Construction and Building Materials 256 (2020) 119462

Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Degradation mechanism of glass fiber/vinylester-based composite materials under accelerated and natural aging

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HIGHLIGHTS

- Degradation mechanisms under physical and chemical aging of FRP composites.
- Aging of glass fiber and vinyl-ester composites in laboratory under natural environment.
- Prediction of composites degradation using Arrhenius model and time shift factors.
- Service knock-down factors for the design of FRP composites over a 100-year service life.

ARTICLE INFO

Article history:
Received 23 January 2020
Received in revised form 16 April 2020
Accepted 4 May 2020
Available online 14 May 2020

Keywords:
Composites
Glass fibers
Vinyl ester
Accelerated aging
Natural aging
Service knock-down factors

ABSTRACT

Glass Fiber Reinforced Polymers (GFRP) composites have become the materials of interest in replacing steel, wood, and concrete in building and construction. However, limited understanding of degradation mechanisms under physical and chemical aging of GFRP composites is still a concern preventing the widespread implementation and use of this new and emerging material in civil infrastructure. In this work, accelerated aging data for GFRP vinyl-ester composites conditioned at varying pH (2 to 13) and temperature (−22 °C to 71 °C) were collected from laboratory testing and from literature, and compared with the natural aging data under natural environment. It focuses on interlaminar shear strength (ILSS) as this is the most significantly affected property when composites are exposed to aggressive environments. High pH environment and high temperature are found to be most detrimental to GFRP composites where loss in ILSS of up to 30% was measured within the first 80 to 100 days of aging. Arrhenius model and time shift factors were used to correlate the accelerated aging data to the degradation of composites in a neutral pH environment under natural conditions. The correlation between field (natural) and accelerated aging data showed that 30% degradation of ILSS in composites occurs within the first 3 to 10 years of service, followed by a more gradual decrease. Service knock-down factors were then established in order to take into consideration the environmental and chemical effects surrounding the design of GFRP composites over a 100-year service life.

1. Introduction

There has been a significant interest in the use of glass fiber reinforced polymer (GFRP) composites in repairing, rehabilitating or replacing deteriorating civil infrastructure. According to the American Infrastructure Report Card from ASCE in 2017 [1], America's infrastructure grade is a D+ which is impeding the progression of its economy and endangering public safety. With limited resources to operate and maintain these infrastructure, GFRP composites provide a unique approach to improving the infrastructure in America. GFRP composites provide many benefits including cost-effectiveness, high durability and high strength to weight ratio for building durable and sustainable infrastructure [2]. Research and developments on GFRP composites into infrastructure have progressed well and resulted in many successful applications of polymer composites around the world. These engineering applications include corrosion resistant pipelines, storage containers, and apparatus in the chemical, oil, and gas industries as well as building, rail, and road transport including bridges [3]. As civil infrastructure needs to be designed to have a service life of at least 100 years, the long-term performance and durability of fiber composite materials need to be investigated to ensure its reliability and sustainability [4]. Being relatively new in infrastructure applica-

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https://doi.org/10.1016/j.conbuildmat.2020.119462
0950-0618/

tions, limited durability information is available in terms of mechanical properties of polymer composites in terms of expected service life of at least 100 years.

In infrastructure applications, GFRP composites are exposed to different environmental conditions which can affect physical, mechanical and chemical properties. A number of studies [5–12] have been conducted to understand the thermo-mechanical durability of GFRP composites under harsh environments for safe implementation and efficient design. In addition, durability studies were performed using accelerated aging tests by exposing GFRP composites to alkaline and acidic environments [13–15], UV radiation, moisture and elevated temperature [16–17] and hygrothermal conditions [18] to expedite the degradation of their flexural [13], compression [13], and tensile [14] strength, stiffness responses, and fiber and matrix interface [15]. These simulated laboratory environments are found harsher to GFRP composites due to exposure in much higher temperatures and in more severe pH environments than the actual field environments. As a result, accelerated aging tests resulted in higher losses in the mechanical properties when correlated with natural aging from field environment [19]. For example, Nkurunziza et al. [20] found that the degradation of GFRP bars in sodium hydroxide (NaOH) solution is more severe than in actual concrete environment due to the higher alkalinity of NaOH and the high movement of $OH-$ ions in a solution than in a fresh and hardened concrete. In fact, Benmokrane et al. [21] did not find any significant changes in the physical and chemical properties as well as microstructure of vinyl-ester-based GFRP bars sampled from a bridge barrier after 11 years of service but found degradation of their properties under accelerated aging tests. Moreover, the correlations of accelerated aging test data to natural degradation (field data) of GFRP composites is inadequate, making it difficult to predict the longevity of these materials in natural (field) environment. Similarly, there is limited naturally aging data in acidic and alkaline environments to correlate results from the accelerated aging (laboratory) data. Further studies in durability and mechanical property degradation is therefore critical to generate information on the natural degradation rates under different field conditions and to be able to incorporate these material properties over 100 year service life in design codes and implement them in both the new construction and in rehabilitation of in-service infrastructure.

This study provides an understanding on the degradation rates of glass fiber reinforced vinyl ester composites in varying pH and temperature environments. The study focused on the evaluation of interlaminar shear strength (ILSS) as a measure of durability of composites because previous researchers [22,23] have shown that the ILSS degradation rates are faster than both the tensile and flexural strength. Moreover, Bazli et al. [16] inter-laminar shear failure controls ultimate strength under bending and the degradation was greater compared to the situation where fibres fracture controls the failure. Arrhenius relationships were then adopted to describe the degradation rates of GFRP composites, and time shift factors were implemented to predict the long-term properties of these materials. With lack of naturally aged data under acidic and alkaline environments, available data from neutral pH environment was referred to in predicting the long-term properties of GFRP composites. Finally, knock-down or strength reduction factors were established based on above correlations to take into consideration the environmental and chemical effects on the properties of GFRP composites for design of structural systems.

2. Accelerated aging of composites

Accelerated aging of glass reinforced thermosets using a vinyl-ester resin were determined by evaluating the interlaminar shear strength after exposure to different solutions simulating different pH environments (acidic with pH of 3, neutral with pH of 7, and alkaline with a pH of 13) and temperatures of −22 °C, 22 °C (room temperature) and 71 °C. Glass fiber was considered in this study due to their strength to cost benefits and their current wide use in civil engineering. Similarly, vinyl ester was used as it is known for its ease of processing, low viscosity, and high level of environmental resistance.

