 60 °C are shown in Fig. 3c, including polynomial fitting with coefficient of correlation in the range 0.92–0.99.

In the case of 20 °C, Fig. 3a, the weight grows until the three materials reach almost the saturation, after about ten months. The weight variation at the saturation is close to 1.2% for both the polyester matrices while it is lower for the vinylester matrix

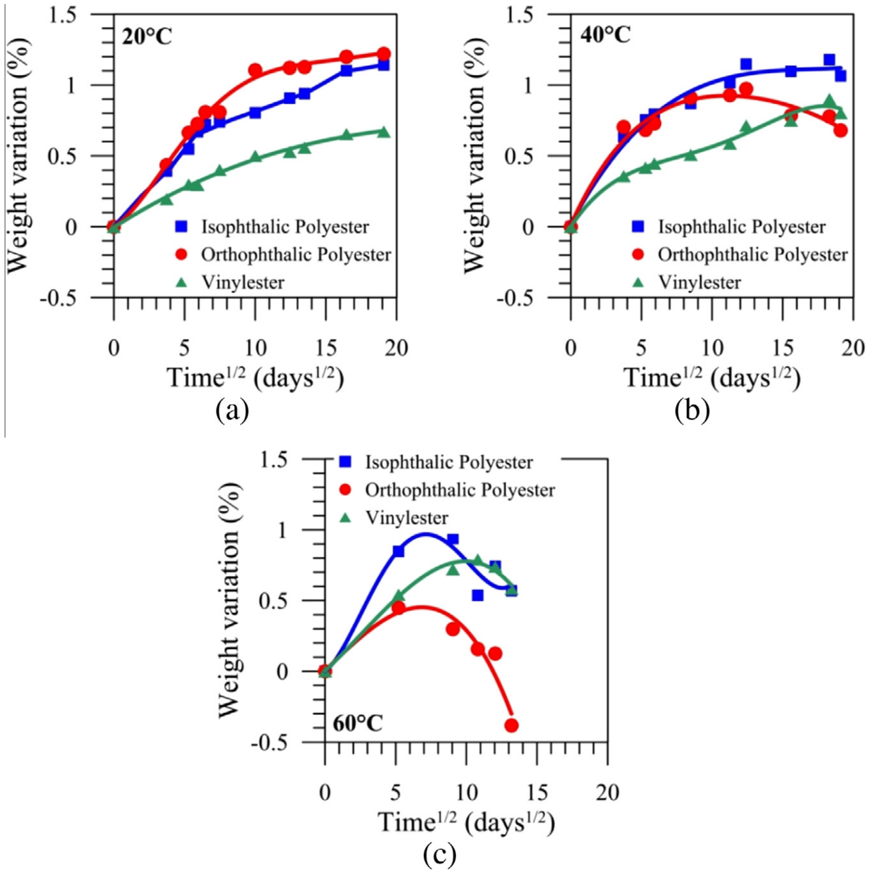

Fig. 3. Weight variation versus square root of time for the GFRP specimens immersed in water at: (a) 20 °C, (b) 40 °C and (c) 60 °C.

($\sim$0.6%). This is in agreement with the lower water permeability of the vinylester respect to polyester matrices [20].

At 40 °C, Fig. 3b, the weight of the specimens increases constantly for the initial months and then it shows a reduction in the final months of conditioning. This is particularly evident for the specimens of orthophthalic polyester that start losing weight after five months of immersion. The reduction in weight is less evident in the case of both the isophthalic polyester and the vinylester matrices, and it is limited to the last month of exposition.

The response of the FRPs immersed in deionised water is roughly Fickian in nature [21,22] at both 20 °C and 40 °C (see the initial linear trend of curves in Fig. 3a and b). For long exposure, the weights reach almost a plateau showing the level of moisture uptake at saturation [14].

For specimens immersed at 60 °C, Fig. 3c, a different behaviour has been observed. In particular, the isophthalic polyester showed a consistent water uptake during the initial period of immersion (higher than those showed for the other two temperatures) followed by a quite consistent drop. The weight of the specimens after six months reduces to $\sim$0.55% after being increased to $\sim$1% at the initial stage of exposure. The orthophthalic polyester matrix is even more affected by the warm temperature (Fig. 3c). The composite initially showed a water uptake, reaching a weight increase of $\sim$0.45% after one month, then a continuous and rapid loss resulting in global reduction of about $-$0.35% at the end of the experimentation (six months). This behaviour was already discussed in the literature for warm temperatures [23]. The vinylester matrix, instead, showed a constant mass growing, only decreasing after five months of immersion.

