

1 **Laboratory Assessment and Durability Performance of Vinyl-Ester, Polyester, and Epoxy**
2 **Glass-FRP Bars for Concrete Structures**

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25 **Abstract**

26 In the last decade, noncorrosive glass fiber-reinforced-polymer (GFRP) bars have become more
27 widely accepted as cost-effective alternatives to steel bars in many applications for concrete
28 structures (bridges, parking garages, and water tanks). Also, these reinforcing bars are valuable
29 for temporary concrete structures such as soft-eyes in tunneling works. The cost of the GFRP
30 bars can be optimized considering the type of resin according the application. Yet limited
31 research seems to have investigated the durability of GFRP bars manufactured with different
32 types of resin. In this study, the physical and mechanical properties of GFRP bars made with
33 vinyl-ester, isophthalic polyester, or epoxy resins were evaluated first. The long-term
34 performance of these bars under alkaline exposure simulating a concrete environment was then
35 assessed in accordance with ASTM D7705. The alkaline exposure consisted in immersing the
36 bars in an alkaline solution for 1000, 3000 and 5,000 h at elevated temperature (60°C) to
37 accelerate the effects. Subsequently, the bar properties were assessed and compared with the
38 values obtained on unconditioned reference specimens. The test results reveal that the vinyl-ester
39 and epoxy GFRP bars had the best physical and mechanical properties and lowest degradation
40 rate after conditioning in alkaline solution, while the polyester GFRP bars evidenced the lowest
41 physical and mechanical properties and exhibited significant degradation of physical and
42 mechanical properties after conditioning.

43

44 **Keywords:** Glass fiber; vinyl ester, polyester, epoxy; fiber-reinforced polymer (FRP); glass FRP
45 (GFRP) rebars; physical and mechanical properties; durability performance; alkaline; accelerated
46 aging; microstructural, concrete structures.

47 **Introduction**

48 Fiber-reinforced-polymer (FRP) bars have been well accepted as internal and external
49 reinforcement for concrete structures (ACI 440.1R [ACI 2015]; Benmokrane et al. 2016a; Ali et
50 al. 2016a; Mohamed et al. 2016). This reinforcing material offers better resistance to
51 environmental agents as well as high stiffness-to-weight and strength-to-weight ratios when
52 compared with conventional construction materials such as steel. Extensive research and
53 development efforts have demonstrated that FRP bars are effective reinforcement in concrete
54 members subject to bending (Maranan et al. 2015), shear (Ali et al. 2013 and 2016b),
55 compression (Maranan et al. 2016), and impact (Goldston et al. 2016). Material specifications
56 and design guidelines (ACI 440.6M [ACI 2008]; CAN/CSA S807 [CSA 2010]) have also been
57 developed to encourage the construction industry to use FRP bars. This has resulted in many
58 demonstration projects and field applications, such as bridges (Benmokrane et al. 2004), parking
59 garages (Benmokrane et al. 2012), water-treatment plants (Mohamed and Benmokrane 2014),
60 bridge barriers (El-Salakawy et al. 2005), concrete pavement (Benmokrane et al. 2008), and
61 jetties (Manalo et al. 2014).

62 Different types of fibers are used in manufacturing FRP bars such as carbon, glass,
63 aramid, and basalt. Many studies have been carried out on the performance and use of FRP bars
64 made with these different fibers, providing good insight into their physical and mechanical
65 properties as well as their durability characteristics (Kocaoz et al. 2005; Banibayat and Patnaik
66 2014; Ali et al. 2015; Benmokrane et al. 2016a, b; Li et al. 2015; Abbasi and Hogg 2005;

67 Alsayed et al. 2012; Al-Salloum et al. 2013; Hassan et al. 2016; Tanks et al. 2016). Glass is the
68 most commonly used fiber type in manufacturing FRP bars due to their relatively low
69 comparative cost (ACI 2015). Similarly, Castro et al. (1998) highlighted the importance of the
70 resin system used in manufacturing FRP bars to achieve the desired mechanical properties and
71 durability characteristics. The resin system is important as it acts as a matrix bonding the fibers
72 together and spreading the load applied to the composite between each of the individual fibers.
73 The resin system also protects the fibers from abrasion and impact damage as well as from
74 severe environmental conditions—such as water, salts, and alkalis—which affect the durability
75 of FRP products (SP System 1998). A deterioration of this interface reduces the transfer of the
76 loads between fibers and thus weakens the composite materials (Almusallam et al. 2013). The
77 interface between the fiber and matrix is a nonhomogeneous region about 1 μm thick. This layer
78 is weakly bonded and most vulnerable to deterioration. The three dominant deterioration
79 mechanisms include matrix osmotic cracking, interfacial debonding, and delamination (Chen et
80 al., 2007). Moisture diffusion into FRP composites could be influenced by the material's
81 anisotropic and heterogeneous character. Along with diffusion into the matrix, wicking through
82 the fiber/matrix interface in the fiber direction could be a predominant mechanism of moisture
83 ingress (Apicella et al., 1982). Nonvisible dissociation between fibers and matrix could lead to
84 rapid losses of interfacial shear strength (Ferrier et al. 2016; Ashbee and Wyatt, 1969).
85 Unfortunately, limited research attention has been paid to the effect of the resin-system type on
86 the physical and mechanical properties as well as the durability characteristics of GFRP bars.

87 Most of the glass-fiber-reinforced polymer (GFRP) bars available are manufactured with
88 E or ECR glass fibers that are normally wetted with a thermosetting resin such as epoxy or
89 vinyl ester. Numerous studies have investigated FRP bars made with vinyl-ester resin to

90 determine the effect of environmental conditions (water, salts, alkalis) on their physical and
91 mechanical properties (Mouritz et al. 2004; Wang 2005; Zou et al. 2008; Robert et al. 2009;
92 Robert and Benmokrane 2013; Benmokrane et al. 2014, 2015, 2016b, 2016c). Similarly, Soles et
93 al. (1998); Amaro et al. 2013; Ali et al. 2015; and Benmokrane et al. (2016a) are some of the
94 numerous researchers who have investigated the durability performance of FRP bars made with
95 epoxy resins. GFRP bars made with these resin systems are the most commonly used as
96 reinforcement for concrete structures given their high performance and very good durability
97 characteristics. Studies into the behavior of fiber-reinforced isophthalic polyester-resin
98 composites have primarily addressed industrial and nonstructural products such as natural-fiber
99 composites (Manalo et al. 2015). GFRP bars manufactured with isophthalic polyester resin are
100 normally used for temporary structures such as soft-eyes in underground excavations and
101 tunneling works (Schurch and Jost 2006). In these proprietary applications, GFRP-bar durability
102 is not a concern. The key advantage of GFRP bars is the low cost of polyester resin and the fact
103 that GFRP bars can be cut without damaging the drilling equipment's cutter heads. Comparisons
104 performed by some researchers [Ashbee et al, 1967; Ashbee and Wyatt, 1969; Abeysinghe et al.,
105 1982] have indicated that the matrix formed by vinyl ester, which contains many fewer ester
106 units compared to polyester, experiences very little deterioration caused by hydroxyl ions
107 compared to a polyester matrix. As a result, CSA S807 (2010) classifies isophthalic polyester-
108 based GFRP bars as having moderate durability (D2), while classifying epoxy- and vinyl-ester-
109 based GFRP bars as having high durability (D1). Obviously, these classifications were
110 established based on the results obtained by different researchers on GFRP bars manufactured
111 with a specific resin system, i.e., either vinyl esters or epoxies (with very few studies on
112 isophthalic polyesters). Consequently, no sound generalizations can be made. Clearly, a single

113 approach is needed to confirm that GFRP bars manufactured with different types of resin will
114 have different physical and mechanical properties as well as different durability characteristics,
115 therefore providing for direct comparison of these important properties.

116 This paper presents an experimental investigation aimed at assessing and comparing the
117 physical and mechanical properties of three different types of GFRP bars made with vinyl-ester,
118 isophthalic polyester, or epoxy resins. The tests findings on the long-term durability of these bars
119 conditioned in an alkaline solution simulating a moist concrete environment at high temperature
120 are also presented. The aim is to further understanding of the various resin options available for
121 GFRP bars and their associated behavioral characteristics, yielding useful information about
122 materials specifications and design standards.

123 **Experimental-Program Outline**

124 This experimental investigation was conducted on three different GFRP bars that were 12 mm in
125 diameter: glass/polyester, glass/vinyl-ester, and glass/epoxy as shown in Figure 1. The
126 experimental work was divided into three phases. Phase I included the determination of physical
127 properties, which were compared to those obtained after conditioning. Phase II included
128 mechanical characterization, including transverse shear strength, flexural strength, flexural
129 modulus of elasticity (stiffness), and apparent horizontal shear strength (interlaminar-shear
130 strength). The test results also served as references for residual strength after conditioning. Phase
131 III included a preliminary durability assessment and long-term performance assessment of the
132 GFRP bars immersed in alkaline solutions simulating concrete pore solution at 60°C for different
133 times up to 5,000 h. The changes in the physical and mechanical characteristics were assessed by
134 comparing the characteristics of the conditioned bars to those of the reference bars from Phases I
135 and II. The effects of conditioning on the glass transition temperature (T_g) and chemical

136 composition of the materials were also assessed with differential scanning calorimetry (DSC)
137 and Fourier transform infrared spectroscopy (FTIR), respectively. In addition, bar microstructure
138 was investigated using scanning electron microscopy (SEM) for both conditioned and
139 unconditioned bars to assess changes and/or degradation.