2.1. Sample preparation

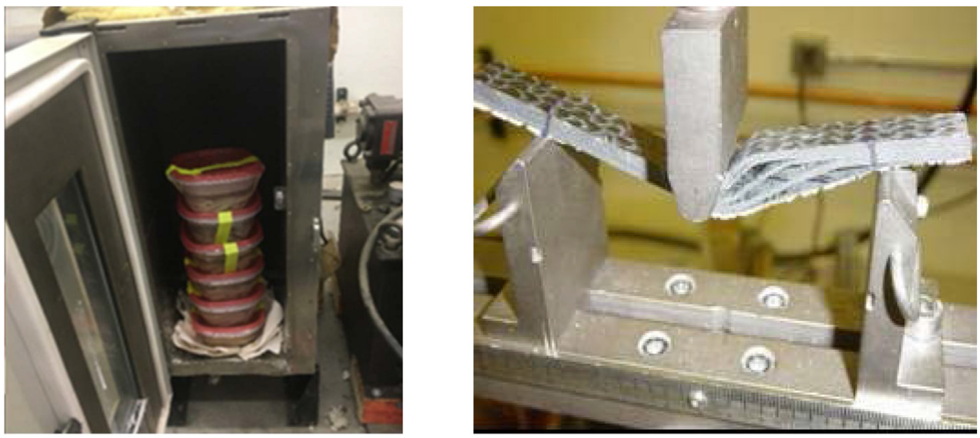
Composite plates with dimensions of 600 mm × 600 mm × 6.25 mm were manufactured from E-glass fibers (2.56 g/cm^3) and vinyl ester resin (1.2 g/cm^3) through compression molding process. The resin is made up of 99% vinyl-ester, 0.75% of methyl ethyl ketone peroxide (MEKP), and 0.25% cobalt naphthenate. These composite plates consisted of four layers of 305 gsm knitted glass fiber mats with a fiber volume fraction of 34% (54% by weight). Test specimens for ILSS with dimensions of 75 mm × 12.5 mm × 6.2 mm thick were then cut from the composite plates using a diamond blade circular saw. Three replicates for each specimen type were prepared. All dimensions and testing protocol are in accordance with the short-beam shear testing standards specified in the ASTM D2344/D2344M–13 [24]. The specimens were tested at a span of 50 mm and the load was applied at a rate of 1.3 mm/min. A total of 510 specimens were prepared, conditioned and tested in the longitudinal (264 specimens) and in the transverse (246 specimens) directions. Thicknesses of 6.2 mm and 3.1 mm were also prepared and tested to evaluate the differences in degradation rates of the GFRP composites in similar environments. Accelerated aging of the vinyl-ester (VE) resin used in the manufacture of the composites was also implemented. Two types of VE resin specimens were prepared for conditioning at 71 °C, i.e. specimens that are already pre-cut prior to aging, and specimens that are aged as a plate and cut after conditioning. All the VE specimens have a thickness of around 3.1 mm. Table 1 shows a summary of the sample types and environmental conditions, temperature and time for each condition.

2.2. Conditioning

The specimens, both in the longitudinal and transverse directions, were conditioned at different temperatures and pH levels (Fig. 1a). Prior to conditioning, the edges of the composite specimens were coated to prevent absorption of the solution into the cut surfaces. Solutions with pH of 3, 7, and 13 were used to simulate an acidic, neutral, and alkaline environment, respectively. Hydrochloric acid was mixed with tap water until a pH level of 3 was met. Acidic environment is considered as the used of GFRP composites is increasing in mining and chemical plants. Tap water was used for neutral environment. Sodium hydroxide was mixed with tap water until a pH level of 13 was met. The GFRP composites soaked in the solutions were then exposed to temperature of −22 °C, 22 °C and 71 °C to simulate freezer, room, and elevated temperature, respectively, and tested at specific days up to 150 days. The selected conditioning temperatures are well below the glass transition temperature of the glass fiber reinforced vinyl ester composites of 105 °C. Moreover, these levels of temperature were considered to create a large range of data needed for the determination of time shift factors. On the other hand, the GFRP composites with different thicknesses as well as the VE resin were conditioned only for 14 days. All specimens were air dried for 4 h prior to testing in a room temperature environment.

Table 1
Summary of samples and test conditions.

Type samples	Thickness (mm)	pH	Conditioning temperature (°C)	Duration (days)
GFRP laminates – Longitudinal direction	6.2	3, 7, 13	−22, 22, 71	1, 4, 7, 14, 21, 30, 60, 90, 150
GFRP laminates – Longitudinal direction	3.1	13	22, 71	1, 4, 7, 14
GFRP Laminates – Transverse direction	6.2	3, 7, 13	−22, 22, 71	1, 4, 7, 14, 21, 30, 60, 90, 150
Pre-cut VE resin	3.1	7	22, 71	1, 4, 7, 14
VE resin cut after conditioning	3.1	7	71	1, 4, 7, 14



(a) Conditioning in the oven

(b) Testing of composites under short beam shear

Fig. 1. Conditioning and testing of composites.

2.3. Accelerated aging for 150 days

The ILSS of unconditioned composites in the longitudinal and transverse directions are 34.3 MPa and 29.5 MPa, respectively with a coefficient of variation of 3.1% and 3.5%, respectively. After conditioning for up to 150 days, the ILSS of these composites were evaluated using the short-beam shear test method (Fig. 1b) described in ASTM D2344/D2344M–13 [24]. Table 2 summarizes the average % retention in the ILSS of composites after conditioning to different levels of temperature and pH environment. Generally, the ILSS decreased as the exposure temperature and duration increased under all exposure conditions. This can be attributed to the increase in moisture absorption of composites with exposure duration. Vinyl ester resin is known to contain large number of hydroxyl groups, which promote the absorption of moisture [25]. Once moisture is absorbed, the matrix will expand which leads to degradation of the fiber–matrix interface. This explains the degradation of the ILSS as this property is controlled by the interfacial adhesive strength at the fiber–matrix interface. Nkurunziza et al. [20] indicated that any deterioration of this interface reduces the transfer of the loads between fibers, and thus weakens the ILSS of composite materials. Bazli et al. [13] and Guo et al. [15] further explained that the absorbed moisture would reduce the glass transition temperature (T_g) of the polymers, and change the phase of material from glassy to rubbery, thereby reducing the mechanical properties of GFRP composites.

The reduction of ILSS in the neutral environment ($pH \approx 7$) is largely due to absorption of moisture and thermal variations. As the matrix swells due to the absorbed moisture, residual stresses and strains are induced inside the composites. Exposure to temperature induced thermal stresses between fibers and matrixes due to differential thermal expansion between vinylester resin and glass fibers. This differential thermal expansion of the fiber and matrix may lead to the formation of micro cracks at the fiber/ma-

Table 2
ILSS retention (%) of VE-based composites up to 150 days at different temperatures.

Days	Longitudinal									Transverse								
	Neutral pH			pH = 3			pH = 13			Neutral pH			pH = 3			pH = 13		
	Temp (°C)			Temp (°C)			Temp (°C)			Temp (°C)			Temp (°C)			Temp (°C)		
	−23	22	71	−23	22	71	−23	22	71	−23	22	71	−23	22	71	−23	22	71
1	100	89	94	97	95	93	94	85	69	97	98	94	97	101	93	93	89	69
4	92	91	87	97	94	81	93	78	62	95	97	93	99	99	90	93	69	45
7	94	88	95	90	91	94	89	74	50	95	97	88	96	97	91	90	68	43
14	96	92	91	95	91	85	82	67	53	92	95	85	98	93	87	79	55	39
21	96	94	92	93	84	86	78	57	43	100	97	85	96	83	80	80	46	34
30	97	85	81	94	90	77	80	50	38	93	92	80	102	96	86	80	41	29
60	96	92	86	92	91	86	84	51	36	100	95	83	97	95	86	73	44	26
90	97	92	83	96	93	82	73	48	32	100	89	83	102	101	86	65	50	29
150	94	–	86	97	91	–	79	52	–	94	90	83	97	93	82	66	47	27

trix interface [6]. As a result, the ILSS is weakened as this property relies on the transfer of the loads through the fiber/matrix interface. Interestingly, the reduction in ILSS of GFRP composites in both the longitudinal and transverse directions is almost same, i.e. only at a maximum of 17% when exposed at 71 °C. This result shows that at neutral environment, the degradation of the composites is initiated in the matrix mostly due to temperature exposure. This can be further noted on the high ILSS retention of composites at low temperature (−23 °C). Shi et al. [12] suggested that the stiffnesses of polymer composites increase at low temperature due to matrix hardening. This reduction in deformation of the matrix resulted in improvements in interfacial shear strength of laminated composites. Similar findings were reported by Jafari et al. [17] and Bazli et al. [26] where they observed unidirectional GFRP laminates are more resistant to freeze/thaw cycles than UV radiation and moisture cycles.