A possible explanation of the observed behaviour is related to the imposed temperature (60 °C), high enough to generate a consistent degradation of both the polyester matrices. This generates an increase of failure in the chemical bonding within the matrix and then a significant loss in weight. This phenomenon is even more evident in the case of the orthophthalic polyester for its inherent chemical and physical stability responsible of the Heat Deflection Temperature, and T_g, lower than the other two matrices.

As it has been observed in the literature [11,23], the matrix degradation is more severe for specimens immersed in water respect to specimens conditioned in air despite this phenomenon has not been observed from the authors, at least not with this magnitude, during the exposure of the GFRP specimens to both natural atmosphere and to artificial ageing within a climatic chamber [12].

Furthermore it has been mentioned in the literature that the increase of water uptake in composites produces plasticization in the short-term, and hydrolysis by attack on the ester linkage in the long-term [24]. Both these processes result in a higher induced molecular mobility and, as a consequence, a decrease of the T_g [25,4]. Therefore the water temperature of 60 °C affected the orthophthalic matrix with a probable reduction of the T_g.

4.2. Mechanical testing

Initial values for the flexural strength of the three GFRP compositions in roving direction (0°) are reported in Table 3. They refer to the specimens un-aged and have been used to assess the variation of the mechanical performance.

Table 3
Flexural strength in the roving direction (0°) for the un-aged composites.

GFRP composite	Average (MPa)	Stan. Dev. (MPa)
Isophthalic Polyester/E-glass	472.62	17.26
Orthophthalic Polyester/E-glass	485.13	15.03
Vinylester/E-glass	534.78	35.79

Diagrams in Fig. 4 show the percentage variation after conditioning in the water baths of the average flexural strength with respect to the un-aged materials.

It can be generally observed that the flexural strength shows a consistent reduction for the three temperatures. At 20 °C, Fig. 4a, it has been noticed a decrease, starting after the first month until the sixth month, with a maximum reduction of 18.8% for the isophthalic polyester, 17.3% for the orthophthalic polyester and 16.2% for the vinylester. After six months it has been noticed a slight recover for all composites until the end of the experimentation. This can be consequence of the slight strength recovery of the resin as observed in [23]. Twelve months of ageing generated a flexural strength decrease of 8.3%, 9.7% and 9.1% for the isophthalic polyester, the orthophthalic polyester and the vinylester, respectively.

Specimens immersed in water at 40 °C, Fig. 4b, show a continuous reduction of the mechanical performance until the end on the experimentation with a total final loss, after twelve months, of 36.0% in the case of the isophthalic polyester, 37.4% for the orthophthalic polyester and 32.7% for the vinylester.

The behaviour for specimens immersed at 60 °C, Fig. 4c, shows an even more consistent reduction after four months of immersion, if compared to the previous two cases. At the end of the ageing, after sixth months, the total loss for the isophthalic polyester was 38.7%, for the orthophthalic polyester was 31.5% and 40.7% for the vinylester. The last had a peak of $-$43.3% after five months ageing.

By comparing the results obtained for the weight variation, in Fig. 3, and those for the variation of the flexural strength, in Fig. 4, it seems visible a correlation existing between the water uptake and the variation of mechanical performance for GFRP composites, as observed in [12,26]. In particular, the values for the water absorption are the highest for the isophthalic and orthophthalic polyester both at 20 °C and 40 °C. As counterpart they show the highest reduction of the mechanical performance than vinylester composite after six months at 20 °C and 40 °C. In the second six months of testing a similar trend is observed with a higher decrease of performance for the orthophthalic polyester at both temperatures. Nevertheless, after the complete exposition at 40 °C the weight of the orthophthalic polyester starts slightly reducing with respect to the six months value (see Fig. 3b). This apparent contradiction can be justified having on one side an increase of weight due to water absorption and, on the other, a higher reduction of weight due to the loss of molecular components.