140 **Material Properties and Test Specimens**

141 The glass/polyester, glass/vinyl-ester, and glass/epoxy FRP bars were manufactured with
142 continuous glass fibers impregnated in polyester, vinyl-ester, or epoxy resins using the pultrusion
143 process. Table 1 lists the typical properties of these thermosetting-resin systems as reported by
144 Bank (2006). The three types of GFRP bars (Figure1) were manufactured by Firep International
145 AG (Switzerland) using the same fabrication process and equipment, same glass fiber, and same
146 additives to ensure bar consistency. The GFRP bars had a nominal diameter of 12 mm and were
147 deformed with helical wrapping (Fig. 1). The nominal cross-sectional area of the three GFRP
148 bars was 113 mm^2 , as reported by the manufacturer. The mechanical properties reported herein
149 were calculated using the nominal cross-sectional area.

150 For this study, the GFRP bars were provided in 170 and 240 mm lengths so that the transverse
151 shear-strength test and flexural test could be performed according to ASTM D7617 (ASTM
152 2011), and ASTM D4476 (ASTM 2009), respectively. In addition, some specimens were cut into
153 83 mm lengths so that the short-beam shear test could be performed according to ASTM D4475
154 (ASTM 2008) on the three types of GFRP bars.

155 **Testing, Results, and Discussion**

156 **Phase I: Physical Characterization**

157 Physical properties for the reference (unconditioned) GFRP bars were determined according to
158 ACI (2008) and CSA (2010) requirements, including: (1) fiber content, (2) moisture absorption,
159 (3) cure ratio, and (4) glass transition temperature.

160 ***Fiber content***

161 Glass fiber content was determined by thermogravimetry according to ASTM E1131. A very
162 small piece of material (a few tenths milligrams) was cut from the center of the bar, placed in
163 platinum crucible and then heated up to 550°C under inert atmosphere. The weight loss (W_L) has
164 been recorded at a temperature equal to 550°C. Since the material only contains carbon fibers
165 and resin, fiber content by weight was then calculated according to the following equation:

$$166 \quad \text{Fiber content by weight} = 100 \cdot (W_T - W_L) / W_T \quad (1)$$

167 where, W_T is the total weight before burn off.

168 ***Water-immersion test***

169 The moisture uptake at saturation of the GFRP bars was determined according to ASTM D570,
170 except that the immersion was in tap water instead of distilled water. Three 50 mm long
171 specimens were cut, dried, and weighed prior to immersion in water at 50°C for three weeks. The
172 samples were removed from the water after three weeks, surface dried, and weighed.

173 The water content at saturation in weight percent (W_s) was calculated using Equation 2

$$174 \quad W_s = 100 \cdot (P_s - P_d) / P_d \quad (2)$$

175 where P_s and P_d are the sample weights in the saturated and dry states, respectively.

176 ***Cure ratio***

177 Cure ratio was determined according to ASTM D5028 and CSA (2010). The enthalpy of
178 polymerization of the sample was measured by DSC and compared to the enthalpy of
179 polymerization of pure resin, taking into account the weight percentage of resin in the matrix.

180 Thirty to fifty milligrams of sample were accurately weighed and placed in an aluminum
181 crucible. The samples were then heated from room temperature to 200°C at a heating rate of
182 20°C/min, and the area of the peak of polymerization was calculated. The measurement was
183 carried out on 3 specimens.

184 *Transverse coefficient of thermal expansion*

185 The transverse coefficient of thermal expansion was calculated according to ASTM E1131-08
186 (2014). Nine specimens of each type of GFRP bars were tested. The measurements were
187 conducted between -30°C and 60°C at a heating rate of 3°C. A TA Q400 thermomechanical
188 analyzer was used. Cryogenic equipment (liquid nitrogen) was used to reach subzero
189 temperatures. The results show that the coefficients of thermal expansion for the different bar
190 diameters fell between $20.5 \times 10^{-6}/^{\circ}\text{C}$ and $22.0 \times 10^{-6}/^{\circ}\text{C}$, which is only half of the limit of
191 $40.0 \times 10^{-6}/^{\circ}\text{C}$ specified in CSA 807 (2010).

192 Table 2 lists the physical properties of the unconditioned GFRP bars, where the glass transition
193 temperature (T_g) was determined with differential scanning calorimetry (DSC) [ASTM D3418
194 (ASTM 2012b)] (see Fig. 2).

195 As shown in Table 1, the glass/polyester and glass/epoxy FRP bars had approximately the same
196 fiber content (78.8% and 79.4% by weight, respectively), while the glass/vinyl-ester FRP had the
197 highest fiber–content ratio (83.9% by weight). The average cure ratios and transverse
198 coefficients of thermal expansion of the tested bars were around 99.0 ± 1.0 and
199 $19.25 \pm 1.55 \times 10^{-6} \text{C}^{-1}$, respectively, without significant differences between the three types of
200 bars tested. On the other hand, significant differences were observed for T_g and moisture uptake.
201 The vinyl-ester and polyester GFRP bars returned T_g values of 113°C and 93.0°C, respectively,
202 while the epoxy GFRP bars had a T_g value of 126°C. Similarly, the vinyl-ester and polyester

203 GFRP bars had water uptake ratios of 0.63% and 1.15%, respectively, while the epoxy GFRP
204 bars had a moisture-uptake ratio of 0.23%. The limits of water absorption of the bars at
205 saturation were <1% and <0.75% for high and medium durability, respectively, as recommended
206 in CSA S807 (2010). The measured water absorption of the polyester GFRP bars was slightly
207 higher than these limits, probably due to the resin-rich deformation pattern on the bar surface,
208 which absorbed most of the moisture.

209 **Phase II: Mechanical Characterization**

210 The mechanical characterization included testing of representative GFRP bars to determine their
211 transverse shear strength in accordance with ASTM D7617 (ASTM 2011); interlaminar shear
212 (short-beam test) in accordance with ASTM D4475 (ASTM 2008); and flexural strength and
213 flexural modulus of elasticity in accordance with ASTM D4476 (ASTM 2009). These tests were
214 selected as they are primarily related to resin properties and can provide a comparative
215 performance assessment of the three GFRP bars tested herein. Figures 3–5 show the mechanical
216 characterization tests and Table 3 lists the results. The following sections provide brief
217 descriptions and interpretation of the results.

218 **Transverse–Shear Strength Test**

219 Transverse shear is the major structural force on dowels in jointed pavements or on stirrups in
220 concrete beams. Transverse-shear tests were conducted according to ASTM D7617 (ASTM
221 2011) to characterize the tested bars. The setup consisted of a $230 \times 100 \times 110$ mm steel base
222 equipped with lower blades spaced at 50 mm face to face, allowing for the double transverse-
223 shear failure of the specimen caused by an upper blade, as shown in Fig. 3. For each type of bar
224 tested, six unconditioned specimens measuring 170 mm in length were tested under laboratory
225 conditions on an MTS 810 (MTS Systems Corporation, Eden Prairie, Minneapolis) testing

226 machine equipped with a 500 kN load cell. A displacement-controlled rate of 1.3 mm/min was
227 used during the test, which yielded between 30 and 60 MPa/min until specimen failure. The
228 loading was done without subjecting the test specimens to any shock. The transverse-shear
229 strength was calculated with Eq. (1)

$$230 \quad \tau_u = \frac{P_s}{2A} \quad (1)$$

231 where τ_u = transverse-shear strength (MPa); P_s = failure load (N); and A = bar cross-sectional
232 area (mm²).

233 Table 3 shows that the transverse-shear strengths of the polyester and vinyl-ester GFRP bars
234 were 250±33 and 258±32 MPa, respectively. The epoxy GFRP bars had the highest value of
235 transverse-shear strength (270±45 MPa). It is worth mentioning, however, that, although the
236 resin delivers most of the transverse-shear strength, the fiber and the fiber/resin interface also
237 play a role (Montaigu et al. 2013). The ratios between the shear strengths of the polyester and
238 vinyl-ester GFRP bars and that of epoxy bars were 93% and 96%, respectively. The results
239 indicate that the epoxy resin yielded higher transverse-shear strength than the polyester and
240 vinyl-ester resin, although the standard deviation was high. Moreover, these values meet CSA
241 requirements (2010), which specify a minimum transverse-shear strength of 160 MPa for GFRP
242 bars.