The decrease in ILSS for specimens exposed to acidic environment (pH = 3) is due to the degradation of the fiber/matrix interface caused by stress corrosion along with other reasons as enunciated for neutral environment. Surendra et al. [27] indicated that glass composites are susceptible to rapid stress corrosion in acidic environments due to the loss of integrity of the fiber/matrix interface and single fiber transverse cracking. When GFRP composites are exposed to acidic environment, the acid can reach fibers through diffusion of Cl^- ions into the micro cracks and voids in the matrix which weaken the interfacial bond [28]. Once the acid is directly in contact with the surface of the fibers, cracks will begin to grow and the ILSS will decrease. Wang et al. [14] concluded that this degradation can be attributed to the fracture of the interface between resin and fiber, which was caused by physical and chemical interactions in the acidic solution.

The results showed that the alkaline environment is the most aggressive environment for GFRP composites, reducing its ILSS to only 27% at 71 °C. This could be due to dissolution of silica (SiO_2) in glass fibers when chemically react with alkaline ions (OH^-). Nkurunziza et al. [20] indicated that the SiOH by-product forms as a gel on the surface of glass fibers while the SiO by-product forms as a solution which causes weight loss and fiber diameter reduction resulting in loss of strength. This reduction in fiber diameter occurs around the surface of the glass fibers weakening its interfacial adhesion with the surrounding VE resin. Furthermore, Guo et al. [15] and Bazli et al. [29] highlighted that the damages of GFRP composites exposed to alkaline environment are caused by resin leaching, significant resin/matrix debonding and fibre damage, which can reduced their mechanical properties.

From the data in Table 2, the ILSS retention with aging duration for GFRP composites conditioned in alkaline environment (pH = 13) at 71 °C was plotted. Only the specimens exposed in alkaline environment were plotted as seen from the data in Table 2 that the neutral pH and acidic (pH = 3) environments are nowhere near the level of concern as alkaline environments. Fig. 2a and b show the relationship of the ILSS retention of composites along the longitudinal and transverse directions, respectively. These degradation curves represent the ratio of ILSS at the day of testing over the original strength (retention %) with the aging duration (days). The R^2 of the regression line is at least 0.71, indicating the adequacy of the linear line to represent the relationship between the retention and aging duration. In these graphs, y represents the ILSS retention (%) and x is the aging duration in days. As shown in the Fig. 2(a and b), alkaline environments at high temperature present an early aggressive decrease of interlaminar shear strength in the composites (70% loss within the first 30 days of accelerated aging at 71 °C and 60% loss within the first 30 days at room temperature), both in the longitudinal and transverse directions.

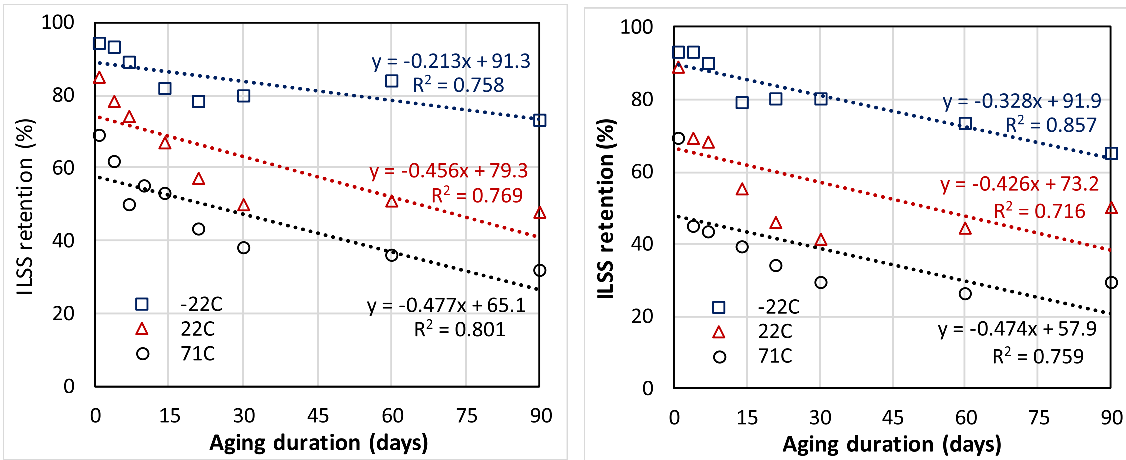
2.4. Effect of composite thickness

The effect of specimen thickness on the durability of composites was investigated by conducting a 14-day accelerated aging tests on a 6.2 mm and 3.1 mm thick GFRP composites. To eliminate variation from the manufacturing, the thinner specimens (3.1 mm) were prepared at the same time using the same manufacturing process as the 6.2 mm thick specimens. A total of 27 specimens for 3.1 mm thick GFRP composites were cut, conditioned and tested. The specimens were only exposed to an alkaline environment at room temperature and 71 °C.

Fig. 3a shows the ILSS retention of GFRP composites along the longitudinal directions exposed to alkaline environments (pH of 13) at 22 °C and 71 °C. In the graph, the specimen notations were based on the exposure temperature and laminate thickness. For example, specimen 22C_6.2 mm is a 6.2 mm thick laminates conditioned at 22 °C. From the results, similar behavior was observed for 3.1 mm and 6.2 mm thick specimens at room temperature where both specimens 22C_6.2 mm and 22C_3.1 mm retained at least 65% of its ILSS. However, higher rate of losses were observed from 3.1 mm thick specimens conditioned at 71 °C than the 6.2 mm thick specimens. Interestingly, loss in ILSS of approximately 90% was measured in the 3.1 mm thick sample (71C_3.1 mm) within the first 14 days. On the other hand, a loss of almost 50% of ILSS was observed for 6.2 mm thick specimens (71C_6.2 mm). It is to be noted that the moisture uptake of GFRP composites is a function of the shape ratio or the ratio between the specimen's surface area and volume as was found by Benmokrane et al. [6]. Their results showed that composites with a high shape ratio will absorb more moisture than the composites with a low shape ratio. As the investigated GFRP composites in this study have same overall dimensions except for the thickness, then the 3.1 mm thick specimens will have a shape ratio higher than the 6.2 mm thick specimens. Thus, it is expected that the absorbed moisture in 3.1 mm thick specimens is more than the absorption in 6.2 mm thick composites, which explains the higher reduction in the ILSS at 71 °C. Moreover, Bazli et al. [16,29] highlighted that GFRP composites with bigger sections can retain higher mechanical properties compared to smaller cross sections due to the better protection of outer layers and consequently lower damage penetration into the inner layers.

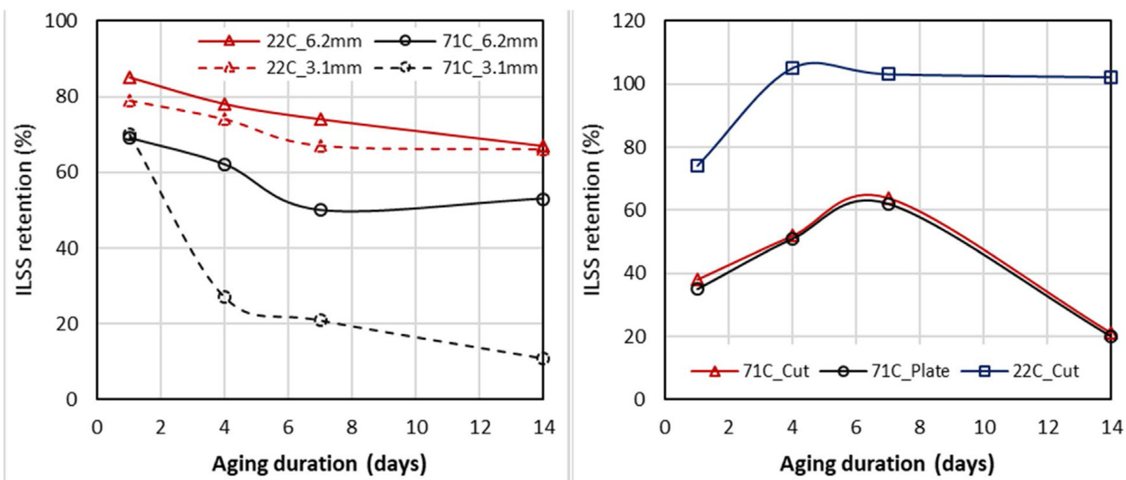
2.5. Accelerated aging of vinyl-ester resin