At 60 °C, instead, the water uptake for the orthophthalic polyester is lower than both the isophthalic and the vinylester, as a consequence it has been noticed a lower reduction of the mechanical performance.

5. Long-term performance prediction

5.1. Apparent diffusion coefficient and activation energy

The activation energy E_a represents an indication of the energy barrier that has to be overcome for diffusion of moisture to take place within the composite. The following Arrhenius type relationship can be used to calculate it [14,27]:

$$D = D_0 \exp\left(\frac{-E_a}{RT}\right) \tag{2}$$

where D_0 is a constant coefficient; R is the universal gas constant equivalent to 8,3144 J/mol * K; D is the apparent diffusion coefficient, and T is the temperature in Kelvin. Therefore, the curve ln(D) versus ($1/T$) provides the activation energy (E_a) of the composites.

The value of the apparent diffusion coefficient, D, can be determined assuming a moisture absorption process according to the Ficḱs law [21,22]. Hence it is obtained by (see e.g. [28]).

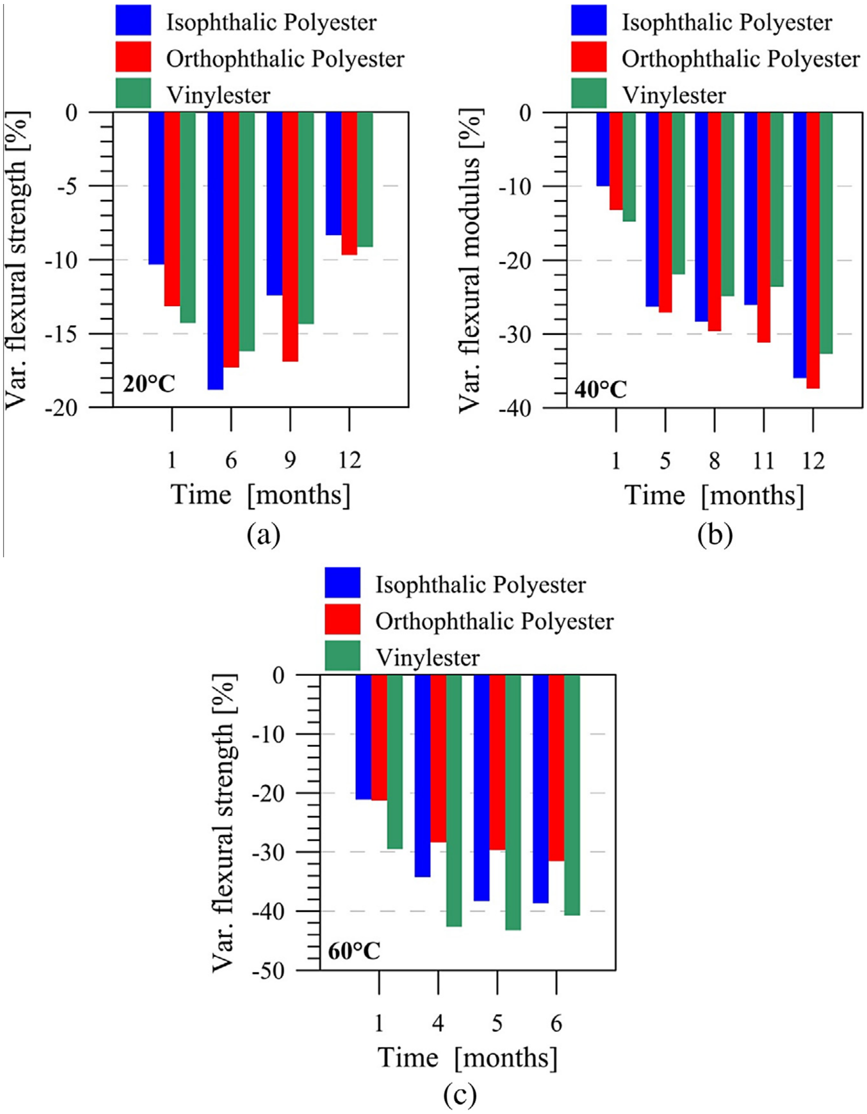

Fig. 4. Results of the three point bending test. Variation of the average flexural strength in roving direction for (a) 20 °C, (b) 40 °C and (c) 60 °C.

$$D = \pi\left(\frac{h}{4M_m}\right)^2\left(\frac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}}\right)^2\left(1 + \frac{h}{l} + \frac{h}{n}\right)^{-2} \qquad (3)$$

where h, l and n are the thickness, the length, and the width of the specimens, respectively. M_1 and M_2 are the moisture contents at times t_1 and t_2; with t_1 and t_2 being sufficiently small to assume the weight varying with $\sqrt{t}$. M_m represents the maximum moisture content absorbed during the immersion period.