243 **Three-Point Flexural Test**

244 Flexural testing is especially useful for quality control and specification purposes. Flexural
245 properties may vary with specimen diameter, temperature, weather conditions, and differences in
246 rates of straining. The flexural properties obtained with this test method—ASTM D4476 (ASTM
247 2009)—cannot be used for design purposes but are appropriate for the comparative testing of
248 composite materials. The test was conducted on specimens 240 mm long over a simply

249 supported span equal to 20 times the bar diameter, as shown in Fig. 4. Six unconditioned
 250 specimens were tested under laboratory conditions as references for each type on an MTS 810
 251 testing machine equipped with a 500 kN load cell. The specimens were loaded at mid-span with
 252 a circular nose; the specimen ends rested on two circular supports that allowed the specimens to
 253 bend. A displacement-controlled rate of 3.0 mm/min was used during the test. The rate of
 254 loading was done without subjecting the test specimen to any shock. The applied load and
 255 deflection were recorded during the test on a data-acquisition system monitored by a computer.
 256 The flexural strength of tested FRP specimens was calculated with Eq. (2). The flexural modulus
 257 of elasticity (stiffness) is the ratio, within the elastic limit, of stress to corresponding strain. It
 258 was calculated with Eq. (3)

$$259 \quad f_u = PLC / (4I) \quad (2)$$

$$260 \quad E = PL^3 / (48IY) \quad (3)$$

261 where f_u = flexural strength in the outer fibers at mid-span (N/mm²); P = failure load (N); L =
 262 clear span (mm); I = moment of inertia (mm⁴); C = distance from the centroid to the extremities
 263 (mm); E = flexural modulus of elasticity in bending (N/mm²); and Y = mid-span deflection at
 264 load P (mm).

265 The maximum outer fiber strain (ϵ_u) was calculated from Eq. (4)

$$266 \quad \epsilon_u = f_u / E \quad (4)$$

267 Table 3 provides the three-point flexural strength, flexural modulus of elasticity, and ultimate
 268 outer-fiber strain of the tested GFRP bars. The elastic behavior of all the specimens was
 269 maintained until flexural failure, at which point the specimens failed due to compression in the
 270 top fibers, as shown in Fig. 4. The polyester GFRP bars showed the lowest flexural strength
 271 (1150±59 MPa), while the epoxy GFRP bars recorded the highest (1573±135 MPa). The vinyl-

272 ester GFRP bars recorded a flexural strength of 1432 ± 75 MPa. The vinyl-ester and epoxy GFRP
273 bars, however, evidenced no significant differences in flexural modulus of elasticity (66.3 and
274 61.8 GPa, respectively). Lastly, the flexural modulus of elasticity of the polyester resin was
275 lower than that of the vinyl-ester and epoxy resin (86% and 92% of the vinyl-ester and epoxy
276 GFRP bars, respectively). The lower flexural strength and modulus of the polyester GFRP bars is
277 expected since the polyester had the lowest mechanical properties of the thermosetting resins
278 considered (Table 1). Castro and Carino (1998) pointed out that the resin system significantly
279 affected the mechanical properties of FRP bars due to the efficiency of the stress transfer among
280 the fibers.

281 **Short-Beam Shear Test**

282 In FRP bars manufactured with a pultrusion process in which the fibers are arranged
283 unidirectionally and bonded with the polymer matrix, the horizontal stresses would be more
284 conducive to inducing interface degradation than transverse-shear stresses (Park et al. 2008). The
285 short-beam shear test was conducted according to ASTM D4475 (ASTM 2008) on six specimens
286 of each type of GFRP bar in order to calculate the interlaminar-shear strength, which is governed
287 by the fiber–matrix interface. The tests were carried out with a 500 kN MTS 810 testing
288 machine. The distance between the shear planes was set to 7 times the nominal diameter of the
289 FRP bars. Figure 5 shows the test setup and typical modes of failure of the tested specimens. A
290 displacement-controlled rate of 1.3 mm/min was employed during the test. The applied load was
291 recorded with a computer-monitored data-acquisition system.

292 The interlaminar-shear strength, S_u , of the GFRP bars was calculated from Eq. (5)

293
$$S_u = 0.849P / d^2 \quad (5)$$

294 where S_u = interlaminar-shear strength (MPa); P = shear failure load (N); and d = bar diameter
295 (mm).

296 The short-beam shear test revealed that the epoxy GFRP bars had the highest interlaminar-shear
297 strength (77.4 ± 2.7 MPa), followed by the vinyl-ester GFRP bars (64.8 ± 4.5 MPa) and the
298 polyester GFRP bars (47.2 ± 0.4 MPa). The results confirm that the interface between the glass
299 fibers and polyester resin was not as strong as that within the vinyl-ester and epoxy GFRP bars.
300 Table 3 shows the apparent horizontal shear strength of the tested GFRP bars. It is worth
301 mentioning that the high values of the interlaminar-shear strength reveal a strong interface
302 between the resins and reinforcing fibers, which will be clarified in the SEM analysis to follow.

303 **Phase III: Durability Study in Alkaline Solution**

304 **Conditioning of the GFRP Bars in Alkaline Solution**

305 Accelerated aging tests were conducted in accordance with ASTM D7705 (ASTM 2012a). The
306 conditioning of the bars included the combined exposure to a harsh alkaline environment and
307 elevated temperature. Immersion in an aqueous media (alkaline solution) at high temperature
308 accelerates degradation. The alkaline solution was prepared with calcium hydroxide, potassium
309 hydroxide, and sodium hydroxide (118.5 g of $\text{Ca}(\text{OH})_2$ + 0.9 g of NaOH + 4.2 g of KOH in 1 L
310 of deionized water) according to ASTM D7705 and CSA S806 (CSA 2012) . The pH of the
311 alkali solution was 12.8. The three types of FRP bars—glass/polyester, glass/vinyl-ester, and
312 glass/epoxy—were immersed in this solution at 60°C for up to 5,000 h. The timing of
313 conditioning started once the solution had reached the prescribed temperature. Robert et al.
314 (2009) reported that the degradation reaction rate increased almost linearly between room
315 temperature and 50°C , whereas the increase was exponential at higher temperatures (over 60°C).

316 Therefore, to avoid any thermal degradation, the maximum conditioning temperature used in this
317 study was 60°C, as specified in ASTM D7705 (ASTM 2012a).

318 The GFRP specimens were placed in hermetically sealed stainless-steel containers to prevent
319 excessive evaporation and the reaction of atmospheric CO₂ with calcium hydroxide. The
320 containers were placed in an environmental chamber adjusted to the prescribed temperature
321 (60°C) under isothermal conditions. The bars were weighed and their diameters measured
322 throughout the conditioning period to monitor water absorption and eventually characterize the
323 mass and diameter changes. Observation revealed no changes in diameter during conditioning.
324 Six specimens of each type of FRP bar were removed from the solution and tested to determine
325 their transverse-shear strength, interlaminar-shear strength, flexural properties, and physical
326 properties after 1,000, 3,000, and 5,000 h at 60°C. Durability was assessed using tests for
327 transverse-shear strength [ASTM D7617 (ASTM2011)], interlaminar shear (short-beam test)
328 [ASTM D4475 (ASTM 2008)], flexural strength, and flexural modulus of elasticity
329 [ASTMD4476 (ASTM 2009)]. Degradation mechanisms in FRP bars are typically denoted as
330 (1) fiber dominated; (2) matrix dominated; and (3) fiber–matrix interface dominated or combined
331 mechanisms. Changes in mechanical properties determined by these tests are indicators of the
332 three specific modes of degradation of the FRP constituent materials given earlier: fibers
333 (flexural tests), resin (transverse and short-beam shear tests), and interface region (short-beam
334 shear and flexural tests). The results for the conditioned specimens were compared to those of
335 the reference ones.

336 **Transverse-Shear Strength of the Conditioned GFRP Bars**

337 Table 4 shows the transverse-shear strength and strength-retention ratios of the tested bars after
338 1,000, 3,000, and 5,000 h of immersion in the alkaline solution at 60°C. Table 4 indicates that

339 the polyester GFRP bars were highly affected by accelerated aging with a transverse-shear
340 strength reduction of 22.5% after 5,000 h of immersion, while the vinyl-ester and epoxy bars had
341 transverse-shear strength reductions of 15.9% and 11%, respectively. Figure 6(a) shows the
342 effect of the alkaline solution on the transverse shear strength after different exposure times.
343 Contrary to the polyester bars, the vinyl-ester and epoxy GFRP bars exhibited no significant
344 reductions in the early stages (less than 3,000 h).

345 **Flexural Strength of the Conditioned FRP Bars**

346 Table 4 provides the flexural strength and strength-retention ratios of the tested FRP bars after
347 1,000, 3,000, and 5,000 h of immersion. Both the polyester and epoxy GFRP bars had similar
348 flexural-strength reductions after 5,000 h (25 and 23%, respectively), while the vinyl-ester GFRP
349 bars showed a lower reduction of 17%. These observations confirm that the bond between the
350 GFRP fibers and polyester resin—before and after conditioning—was lower than that between
351 the glass fibers in the vinyl-ester or epoxy resin. Consequently, debonding occurring at the fiber–
352 matrix interface caused the fibers to separate from the resin. Figure 6(b) shows the effect of the
353 alkaline solution on flexural strength. The lowest reduction rate was observed with the vinyl-
354 ester GFRP bars, which yielded the lowest degradation at the interface. The high degradation of
355 the epoxy GFRP bars after 1,000 h of conditioning resulted from the ingress of the alkaline
356 solution through the initial voids. The polyester GFRP bars, however, returned an almost steady
357 degradation rate between 1,000 and 5,000 h.