Fig. 3b shows the ILSS retention of VE resin exposed at neutral environment and at room temperature and at 71 °C. Similar to GFRP composites, there was a decrease in the ILSS of VE resin after 1 day of aging. However, post curing occurred in the VE resin as shown by the increase in the ILSS up to 7 days in both room temperature and at 71 °C. This was followed by the degradation of the specimens conditioned at 71 °C while there was no loss observed in the ILSS for VE specimens within the first 14 days conditioned at room temperature. A loss of approximately 80% in the ILSS was however observed from VE specimens exposed to 71 °C is recorded. There was also no significant difference on the ILSS between specimens cut prior to aging and specimens cut on corresponding testing days (specimens 71C_Cut and 71C_Plate, respectively) indicating that there is no size effect during conditioning and aging duration. This can be due to most of the moisture uptake of the VE resin occurred mostly at the top and bottom surfaces as the area of the specimen top and bottom surfaces is significantly greater than the area of the sides. The significant reduction in the ILSS after 14 days and exposed at 71 °C indicates that the VE resin degrades at both the high moisture and temperature levels. In contrast, glass fibers degrade drastically in a presence of an alkaline environment. When these effects are combined, one would expect



(a) Longitudinal (b) Transverse

Fig. 2. ILSS retention with aging duration for GFRP composites.



(a) Composites (b) Vinylester resin

Fig. 3. ILSS retention of composites with different thicknesses and Vinylester resin.

the significant decrease in the ILSS of composites as can be found in Fig. 3a.

2.6. Accelerated aging data for 150 days and longer

The accelerated aging data for more than 150 days were collected from the works of Dittenber et al. [22] and Lorenzo et al. [23]. The data were limited to ILSS of vinyl-ester GFRP composites. Some of these data are from composites aged for as long as 540 days in the laboratory. It is important to note that the GFRP composites represented by these data were only affected from pH and temperature, and no other external agents as also elaborated by Lorenzo et al. [23] and Dittenber et al. [22]. It is to be noted that the data for the GFRP composites exposed in neutral environment were taken mostly from Dittenber et al. [22] while for composites exposed in pH of 3 and 13 were referred from Lorenzo et al. [23]. These data were organized according to the exposure temperature and pH environments to align with the results obtained with the accelerated aging studies for up to 150 days in the neutral, acidic, and alkaline solutions conducted by the authors. Based on the available data, the percentage retention of the ILSS was categorized as T1 (-10 °C to 15 °C), T2 (15 °C to 40 °C), T3 (40 °C to 60 °C), and T4 (above 60 °C). A summary of these data is listed in Table 3.

As can be seen from Table 3, GFRP composites will retain most of its ILSS even after 540 days of exposure in neutral pH environment and at temperature between −10 °C and 15 °C (T1). This result indicates that the rate of degradation of GFRP composites at freezing environments causes no immediate danger to infrastructure. Similar to the findings from the accelerated tests of up to 150 days, exposure to alkaline environment (pH of 13) and elevated temperature (T2, T3, and T4) were detrimental to GFRP composites. Thus, the accelerated aging data from 150 days of experimental investigation and beyond, and the collected data for GFRP composites exposed at alkaline environment, especially at elevated temperature (71 °C and T4) were further analyzed.

Fig. 4 shows the ILSS retention of composites for exposure up to 420 days. This graph was plotted by combining the results of the accelerated aging tests in an alkaline solution (pH = 13) and a temperature of 71 °C, i.e. specimen 71C_pH = 13. For comparison, the ILSS retention of composites exposed to neutral environment and at room temperature (specimen 22C_pH = 7) and composites

Table 3
ILSS retention (%) of Vinylester-based GFRP composites at 150 days and more.

	Neutral pH				pH = 3			pH = 13		
Days	T1	T2	T3	T4	T2	T3	T4	T2	T3	T4
150	94	92	80	69	88	–	–	52	–	–
180	95	88	73	47	–	–	–	50	31	20
210	94	98	70	43	–	–	–	–	–	–
240	94	94	70	39	92	66	53	35	37	25
270	92	84	64	49	89	75	63	–	–	–
360	89	91	76	56	100	87	65	–	–	–
420	87	75	74	–	80	71	45	40	30	21
540	86	–	–	–	–	–	–	–	–	–

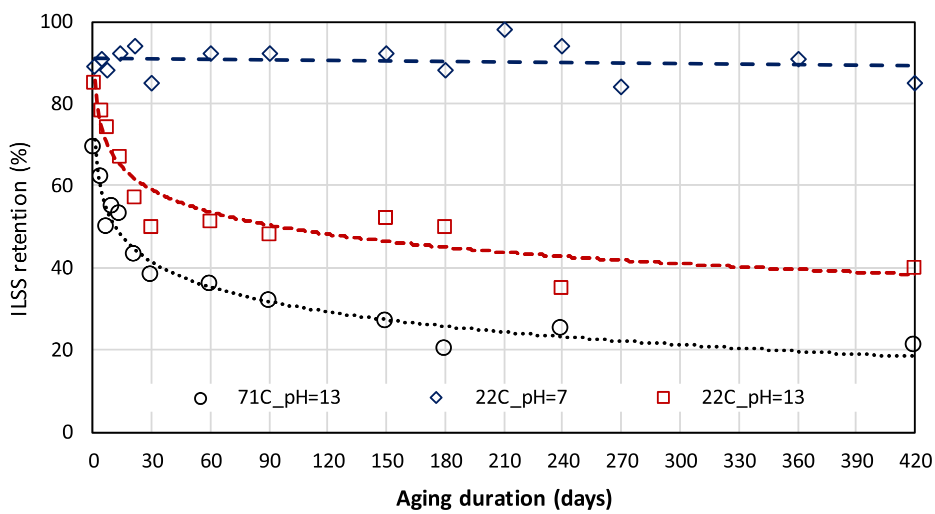


Fig. 4. ILSS retention of composites up to 420 days.

exposed to alkaline solution and at room temperature (specimen 22C_pH = 13) is included in Fig. 4. The graph shows that the ILSS degradation of composites from accelerated aging tests can be described by a significant decrease (up to 70% for specimen 71C_pH = 13) from 0 to 150 days of exposure then a gradual decrease thereafter. This result shows that these two trends should be analyzed separately to accurately describe the knock-down factors and to account for the environmental and chemical effects in the design of GFRP composites or assume that the initial design value is the reduced values at the time of 150 days. Moreover, composites exposed to neutral environment and at room temperature will retain most of its ILSS even with the increase in exposure duration, which indicates that this exposure condition can be taken as the natural aging data for GFRP composites.

2.7. Natural aging data

Natural aging data representing the data collected from the previous studies [10,11] and on-going durability studies conducted at WVU's Constructed Facilities Center (CFC) are presented in this section. Also included in these data are the results from the previous works by Dittenber et al. [22] and Lorenzo et al. [23]. These data represent investigation of the ILSS of GFRP composites made using vinylester resin aged in neutral environment (pH of 7) and at room temperature which are manufactured between 1992 and 2009. These are results from 83 specimens aged in indoors (50 specimens), partial indoor (5 specimens), and actual (outdoor) environments (28 specimens). These natural durability data are then correlated to the results of the accelerated aging tests presented in the previous section using the Arrhenius relationship and Time-Temperature superposition principle.