Eq. (3), assuming the obtained weight variations, provides the values of the diffusion coefficients for the three GFRP composites listed in Table 4. As expected, the diffusion coefficient increases when increasing the temperature.

Comparing the diffusion coefficients obtained for the three composites, the vinylester one shows the lowest values, while the isophthalic polyester composite has a lower value with respect to the orthophthalic polyester.

Fig. 5 shows the diagrams ln(D) versus temperature in Kelvin. The activation energy for the composites is obtained as the slope of the linear fitting divided by the Boltzman constant. All fitting curves have a coefficient of correlation $R^2 > 0.98$, meaning a very good reliability.

Table 4
Apparent diffusion coefficient for the three GFRP composites at different temperatures.

GFRP composite	Apparent diffusivity coefficient (mm^2/s)		
	20 °C	40 °C	60 °C
Isophthalic polyester/E-glass	1.0×10^{-7}	1.7×10^{-7}	3.6×10^{-7}
Orthophthalic polyester/E-glass	1.1×10^{-7}	2.1×10^{-7}	3.6×10^{-7}
Vinylester/E-glass	7.6×10^{-8}	1.4×10^{-7}	2.8×10^{-7}

The activation energy for the isophthalic polyester based GFRP, for the orthophthalic polyester and for the vinylester are 26.1 kJ/mol, 24.8 kJ/mol and 26.7 kJ/mol respectively.

The lower activation energy obtained for the orthophthalic matrix indicates a weaker diffusion barrier and, as consequence, a greater absorption of water for the composite, a higher degradation of the matrix and a worst long-term mechanical performance. This was observed in the mechanical tests summarized in Fig. 4a and b. On the contrary, the higher values for the isophthalic polyester and the vinylester indicate a lower degradation.

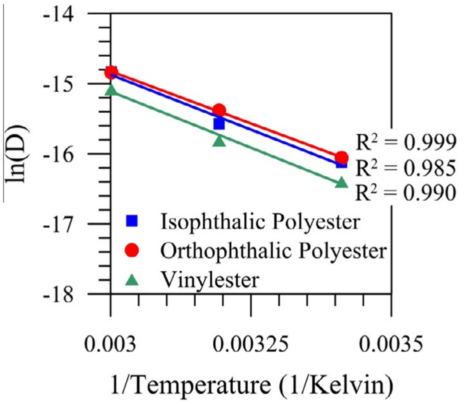

Fig. 5. Activation energy for the three composites.

5.2. Arrhenius plots

The long-term mechanical performance of the composite materials can be predicted assuming the Arrhenius principle. It states that the rate at which the chemical degradation occurs is dependent on temperature [11]. If the rate of change in mechanical properties in the service environment is known, then the service life of the material can be estimated. The model is described by [29]:

$$k = Ae^{-E_a/RT} \tag{4}$$

where k is the rate of degradation, A is a pre-exponential factor characteristic of the failure mechanism and test conditions, T is the absolute temperature in Kelvin at the time of failure, E_a is the activation energy, as previously calculated, and R is the Boltzman constant.

A first step requires calculating the experimental property retention values for the three composites. Fig. 6 shows the retention of the flexural strength for the three considered composites.

The Arrhenius plots, in Fig. 7, are obtained setting the property retention as function of the inverse absolute temperature for various selected lifetimes. This is achieved substituting the time values, representing service life times, into the regression equations in Fig. 6.

As from Fig. 7, using the equations of the regression lines and selecting a specific temperature it is possible to define the strength retention at the desired time. Therefore, knowing the minimum acceptable value of the flexural strength retention for a specific application, the diagram provides the expected service life for the three GFRP composite materials.