358 **Flexural Modulus of Elasticity of the Conditioned GFRP Bars**

359 Table 4 gives the flexural modulus of elasticity and the retention ratio of the tested FRP bars
360 after 1,000, 3,000, and 5,000 h of immersion. The three bar types had no significant differences
361 in flexural modulus of elasticity after 5,000 h. The reduction ranged from 10.7% to 12.6% in

362 comparison to the references. Figure 6(c) illustrates the effect of the alkaline solution on the
363 flexural modulus of elasticity, with all types of bar specimens exhibiting a steady reduction rate.

364 **Interlaminar-shear strength of the Conditioned GFRP Bars**

365 Table 4 also shows the apparent horizontal shear (interlaminar shear) strength and strength-
366 retention ratios of the tested FRP bars after 1,000, 3,000, and 5,000 h of immersion. As for
367 flexural testing, the vinyl-ester and epoxy GFRP bars offered excellent stability and durability
368 after immersion in the alkaline solution, followed by the polyester GFRP bars. The reduction
369 ratios for the vinyl-ester, epoxy, and polyester GFRP bars after 5,000 h were 13%, 13%, and
370 21%, respectively. Again, this observation confirms the strong fiber–resin interface in the vinyl-
371 ester GFRP bars, followed by the epoxy and polyester GFRP bars. As evidenced from these
372 results, the fiber–resin interface stands out as one of the most important issues in manufacturing
373 glass FRP. Figure 6(d) shows the effect of the alkaline solution on the interlaminar-shear
374 strength, with the vinyl-ester GFRP bars exhibiting the lowest rate of degradation. Interestingly,
375 the 21% reduction in the interlaminar-shear strength of the polyester GFRP bars in this study is
376 significantly lower than with the polyester E-glass composite rods tested by Micelli and Nanni
377 (2004), who observed a more than 90% reduction in interlaminar-shear strength. This indicates
378 that the development of new material systems and advanced manufacturing methods now yield
379 high-quality FRP bar products.

380 **Microstructural Analysis of the Reference and Conditioned GFRP Bars**

381 SEM observations were performed to investigate microstructural changes in the GFRP bars
382 before and after conditioning. The specimens were cut, polished, and coated with a thin layer of
383 gold/palladium in a vapor-deposit process. The analysis was carried out on a JEOL JSM-840 A
384 microscope (JEOL, Akishima, Tokyo, Japan). Figure 7 shows the SEM micrographs of the cross

385 section of the reference GFRP bars, while Figs. 8 to 10 provide the SEM micrographs of the
386 5,000 h conditioned specimens.

387 SEM analysis of the reference and conditioned specimens (Figs. 7 to 10) indicates that the GFRP
388 bars made with vinyl-ester and epoxy evidenced no significant changes, but presented a slight
389 debonding at the interface between the fibers and vinyl-ester resin. Consequently, the vinyl-ester
390 GFRP bars evidenced higher moisture uptake measured at saturation compared to the epoxy
391 GFRP bars. On the other hand, the GFRP bars containing the polyester resin evidenced
392 significant impact on the coating with the 5000 h conditioning. Moreover, these bars experienced
393 greater debonding at the fiber–resin interface than did the vinyl-ester and epoxy GFRP bars.
394 Accordingly, the polyester GFRP bars had higher moisture uptake measured at saturation and
395 higher degradation rate of mechanical properties after conditioning.

396 SEM was also performed on the fracture zones of the 1,000 h specimens after short-beam testing
397 (Fig. 11) to investigate the mechanisms of failure at the interface fiber–matrix. The fiber surface
398 of the vinyl-ester and epoxy GFRP bars had more resin coverage (Fig. 11 [b and c]) than the
399 polyester GFRP bars (Fig. 11[a]). This observation corroborates the reduction ratio of the
400 interlaminar-shear strength and flexural strength after conditioning in the alkaline solution and
401 characterizes the higher bonding of the glass fiber to the vinyl-ester and epoxy resins than the
402 polyester resin.

403 **Glass Transition Temperature and Cure Ratio of the Conditioned GFRP Bars**

404 Differential scanning calorimetry (DSC) is used to obtain information about the thermal behavior
405 and characteristics of polymeric materials and composites, such as the glass transition
406 temperature (T_g) and curing process. In this study, 30–50 mg specimens from both unconditioned
407 and conditioned specimens were sealed in aluminum pans and heated in a TA Instruments (New

408 Castle, Delaware) DSC Q10 calorimeter to 200°C at a rate of 20°C/min. The glass transition
409 temperature (T_g) was determined in accordance with ASTM D3418 (ASTM 2012b).

410 Table 5 presents the T_g values for the reference and specimens conditioned for 5,000 h. The T_g of
411 the conditioned polyester GFRP bars were slightly higher than that of the reference specimens,
412 as a result of post-curing at high temperature. The vinyl-ester and epoxy GFRP bars were almost
413 fully cured (99.1% and 100%, respectively); their T_g values were lower than that of the reference
414 specimens by 11.5% and 10.3%, respectively. Epoxy resin is known to lower T_g when water is
415 absorbed (plasticizing effect). The water absorption of the epoxy GFRP bars was 0.2%.

416 **Chemical Changes in the Conditioned GFRP Bars**

417 Fourier transform infrared spectroscopy (FTIR) was used to identify any chemical
418 change/degradation after 5,000 h of conditioning at 60°C. FTIR spectra of the surface and core of
419 the material specimens were recorded using a Jasco 4600 spectrometer equipped with an
420 attenuated total-reflectance device. Five hundred and twelve scans were routinely acquired at a
421 resolution of 4 cm^{-1} . Chemical degradation in the alkaline solution is mainly due to a hydrolysis
422 reaction, which forms new hydroxyl (-OH) groups from sensitive units, such as ester groups.
423 Hydroxyl groups appeared as a broad peak between 3200 and 3650 cm^{-1} , which corresponds to
424 the stretching mode of the hydroxyl groups in the polyester, vinyl-ester, and epoxy resins.

425 Figure 12 shows the FTIR spectra of the unconditioned and conditioned polyester, vinyl-ester,
426 and epoxy GFRP specimens conditioned in the alkaline solution for 5,000 h at 60°C. For each
427 specimen—reference and conditioned—spectra of the surface and core of the specimen were
428 recorded and the areas of the O-H and C-H peaks calculated as presented in Fig. 13. Table 6
429 presents the ratio of the (OH⁻) peak to the resin's carbon-hydrogen (C-H) stretching peak. The
430 table indicates that none of the hydroxyl peaks for any of the tested vinyl-ester and epoxy GFRP

431 specimens evidenced any significant changes, which equates to no increase in the amount of
432 hydroxyl groups in the resins. This observation shows that the vinyl-ester and epoxy resins used
433 did not degrade chemically while the specimens were immersed at 60°C for 5,000 h. On the other
434 hand, the polyester resin showed significant differences on the surface and in the core of the
435 tested specimens (see Table 6). The experimental O–H/C–H for the core and surface of the vinyl-
436 ester and epoxy GFRP bars immersed for 5,000 h were 1.5, 1.8, 1.2, and 1.5, respectively,
437 compared to 1.80, 2.40, 1.25, and 1.6 for the unconditioned specimens, while the experimental
438 ratios for the core and surface of the polyester GFRP bars immersed for 5,000 h at 60°C were 3.5
439 and 14.30, respectively. These results led to the conclusion that chemical degradation of the
440 polymer occurred on the surface of the polyester bars, which was in direct contact with the
441 solution during immersion. This observation explains the losses in mechanical properties of the
442 polyester GFRP bars.

443 **Moisture Uptake at Saturation of the Conditioned GFRP Bars**

444 The moisture uptake at saturation of the reference and conditioned FRP bars was determined
445 according to ASTM D570 (ASTM 2010). The gain in mass was corrected to account for
446 specimen mass loss due to possible dissolution phenomena. This correction was achieved by
447 completely drying the immersed specimens in an oven at 100°C for 24 h and comparing their
448 masses to their initial masses. The mass loss may have occurred due to several causes:
449 dissolution of soluble chemicals present on the surface; sand debonding in the case of sand-
450 coated bars; and chemical degradation of one of the components, such as resin hydrolysis. In this
451 study, the moisture-uptake ratios at saturation for the reference specimens were 1.15%, 0.63%,
452 and 0.23% for the polyester, vinyl-ester, and epoxy GFRP, respectively, while these ratios for the
453 conditioned specimens were 1.36%, 0.38%, and 0.20% for the polyester, vinyl-ester, and epoxy

454 GFRP bars, respectively. The epoxy GFRP bars had the lowest water uptake, which is consistent
455 with the lowest degradation of the fiber–resin interface as determined by DSC analysis and SEM
456 observations.