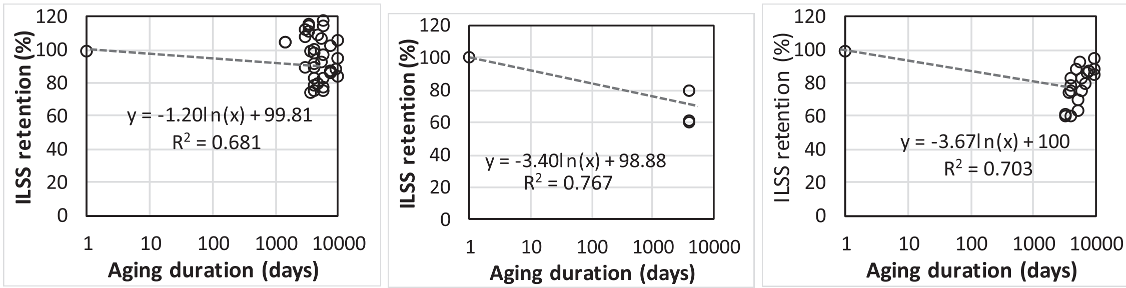
Fig. 5 shows the relationship between the ILSS percentage retention and the aging duration in days of the GFRP composites exposed in neutral environments. Trend lines are created to represent an average degradation curve for the given environmental exposures. In these graphs, y represents the ILSS retention (%) and x is the aging duration in days. The R^2 of the regression line is at least 0.68, indicating the adequacy of the linear line to represent the relationship between the ILSS retention and logarithmic of aging duration. The analyses and findings from these graphs are discussed next.

2.7.1. Indoor environment

Composites that are aged in indoor environment are those specimens that are conditioned and tested inside the laboratory (Fig. 5a). Some specimens were aged up to 26 years. This indoor environmental data was not used in correlation but was presented to provide an understanding of how GFRP composites age without any exposure to environmental conditions. It can be noted that some specimens increased in ILSS with other specimens decreased by 20%. This increase can be due to several reasons, i.e. small sample size tested, post curing reactions, etc. An overall analysis of the test data showed small reduction in ILSS indicating that composites made from glass fibers and vinylester resins are resistant to degradation in an indoor environment with a neutral pH level.

2.7.2. Partial environmental exposure

GFRP composites that are categorized under the partial environmental exposure (Fig. 5b) are those specimens that are exposed outdoors to a neutral environment (pH of 7) during the year of their manufacture and are moved indoors in 2013. From 2013, these specimens are conditioned inside the laboratory until 2018.



(a) Indoor environment (b) Partial environment (c) Full exposure

Fig. 5. Natural aging plots for composites exposed to different neutral environments.

Only a small sample size was tested (5 specimens) with exposure degradation plot shown in Fig. 5b. Due to limited data points, this result was not used in correlation. It is important to note however that some specimens in this category are exposed to actual environment for more than 10 years wherein reduction of up to 25% of ILSS from original values was measured. This result further showed that the rate of degradation in strength is higher during the first years of aging and became slower subsequently. One probable reason for this can be due to the degraded surface of the GFRP composites, acting as a protective coating to the composites which impede the penetration of moisture that affects the durability of the composites.

2.7.3. Full environmental exposure

Composites evaluated under this category are those specimens exposed outdoors of the West Virginia University Major Units Laboratory. These specimens were exposed to UV radiation, precipitation, humidity, and any other environmental exposure that would be seen in typical weathering conditions of West Virginia. Specimens were exposed outdoors from 2007 to 2018 before testing in the second half of 2018 and first half of 2019. A total of 28 specimens were aged and tested under this condition. The degradation curve in Fig. 5c was used to correlate with the results of the accelerated aging tests in the laboratory. As can be seen from the graph, up to 40% loss in ILSS was recorded, but only an average of 25% loss after 11 years of full exposure to the environment.

3. Correlation between accelerated and natural aging

The accelerated aging data obtained from the experiments (Table 2) and from previous works reported in literature (Table 3) were correlated with the naturally aging data presented in Fig. 5c. The correlations are made by applying the Arrhenius model and Time-Temperature superposition principles to create degradation plots for the accelerated aged data. Also, only the environmental effect caused by the temperature is considered and is taken as the basis of the normalization process. From these plots and correlations, the strength reduction factors are established to account for the effect of environment and aging in the design of GFRP composites.

3.1. Arrhenius model

Arrhenius model was used to analyze the degradation measured from the accelerated aging tests and to correlate with the natural aging data. This approach has been used by a number of researchers in predicting the long-term performance of composites materials and structures [30–32]. The primary assumption of this model is that the degradation mechanism of the materials' strength is not affected by temperature during exposure and the rate of degradation is accelerated with the increase in temperature. Therefore, this model is based on temperature being the dominant factor of acceleration in the aging process and can be written:

$$k = A\exp\left(\frac{-E_a}{RT}\right) \text{or} \ln k = \ln A + \frac{-E_a}{RT} \quad (1)$$

where: k = degradation rate (1/time); A = constant of the material and degradation process; E_a = activation energy; R = universal gas constant; and T = temperature in Kelvin. The logarithm of $1/k$ is the time for a material property to degrade to a certain value and with the slope E_a/R [33].

From the results of the experimental investigations (Fig. 4), the most severe degradation in GFRP composites occurred within the first 30 days with almost 70% of ILSS loss and a gradual decrease thereafter. Thus, Arrhenius equations for 0 to 150 days and for longer than 150 days were separately established using transformed logarithmic retention plots that represent the amount of time needed to reach a specific strength retention level (i.e. 90%, 80%, etc.) against the inverse of temperature (in Kelvin). The slope of the best-fit line in the Arrhenius plots is the activation energy (E_a) or the amount of energy which the reacting materials must possess in order to undergo a specified reaction. The higher the slope of this plot, the more the material is susceptible to temperature changes. From these plots, the values of the activation energy over the gas constant (R) for the different aging and pH environments are recorded for room temperature [0.0034 (1/T°K^{-1})], 40 °C [0.0031 (1/T°K^{-1})] and 71 °C [0.00295 (1/T°K^{-1})], and are summarized in Table 4. From the calculated Ea/R values, it can be noticed that the alkaline environment does the most damage in the early aging period, then it begins to slow down after 150 days. As discussed earlier, this can be due to the degraded surface of the GFRP composites acts as a protective coating which impede the penetration of moisture that affects the durability of composites. Also, the neutral and acidic environments only begin to cause degradation to composites after 150 days of exposure. Thus, these activation energy values can be used to determine the aging with time and the remaining strength of composites in different environments.

3.2. Time-dependent superposition

The Time-Temperature Superposition (TTS) principle is implemented to predict the degradation of GFRP composites in a natural environment by using accelerated aging data and correlating with the naturally aged data. By using time shift factors (TSF), one can shift the accelerated aging retention plots to a longer-term degradation plot. For example, with approximately one year of accelerated aging data in neutral environment, the data to roughly 30 years of natural age can be extrapolated using TSF. This principle is used to correlate data at room temperature setting to the

Table 4
Activation energy values and time shift factor for vinylester-based glass fiber composites.