6. Service life predictions of the GFRP pultruded materials

To assess the long-term performance of the GFRP materials in wet environments and to derive the property retention over time, it is necessary to set up an appropriate temperature. This is normally assumed as the average service temperature, considering the annual average service temperature for the specific location in which the GFRP materials are exposed. In literature an average temperature of 23 °C has been proposed [24].

Table 5 details the flexural strength retention of the three GFRP compositions for different life times at 23 °C, as derived using the Arrhenius plots in Fig. 7.

Table 5 mentions the experimental flexural strength retention calculated after 1 year of natural ageing in [12]: ~96% for the isophthalic polyester, ~91% for the orthophthalic polyester and ~89% for the vinylester.

The comparison shows a higher theoretical degradation using the Arrhenius plots than in a natural environment. This depends on the higher rate of degradation of a coupon immersed in water

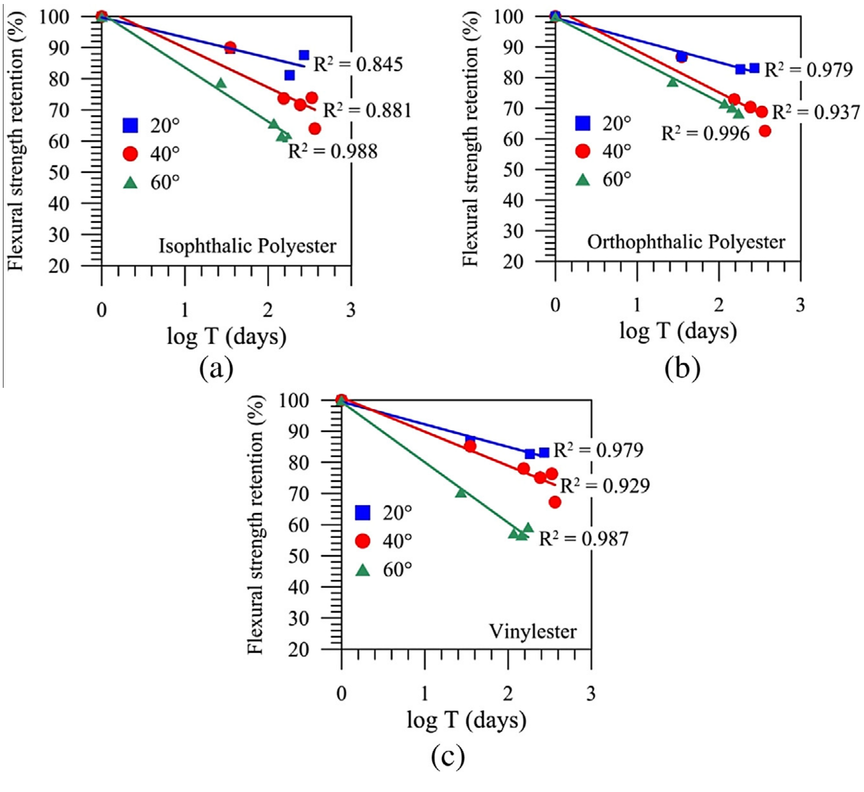

Fig. 6. Flexural strength retention versus time for different temperatures. (a) Isophthalic polyester/E-glass, (b) orthophthalic polyester/E-glass and (c) vinylester/E-glass.