457 **Summary and Conclusions**

458 This study investigated glass-fiber-reinforced polymers (GFRPs) with polyester, vinyl-ester, and
459 epoxy resins. Based on the results, the following conclusions concerning the glass FRP bars
460 made with polyester, vinyl-ester and epoxy resins tested herein can be drawn:

- 461 1. The epoxy and vinyl-ester GFRP bars exhibited higher fiber–resin bond; flexural strength;
462 flexural modulus of elasticity; and interlaminar-shear strength, which is governed by the fiber–
463 matrix interface. In addition, they showed lower moisture uptake.
- 464 2. Both the polyester and epoxy GFRP bars had similar flexural-strength reductions after 5,000 h
465 of immersion (25% and 23%, respectively), while the vinyl-ester GFRP bars returned a lower
466 reduction of 17%. These observations confirm that the bond between the GFRP fibers and
467 polyester resin—before and after conditioning—was lower than that between the glass fibers and
468 the vinyl-ester or epoxy resin.
- 469 3. The unconditioned polyester GFRP bars exhibited lower transverse-shear strength, flexural
470 strength, interlaminar-shear strength, and the weakest fiber–resin interface. The transverse-shear
471 strength of the polyester GFRP bars was significantly affected by accelerated aging (22.5%
472 reduction after 5,000 h), while the epoxy and vinyl-ester GFRP bars were slightly affected by
473 accelerated aging (11% and 15.9 % reductions, respectively, after 5,000 h).
- 474 4. The flexural strength of the polyester GFRP bars was significantly affected by accelerated
475 aging (25% reduction after 5,000 h), while the vinyl-ester and epoxy GFRP bars were affected
476 by accelerated aging (17% and 23% reductions, respectively, after 5,000 h).

477 5. The interlaminar-shear strength of the polyester GFRP bars was highly affected by accelerated
478 aging (21% reduction after 5,000 h), while the vinyl-ester and epoxy GFRP bars were slightly
479 affected by accelerated aging (13% reduction each after 5,000 h). The fiber–resin interface plays
480 a significant role in controlling the degradation due to conditioning.

481 6. The microstructural observations revealed that GFRP bars made with vinyl-ester or epoxy
482 resin were not significantly changed, but presented a slight debonding at the interface between
483 the fibers and vinyl-ester resin. Consequently, the vinyl-ester GFRP bars evidenced higher
484 moisture uptake measured at saturation compared to the epoxy GFRP bars.

485 7. The debonding at the interface between the fibers and polyester resin was higher than in the
486 vinyl-ester and epoxy GFRP bars. Accordingly, the polyester GFRP bars evidenced higher
487 moisture uptake measured at saturation and a higher degradation rate of mechanical properties
488 after conditioning.

489 8. The polyester GFRP bars showed an increase in T_g of about 5°C after conditioning due to
490 post-curing (cure ratio of the reference specimens was 98.1%). The vinyl-ester and epoxy GFRP
491 bars, however, experienced a decrease in T_g after conditioning.

492 9. The polyester GFRP bars absorbed 18% more water than the vinyl-ester and epoxy GFRP bars
493 after conditioning compared to the reference specimen.

494

495 **Acknowledgments**

496 The authors wish to acknowledge the financial support of the Natural Sciences and Engineering
497 Research Council of Canada (NSERC), the NSERC Research Chair in Innovative FRP
498 Reinforcement for Concrete Structures, the Fonds de la recherche du Quebec en nature et
499 technologies (FRQ-NT), the Florida Department of Transportation, and the University of North
500 Florida. The authors would like to thank Firep International AG (Switzerland) for donating the
501 GFRP materials and the technical staff of the structural & materials lab in the Department of
502 Civil Engineering at the University of Sherbrooke.

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669

670 **Table 1.** Typical properties of the thermosetting resins (Bank, 2006)

Property	Resin system		
	Polyester	Vinyl-ester	Epoxy
Glass transition temperature (T_g), °C	100	110	120
Tensile modulus, GPa	4.0	3.5	3.0
Tensile strength, MPa	65	82	90
Elongation at break, %	2.5	6.0	8.0

671

672 **Table 2.** Physical properties of the reference GFRP bars

Property	GFRP bar type		
	Polyester	Vinyl-ester	Epoxy
Fiber content by weight (%)	78.8	83.9	79.4
Cure ratio (%)	98.1	99.1	100
Transverse CTE, ($\times 10^{-6} \text{°C}^{-1}$)	20.8	17.7	19.7
Glass transition temperature, T_g (°C)	93.0	113	126
Moisture uptake (%)	1.15	0.63	0.23

673

674 **Table 3.** Mechanical properties of the reference GFRP bars

Bar type	τ_u (MPa)	f_u (MPa)	E (GPa)	ϵ_u (%)	S_u (MPa)
Glass/polyester	250±33	1150±59	56.9±2.4	2.02±0.16	47.2±0.4
Glass/vinyl- ester	258±32	1432±75	66.3±2.2	2.16±0.089	64.8±4.5
Glass/epoxy	270±45	1573±135	61.8±1.5	2.54±0.015	77.4±2.7

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Table 4. Retention of mechanical properties of the conditioned GFRP bars

Fiber/resin	Conditioned period	τ_u (MPa)	Retention (%)	f_u (MPa)	Retention (%)	E (GPa)	Retention (%)	S_u (MPa)	Retention (%)
Glass/poly ester	1,000	236	94.4	1133	99	55.0	96.6	43.8	93
	3,000	222	88.8	939	81	54.0	94.9	40.8	87
	5,000	194	77.5	863	75	50.8	89.3	37.4	79
Glass/vinyl-ester	1,000	248	96.1	1409	98	64.0	96.5	62.5	97
	3,000	234	90.7	1273	89	61.1	92.2	58.0	90
	5,000	217	84.1	1186	83	58.5	88.2	56.0	87
Glass/epoxy	1,000	267	98.9	1446	92	59.0	95.5	73.7	96
	3,000	248	92.0	1301	83	57.5	93.0	69.6	90
	5,000	239	89.0	1211	77	54.0	87.4	67.0	87

678

679

Table 5. Cure ratio, T_g , and moisture uptake of the reference and conditioned GFRP bars

Property	GFRP bar type					
	Polyester		Vinyl-ester		Epoxy	
	Reference	5,000 h	Reference	5,000 h	Reference	5,000 h
Cure ratio (%)	98	100	99	99	100	100
T_g (°C)	93	98	113	100	126	112
Moisture uptake (%)	1.15	1.36	0.63	0.38	0.23	0.20

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682

Table 6. Ratio of the FTIR peaks

Test location	OH/CH ratio					
	Polyester		Vinyl-ester		Epoxy	
	Reference	5,000 h	Reference	5,000 h	Reference	5,000 h
Surface	2.60	14.3	2.40	1.80	1.60	1.50
Core	1.60	3.50	1.80	1.50	1.25	1.20

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691 mode

692 Figure 4. Setup for flexural testing and typical failure mode: (a) test setup; (b) failure mode

693 Figure 5. Setup for short-beam testing and typical failure mode: (a) test setup; (b) failure mode

694 Figure 6. Effect of conditioning in the alkaline solution at 60°C on mechanical properties:
695 (a) transverse-shear strength; (b) flexural strength; (c) flexural modulus of elasticity;
696 (d) interlaminar-shear strength

697 Figure 7. Micrographs of the cross section of the reference FRP bars

698 Figure 8. Micrographs of the fiber/matrix interface of a glass/epoxy bar before and after
699 conditioning: (a) before conditioning; (b) after conditioning

700 Figure 9. Micrographs of the fiber–matrix interface of a glass/polyester bar before and after
701 conditioning: (a) before conditioning; (b) after conditioning

702 Figure 10. Micrographs of the fiber–matrix interface of a glass/vinyl-ester bar before and after
703 conditioning: (a) before conditioning; (b) after conditioning

704 Figure 11. Micrographs of bars conditioned in the alkaline solution for 1,000 h at 60°C (after
705 interlaminar shear failure): (a) glass/epoxy; (b) glass/polyester; (c) glass/vinyl-ester

706 Figure 12. FTIR spectra of reference and specimens conditioned for 5,000 h

707 **Figure 13.** Peak areas used to calculate a O–H/C–H

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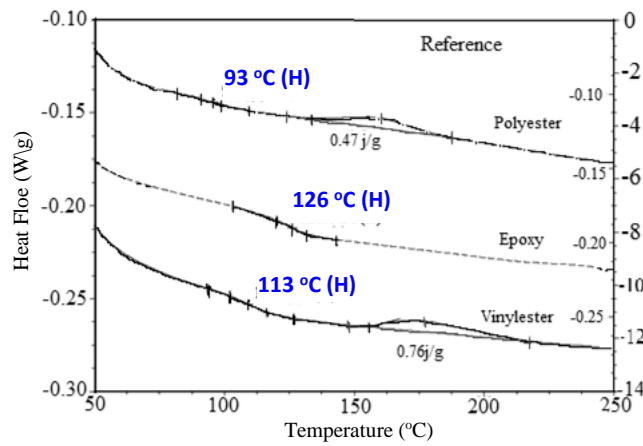