Aging time	Direction	pH	$-E_a/R$	TSF_T 0.0034 (1/T°K^{-1})	0.0031 (1/T°K^{-1})	0.00295 (1/T°K^{-1})
0 to 150 days	Longitudinal	Neutral	5527	1.0	N/A	7.5
		Acidic	4799	1.0	N/A	8.0
		Alkaline	3180	1.0	N/A	4.5
	Transverse	Neutral	5287	1.0	N/A	10.7
		Acidic	4112	1.0	N/A	5.3
		Alkaline	2910	1.0	N/A	3.7
Longer than 150 days	Longitudinal and transverse	Neutral	1682	1.0	2.3	28.6
		Acidic	2350	1.0	2.3	11.4
		Alkaline	4236	1.0	1.8	9.7

data in different temperatures aged at higher than room temperature. This implies that the behavior of composites at one temperature can be related to room temperature by shifting the time to achieve the same level of degradation. It is important to understand that this principle applies only to materials that conditioned at a temperature below their glass transition temperature.

3.3. Time shift factor (TSF)

Time shift factors (TSF) is used to shift the accelerated aging plot results. These factors allow composites to be normalized under different temperature environments based to be able to create degradation plots for specific pH environments. After the time shift factors are obtained, regression curves were created and then correlated with naturally aging data (only for neutral pH environment) to predict strength retentions in the field during the service life of the structure.

The time shift factor due to exposure at different temperature conditions (TSF_T) can be established from the generated Arrhenius relationships. In this process, the data is normalized based on a reference temperature, which is set at room temperature (22 °C). This means that the TSF_T for room temperature is 1.0 and as the temperature increases, the time shift factors increase to account for the faster degradation rates at higher temperature. Temperatures below room temperature are not of a concern knowing that very low degradation rates occur are measured in composites exposed in this temperature, thus the TSF_T for −22 °C was not calculated due to such low degradation values. Following Eq. (1), the TSF_T is obtained as the ratio of the amount of days it takes for a GFRP composites to reach the percentage strength retention based on the reference temperature (T_1) to the amount of days it takes for the composites to reach the same retention level in another temperature level (T_2) of approximately 20 °C below the T_g. The time for a certain reaction to take place is proportional to the inverse of the reaction rate k and the ratio of $t_{1,}$ time required for a certain decrease of a property at temperature T_1 and t_2, time required for the same decrease at temperature T_2 [33]:

$$TSF_T = \frac{t_1}{t_2} = \frac{Ae^{\frac{E_a}{T_2 R}}}{Ae^{\frac{E_a}{T_1 R}}} = e^{\frac{E_a}{R}\left(\frac{1}{T_2}-\frac{1}{T_1}\right)} \tag{2}$$

In the calculation of the TSF_T, the time of degradation of the accelerated aging specimens for each retention level (90%, 80%, 70%, etc.) is extracted from the retention plots in Fig. 1. Using Eq. (2), TSF_T for every temperature tested is calculated and summarized in Table 4. It is important to note that only correlation with neutral environment specimens was implemented as there is no available data for naturally aged test specimens under alkaline or acidic environmental conditions. Using the data, prediction on retention values can be made for neutral environment of GFRP composites for a service life of 100 years.

3.4. TSF_T for different temperature ranges

The TSF_T for different levels of temperature can be extrapolated from the exponential relationship of the TSF_T established from the accelerated aging and inverse of temperature T. Note that only the TSF_T for temperature between room temperature (22 °C) and 71 °C are determined in this paper, which are well below glass transition temperature of composites. Fig. 6 shows the exponential relationship between the TSF (in y-axis) and the $1/T$ (°K^{-1}) (in x-axis) including the R^2 of the regression line. As can be noticed in the figure, the R^2 is at least 0.84 showing the high confidence level on the relationship between TSF_T and temperature. The R^2 of 1.0 for 0 to 150 days exposure is due to 2 data points are only available under natural aging conditions.

3.5. Accelerated data shift for long term degradation

The calculated TSF_T values in Table 4 were used to calculate the degradation of composites under a specific service condition from the data acquired through accelerated aging. This is done by shifting the data from accelerated aging tests in the laboratory and multiplying the accelerated aging time, $t_{accelerated}$ with the TSF_T value for given temperature and environment shown in Eq. (3).

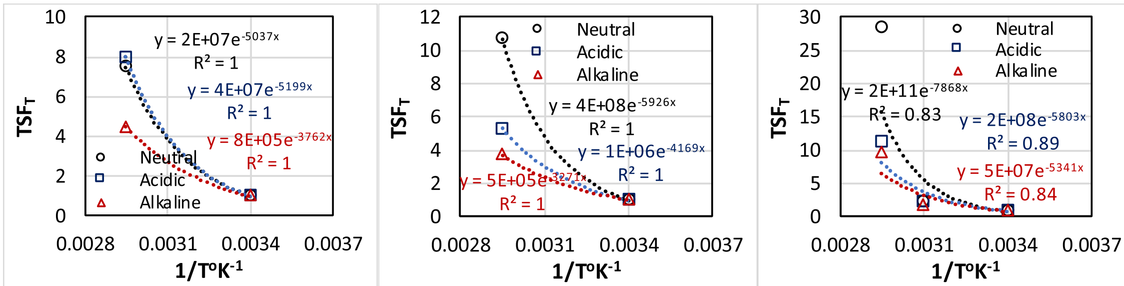
$$t_{shift} = t_{accelerated} * TSF_T \tag{3}$$

where t_{shift} = shifted time in days used to create acceleratedly shifted degradation plots. By multiplying the accelerated time to the corresponding time shift factor for the specific temperature and environment, a shifted time can be created for that retention level in a new degradation plot. Figs. 7–9 show the ILSS retention against the shifted times in days based on the accelerated aging data reported in Tables 2 and 3. These degradation curves are created as a logarithmic function to best represent the data. In the equation of the trend lines, y = retention percentage and x = shifted time in days calculated using TSF_T in Table 4. Knowing that alkaline environment is the most detrimental pH environment, R^2 values for those environments are relatively high for all aging environments (R^2 of at least 0.81), which provides high confidence level in the results.

From the accelerated and shifted degradation plots, it can be noticed that the time for accelerated aging at 0 to 150 days is shifted for up to almost 3 years (960 days) in some cases while the shifted plots for accelerated aging of 150 days and longer can be correlated up to 27 years (10,200 days), based on the data generated by the authors. Thus, this approach can also used to correlate the accelerated aging data with the aging of composites in the field environment.

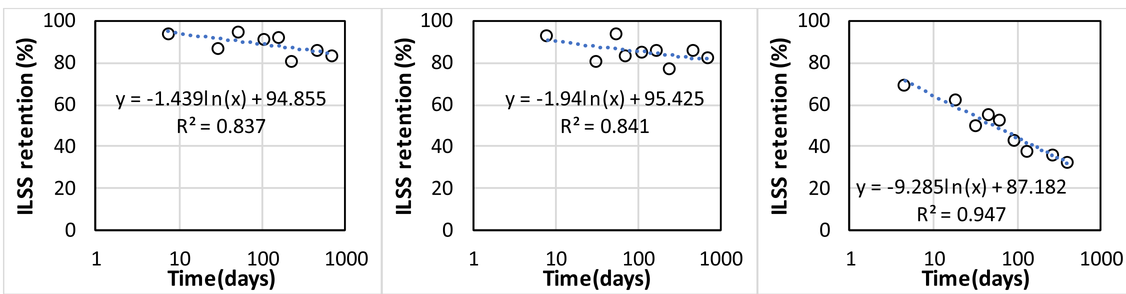
4. Correlation between field and accelerated aging data

Data on natural aging of GFRP composites is very limited and difficult to obtain from literature because these materials are still