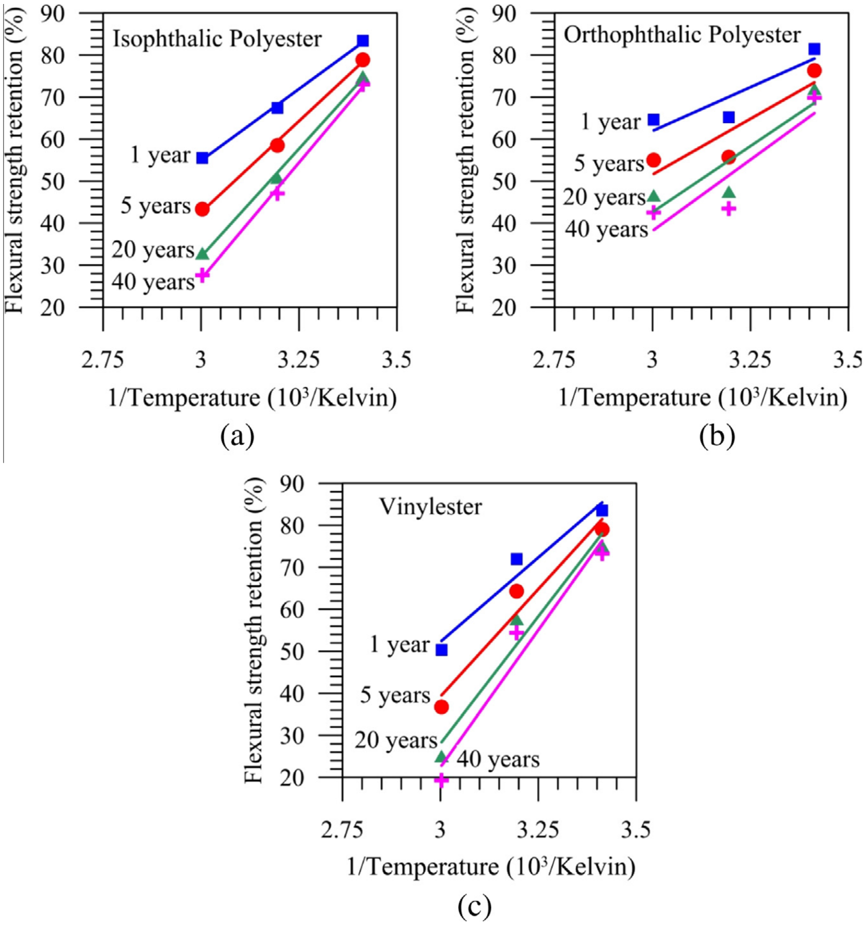

Fig. 7. Arrhenius plots of the flexural strength retention for various lifetimes. (a) Isophthalic polyester/E-glass, (b) orthophthalic polyester/E-glass and (c) vinylester/E-glass.

Table 5
Theoretical value of the flexural strength retention for the three composites at 23 °C in the roving direction (0°). In round brackets the retention after 1 year of natural ageing.

Years	Flexural strength retention at 23 °C (%)		
	Isophthalic polyester/ E-glass	Orthophthalic polyester/E-glass	Vinylester/E-glass
1	80.7 (∼96)	77.7 (∼91)	82.7 (∼89)
2	78.4	75.1	80.6
5	75.4	71.7	77.9
10	73.2	69.0	75.9
20	70.9	66.4	73.9
40	68.6	63.8	71.8
50	67.9	63.0	71.2
75	66.6	61.4	70.0
100	65.7	60.4	69.1

or highly saturated environment than that produced in a dry or low RH environment [11,23]. Hence, the application of the Arrhenius model results in a more conservative assessment of the long-term performance for the considered GFRP composites.

Moreover, Table 5 shows a fast reduction for the flexural strength in the initial years of exposition while the rate reduces consistently when the time increases. This observation is consistent with the assumed Fickian behaviour for the materials, characterised by a consistent initial absorption (linear) of moisture, causing a strong reduction of the mechanical performance, and a subsequent saturation that slows down the rate of the degradation. This behaviour has been experimentally observed in the literature [14].

7. Conclusions

The study presents an experimental procedure assessing the long-term mechanical performance of pultruded GFRP composites by using the Arrhenius relationship and measuring the material's degradation at the short-term, once immersed in water solutions at different temperatures. Some relevant conclusion can be drawn.

The flexural strength retention has a strong reduction during the first months of exposure in water and progressively reaching an almost constant value at the long-term. This is caused by the higher water uptake at the beginning of the environmental exposition before reaching the saturation.

Vinylester matrix composite has the smallest reduction of mechanical performance with respect to the two polyester formulations. This is consistent with the vinylester high activation energy and low water permeation.

Setting a minimum threshold for the flexural strength retention of 65% (as in [30,31] for exterior exposure of GFRP), in an environment with an average temperature of 23 °C, the estimation based on the Arrhenius model shows that the isophthalic polyester/E-glass composite might remain above this threshold for 75 years, the orthophthalic polyester/E-glass for about 20 years, while the vinylester/E-glass for more than 100 years. These correspond to the service life of the GFRP composites for the defined performance. It should be underlined that the Arrhenius model overestimates the effect of artificial ageing comparing to one year degradation of the same materials exposed to a continental natural environment, and, as consequence, gives conservative predictions.

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