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Figure 1. Tested GFRP bars



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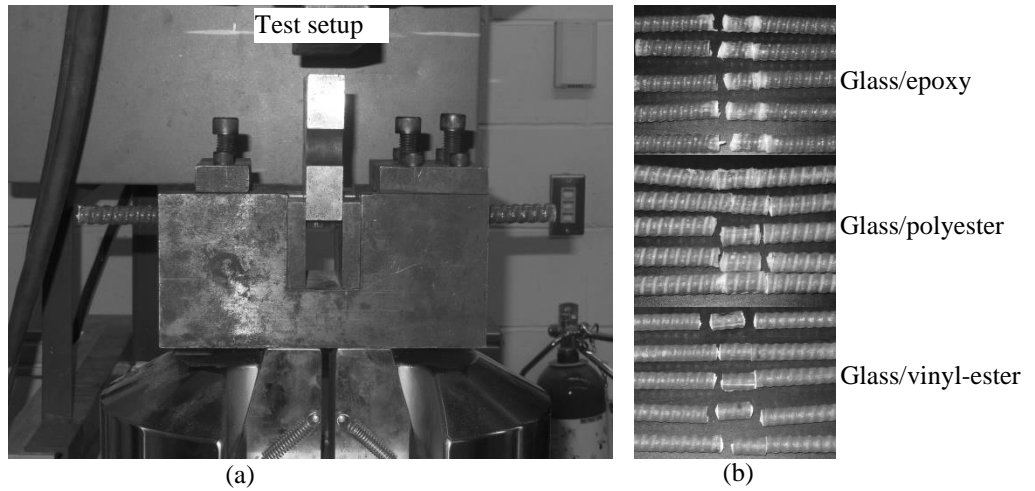
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Figure 2. DSC scans for glass transition temperature (T_g)

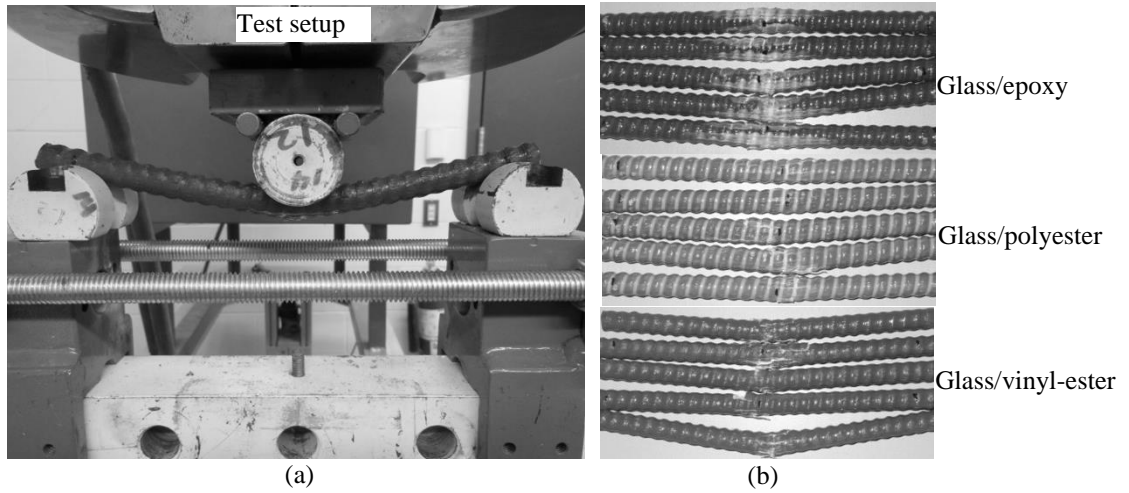


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Figure 3. Setup for transverse-shear test and typical shear failure mode: (a) test setup; (b) failure

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mode



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Figure 4. Setup for flexural testing and typical failure mode: (a) test setup; (b) failure mode

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736 **Figure 5.** Setup for short-beam testing and typical failure mode: (a) test setup; (b) failure mode

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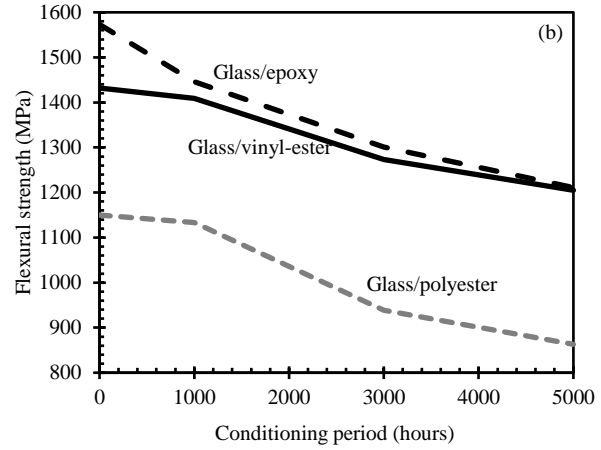
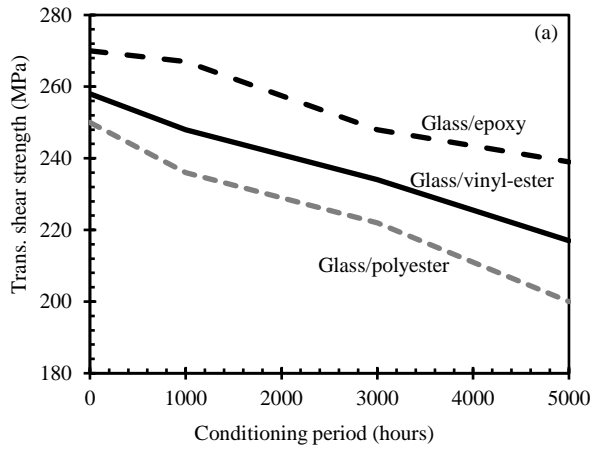
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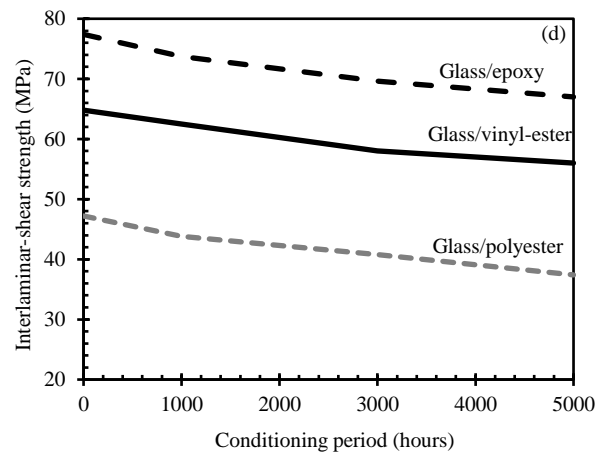
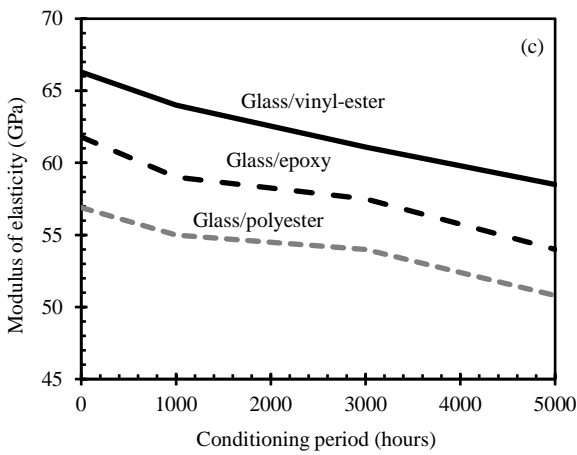


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(a) transverse-shear strength

(b) flexural strength



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(c) flexural modulus of elasticity

(d) interlaminar-shear strength

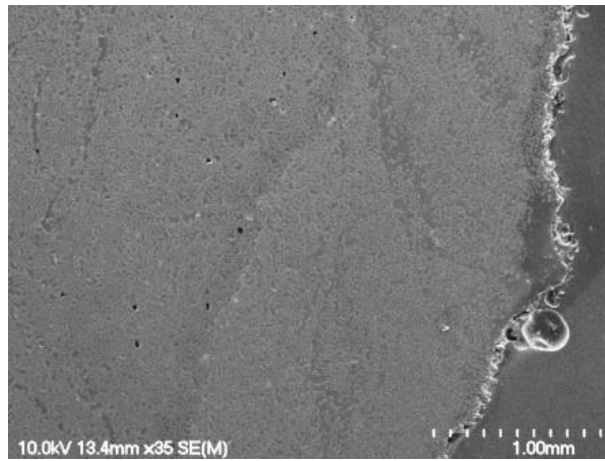
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Figure 6. Effect of conditioning in the alkaline solution at 60°C on mechanical properties:

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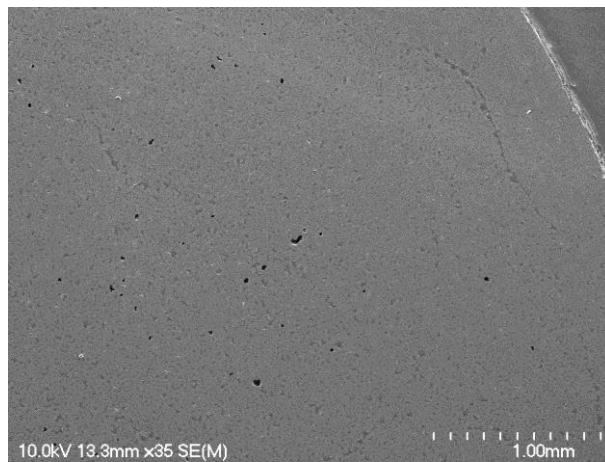
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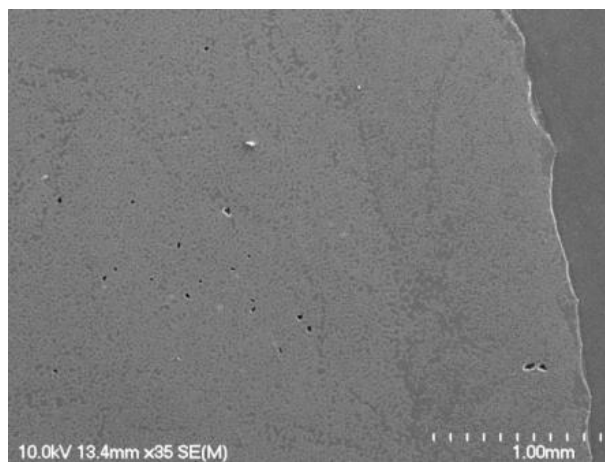
(a) Polyester GFRP bar