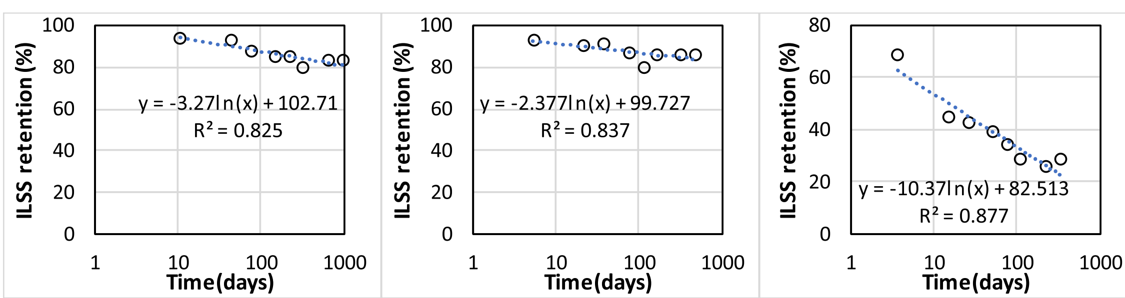
(a) 0 to 150 days (longitudinal) (b) 0 to 150 days (transverse) (c) longer than 150 days

Fig. 6. Relationship of TSF and temperature at different neutral environments.



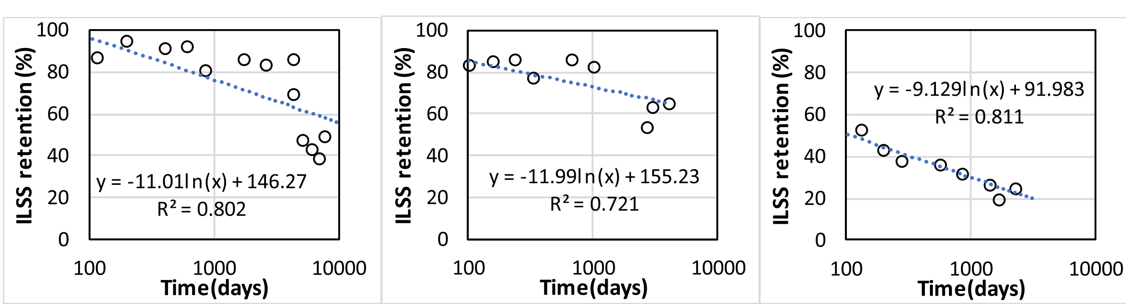
(a) Neutral (b) Acidic (c) Alkaline

Fig. 7. Accelerated shifted degradation plots in the longitudinal direction (0 to 150 days).



(a) Neutral (b) Acidic (c) Alkaline

Fig. 8. Accelerated shifted degradation plots in the transverse direction (0 to 150 days).



(a) Neutral (b) Acidic (c) Alkaline

Fig. 9. Accelerated shifted degradation plots for longer than 150 days.

relatively new in infrastructure applications. The only available field data are those reported by Dittenber et al. [22] and Lorenzo et al. [23], which were presented in Fig. 5c. These data were used to predict the aging behavior of GFRP composites in the field by correlating with the shifted and accelerated aging degradation data for specimens exposed in the neutral environment. The equation of the trendlines of these degradation curves are summarised in Table 5. Data was analyzed separately for the first 150 days (Figs. 7 and 8) and 150 days or longer (Fig. 9) of exposure as it was found earlier that the rate of degradation from the accelerated aging tests are totally different in these aging durations. In these equations, y is the ILSS percentage retention while x is the natural aging time in days for field degradation and the accelerated shifted time for accelerated aging. The equation of the acidic and alkaline degradation curves determined from accelerated aging tests are also presented in the table, but they were not used for correlation. These accelerated aging trends are however useful and can be used in predicting the behavior of GFRP composites made from glass fiber and vinylester resin in acidic and alkaline environments when data on the field degradation exposed in these conditions become available.

Time-Temperature Superposition principle was used to correlate the field degradation of GFRP composites to that of the accelerated aging results. The equations presented in Table 6 can be re-written in general form as Equations (4) and (5) with variables a, b, c, and d. In this correlation, it was assumed that the ILSS of the composites is at 100% at the beginning of the aging (t_0), i.e. $\%ILSS_0$ = b = 100%. The other variables a, c and d can be found from the slope of the degradation curves summarized in Table 5.

$$\%ILSS_{natural} = a\ln(t_{natural}) + b \tag{4}$$

$$\%ILSS_{accelerated} = c\ln(t_{shift}) + d \tag{5}$$

where $\%ILSS_{natural}$ = percentage retention in ILSS from natural aging, $t_{natural}$ = natural aging time in days, and $\%ILSS_{accelerated}$ = percentage retention in ILSS from accelerated aging.

From Eqs. (4) and (5), the time shift factor to correlate the field and accelerated aging data (TSF_{Field}) is calculated by taking the ratio of the time taken by a composites to degrade in the field over the time it would degrade under accelerated aging tests to the same retention level as shown in Eq. (6). The percentage retention being calculated for is used as the "%" variable.

$$TSF_{Field} = \frac{t_{natural}}{t_{accelerated}} = \frac{\exp\left(\frac{\%-b}{a}\right)}{\exp\left(\frac{\%-d}{c}\right)} = \exp\left(\frac{ad - cb + \%(c-a)}{ac}\right) \tag{6}$$

where: $t_{natural}$ = time in days to take natural aged data to reach a specific retention level, and $t_{accelerated}$ = time it takes for accelerated data to reach a specific retention level.

The TSF_{Field} for up to 3 years was calculated by correlating the 0 to 150 days accelerated aging data with the field degradation while the degradation for more than 3 years was calculated using the degradation rate for 150 days and longer. Based on the field and accelerated aging degradation data in Table 6 and using Eq. (6), the average TSF_{Field} for up to 3 years is at 3.2 while it is 1.4 for longer than 3 years. With the TSF_{Field} being only 1.4 for neutral environment for longer than 150 days, it shows that most of the degradation already occurred within the first three years of aging. This is consistent with how GFRP composites degrade with respect to interlaminar shear strength retention with time as shown in Fig. 4. It is to be noted that these correlations are only for ILSS of glass fiber reinforced vinyl-ester composites in a neutral pH environment. The percentage retention of the ILSS in the field ($\%ILSS_{Field}$) can then be calculated by incorporating the TSF_{Field} as shown in Eq. (7). Similarly, the time to reach this level of degradation in the field (t_{Field}) can be calculated by multiplying the accelerated time with TSF_{Field} as shown in Eq. (8).

Table 6
Strength reduction factors for glass fiber reinforced vinyl-ester composites.

Time (years)	Neutral	Alkaline	Acidic
3	0.70	0.40	0.71
5	0.63	0.37	0.63
10	0.55	0.32	0.59
25	0.45	0.24	0.54
50	0.38	0.18	0.51
75	0.33	0.15	0.49
100	0.30	0.12	0.48

$$\%ILSS_{Field} = a_{accelerated} * \ln(t_{Field}/t_{natural}) + 100 \tag{7}$$

$$t_{Field} = TSF_{Field} * t_{accelerated} \tag{8}$$

Using the relationship in Eq. (7), the degradation of composites in the first 3 years and from 3 to 100 years are generated and presented in Fig. 10. As can be seen in Fig. 10a, the ILSS of composites will be reduced by at least 30% in the first 3 years of exposure in the neutral environment (pH = 7) and will retain only 30% after 100 years of service. It is to be noted that however the prediction of the degradation of composites beyond 3 years is only from the natural data of eleven years. A more reliable prediction equation can be derived with more data available for longer degradation of these composites in natural environment.