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(b) Vinyl-ester GFRP bar

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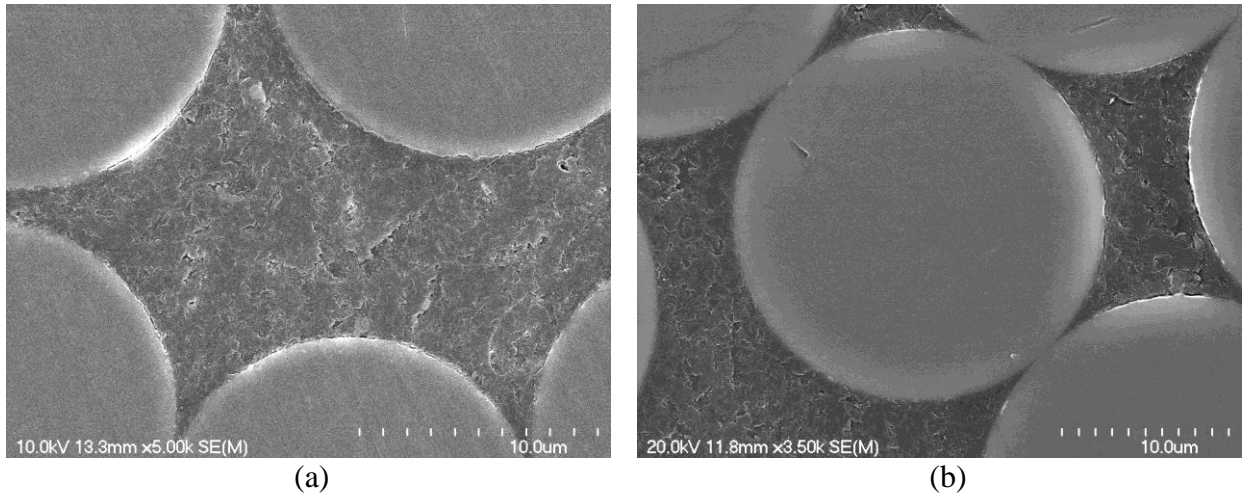
(c) Epoxy GFRP bar

Figure 7. Micrographs of the cross section of the reference GFRP bars

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769 **Figure 8.** Micrographs of the fiber–matrix interface of an epoxy GFRP bar before and after
770 conditioning: (a) before conditioning; (b) after conditioning

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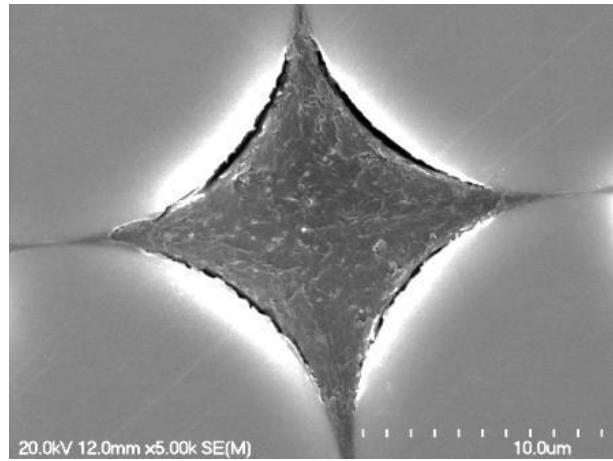
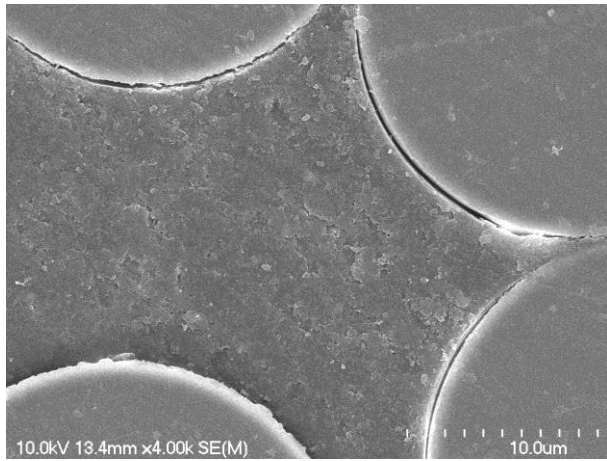
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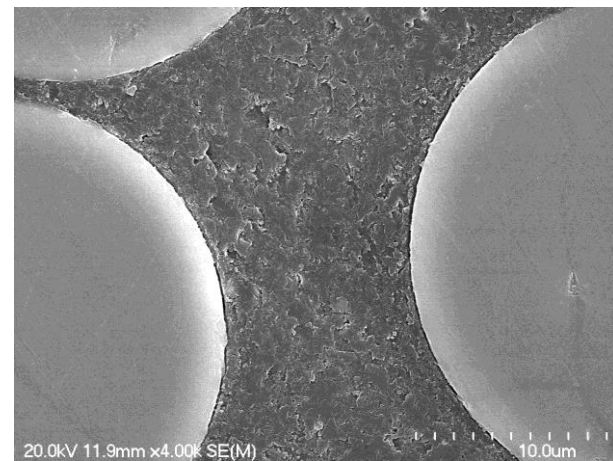
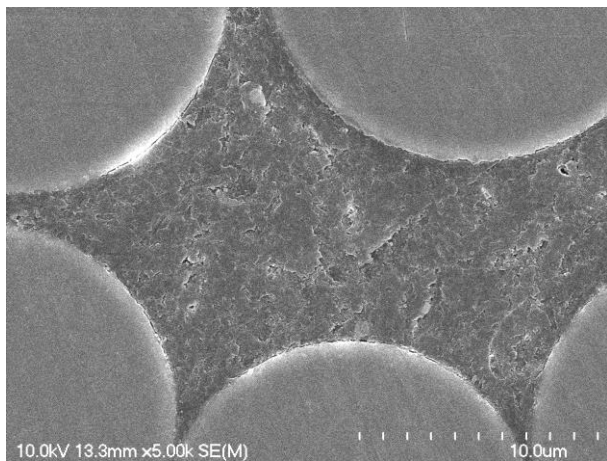
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(a)

(b)

Figure 9. Micrographs of the fiber–matrix interface of a polyester GFRP bar before and after conditioning: (a) before conditioning; (b) after conditioning



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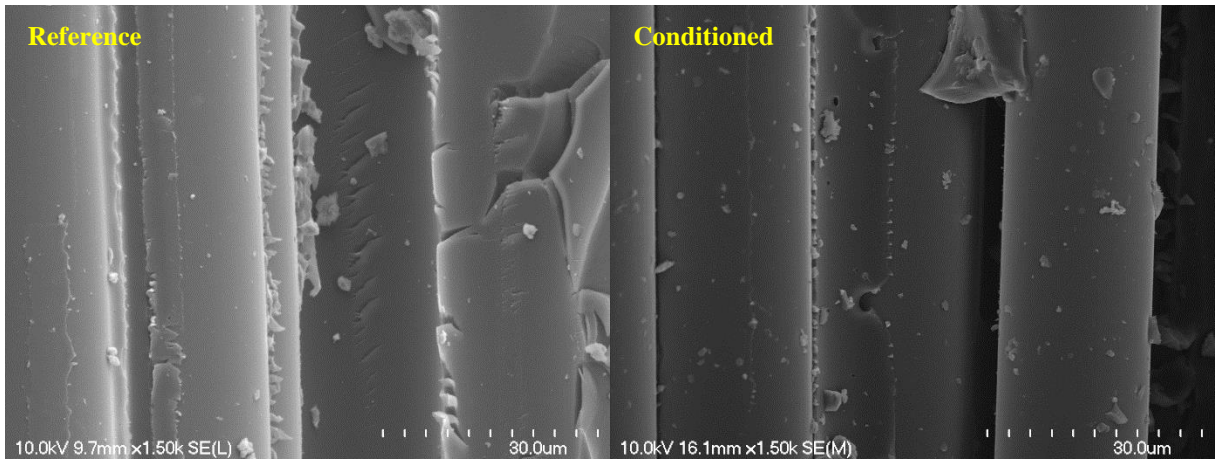
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(a)

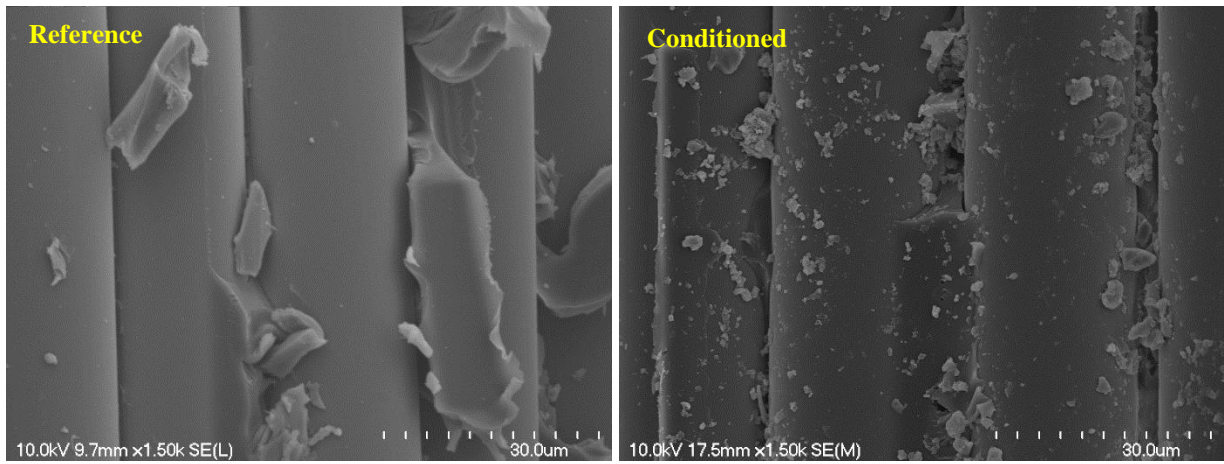
(b)

Figure 10. Micrographs of the fiber/matrix interface of a vinyl-ester GFRP bar before and after conditioning: (a) before conditioning; (b) after conditioning



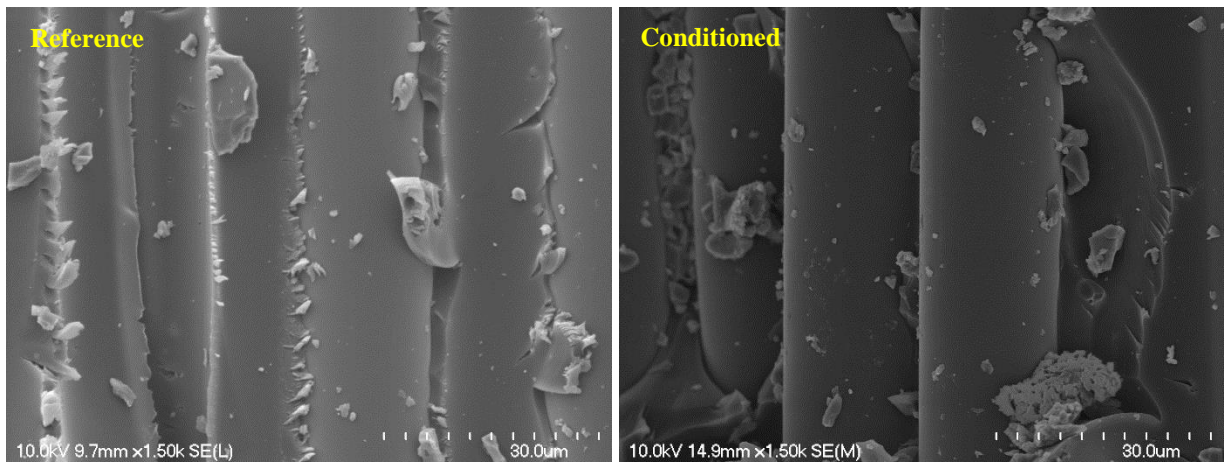
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(a)



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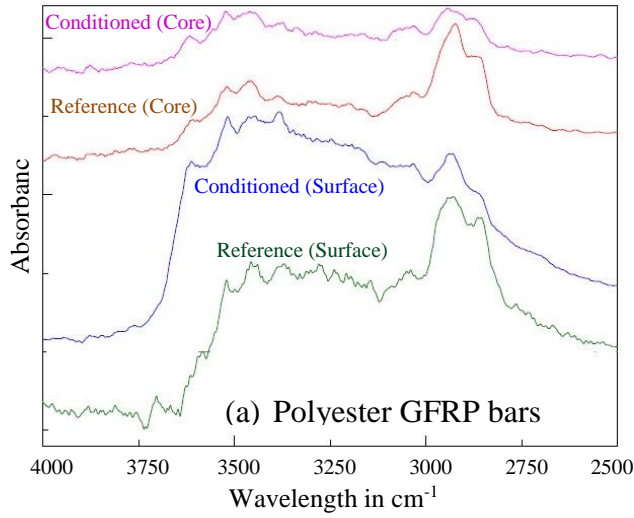
(b)



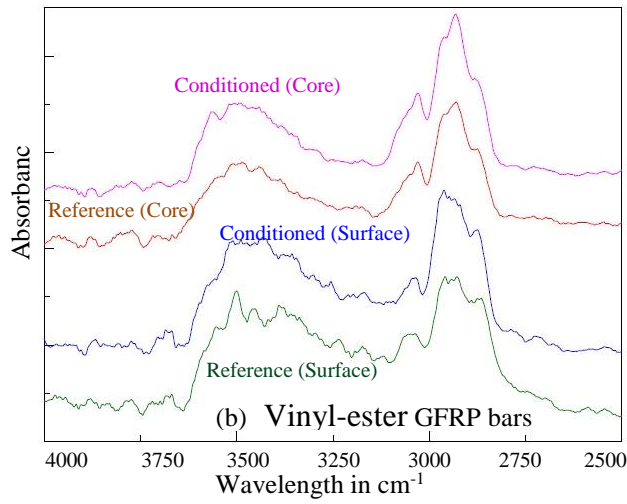
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(c)

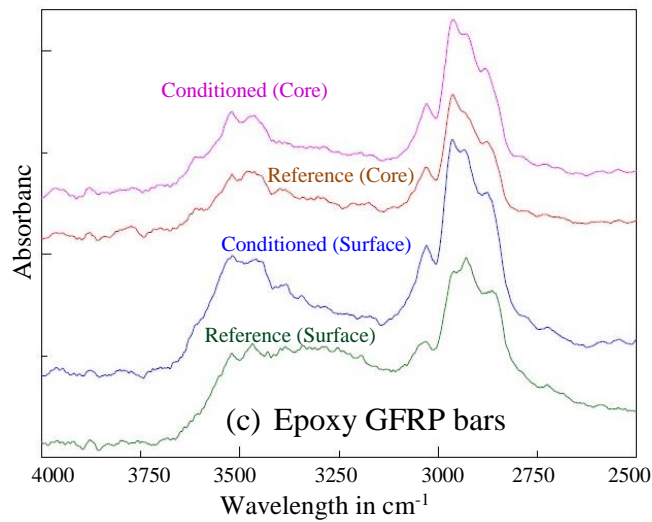
800 **Figure 11.** Micrographs of bars conditioned in the alkaline solution for 1,000 h at 60°C (after
801 interlaminar shear failure): (a) polyester GFRP; (b) vinyl-ester GFRP; (c) epoxy GFRP



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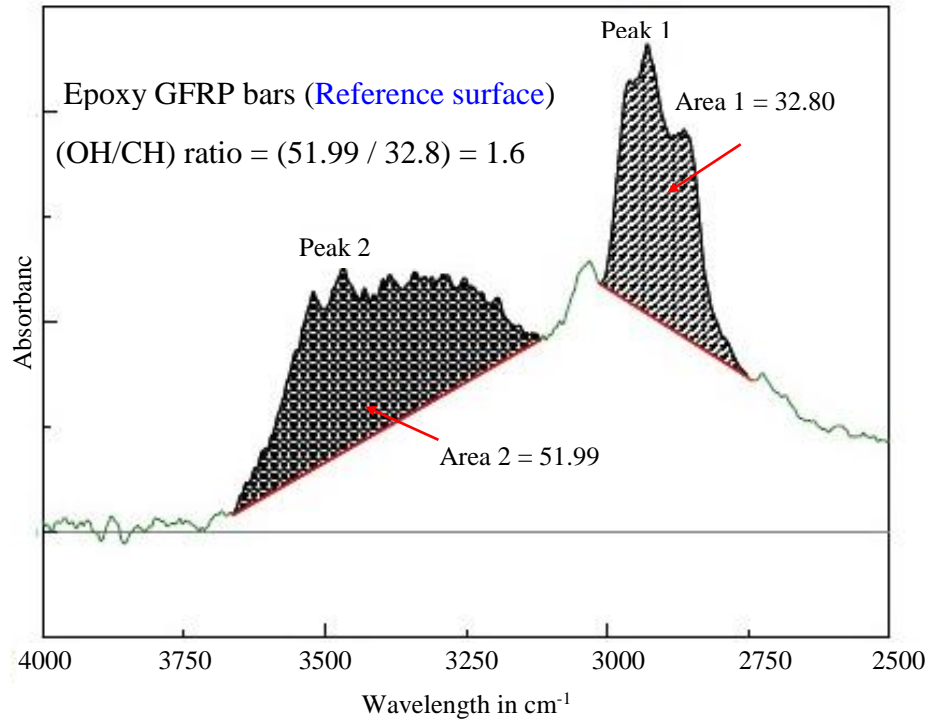


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Figure 12. FTIR spectra of reference and specimens conditioned for 5,000 h



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Figure 13. Peak areas used to calculate a O–H/C–H