5. Strength reduction factors for environmental conditions

The degradation of GFRP composites exposed to different environmental conditions is a huge concern for asset owners and design engineers. As a result, knock-down factors or environmental reduction coefficient is applied in the design to account for long-term performance of GFRP composites and their exposure to different environmental conditions to ensure safety and reliability of structures during their design service life. From the understanding of the degradation of GFRP composites established from this study and the data of natural degradation of up to 11 years of field exposure, strength retention factors for glass fiber reinforced vinyl-ester composites up to 100 years of service are calculated and presented in Table 6. It is to be noted that the retention percentages in alkaline (pH = 13) and acidic (pH = 3) environments are based solely on the shifted accelerated aging data using the Time-Temperature Superposition principle as there is no natural aging data available to correlate with.

The strength reduction factor of the ILSS for GFRP composites exposed in alkaline solution is lower than the strength reduction factors of 0.65 and 0.30 for tensile and compression, respectively as suggested by Bazli et al. [29]. This comparison confirms that the ILSS is a good measure of durability of GFRP composites in alkaline environment. The lower reduction in acidic than in neutral

Table 5
Equations of the field and accelerated aging degradation curves.

Environment	Field degradation	Accelerated aging		
		Long. (0–150 days)	Trans. (0–150 days)	Longer than 150 days
	Fig. 5c	Fig. 7	Fig. 8	Fig. 9
Neutral	y = −3.67ln(x) + 100	y = −1.43ln(x) + 94.8	y = −3.27ln(x) + 102.7	y = −11.01ln(x) + 146.2
Acidic	Not available	y = −1.94ln(x) + 95.4	y = −2.377ln(x) + 99.7	y = −11.99ln(x) + 155.2
Alkaline	Not available	y = −9.28ln(x) + 87.1	y = −10.37ln(x) + 82.5	y = −9.12ln(x) + 91.9

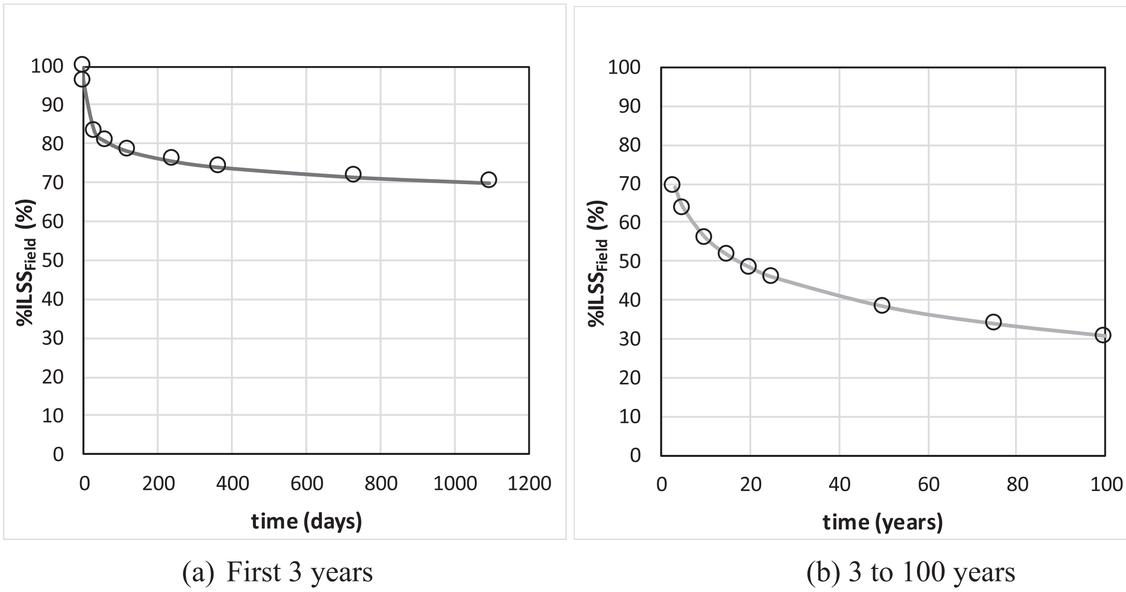


Fig. 10. Predicted field degradation of composites subject to neutral environment.

environment can be due the GFRP composites in the field is affected by other environmental effects such as sustained stress, UV radiation, temperature cycles, humidity cycles, etc. Nevertheless, these strength reduction values can be used as knock-down factors in the design by multiplying with the initial GFRP material resistance in order to take into consideration the environmental and chemical effects surrounding the GFRP over its design service life, which is being followed in the Load and Resistance Factor Design (LRFD). These reduction factors do not account for creep or fatigue related responses of GFRP composites.

6. Conclusions

In this study, accelerated aging for up to 150 days and mechanical property evaluation was conducted under controlled laboratory environments by varying temperatures and pH solutions to understand the degradation mechanism of glass fiber reinforced vinyl-ester composites. Accelerated aged data for 150 days and longer was taken from previous works at West Virginia University. It focused on interlaminar shear strength (ILSS) since it is the most sensitive to aging in terms of mechanical properties. Arrhenius relationship and Time-Temperature Superposition principle were then implemented to predict the long-term properties of composites. From the results of these investigations, the following conclusions are drawn:

- The interlaminar shear strength of GFRP composites under accelerated aging tests exhibited two distinctly different rates of degradation, i.e. significant degradation within the first 150 days and at a slow rate of degradation for 150 days or longer.
- Accelerated aging in alkaline solution caused significant degradation on GFRP composites. Up to 70% and 60% degradation in ILSS is observed within the first 30 days of aging at elevated temperature and at room temperature, respectively.
- Moisture absorption of vinylester resin and the dissolution of silica in the glass fibers caused the weakening of the fiber/matrix interface and the significant ILSS reduction of the glass fiber reinforced vinyl-ester composites in the alkaline solution.
- GFRP composites exposed to neutral and acidic environments have lower ILSS reduction than those exposed to alkaline environment, and are not sensitive at freezing and room temperature. Composites exposed to these environments show degradation only after 150 days of accelerated aging.
- Using the Arrhenius model and time-temperature superposition principle, the accelerated aging tests of composites at 150 days is found equivalent to almost 3 years of exposure while the accelerated aging for longer than 150 days can be correlated up to 27 years of natural degradation.
- In neutral environment (pH = 7), the ILSS of composites will be reduced by at least 30% in the first 3 years of service in the field and will retain only 30% after 100 years of service.
- The established knock-down factors or environmental reduction coefficient of up to 100 years can be used to account for in the design the long-term performance of GFRP composites in actual field conditions.

The results from this study provided a better understanding on the degradation mechanisms of composites in neutral, acidic and alkaline environments. Additional studies are however recommended on the degradation in the field environment to provide a more complete overview of the durability of composites and their application into engineering applications. A more reliable prediction equation can be also derived with more and longer degradation data of composites in natural environment. This will give higher confidence on utilization of GFRP composites in civil engineering and ensuring they will retain their mechanical properties during their design service lives. Similarly, more experimental data is needed on the effect of other environmental factors including UV radiation, thermal fatigue, freeze-thaw cycles, sustained stress, etc. to create a broader understanding on the durability of GFRP composites and to establish a more accurate knock-down factor for engineering design.

CRediT authorship contribution statement

Gangarao Hota: Conceptualization, Supervision, Validation, Visualization, Writing - original draft, Writing - review & editing. **William Barker:** Data curation, Formal analysis, Writing - review

& editing. **Allan Manalo:** Data curation, Formal analysis, writing - original draft, writing - review and editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

The authors acknowledge the support of NSF-IUCRC sponsored Centre for Integration of Civil Infrastructure at West Virginia University in conducting the study. The third author also acknowledges the scholarship granted by the Australian Government Endeavour Executive Leadership Award to undertake his professional development at West Virginia University, Morgantown, USA.

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