

Article

Effect of Harsh Environmental Conditions on the Impact Response of Carbon Composites with Filled Matrix by Cork Powder

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Abstract: Composites are used in a wide range of engineering applications, as a result, exposure to hostile environments is rather common and its mechanical properties degradation is unavoidable. It is necessary to have a complete understanding of the impact of hostile environments on mechanical performance, namely critical solicitations as low velocity impacts. Therefore, this work intends to analyse the low velocity impact response of a carbon fibre/epoxy composite, and a similar architecture with an epoxy matrix filled with cork, after immersion into different solutions: diesel, H₂SO₄, HCl, NaOH, distilled water, seawater, and seawater at 60 °C. These solutions significantly affected the impact properties. In this context, the maximum load, maximum displacement, and restored energy behaviour were studied to understand the influence of exposure time. It was possible to conclude that such impact parameters were significantly affected by the solutions, where the exposure time proved to be determinant. The benefits of cork on the perforation threshold were investigated, and this parameter increased when the epoxy matrix was filled with cork. Finally, cork filled epoxy laminates also show less variation in maximum load and recovered energy than carbon/epoxy laminates.

Keywords: composite laminates; hostile solutions; experimental tests; low velocity impact



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1. Introduction

Fibre-reinforced composites have been used in a variety of engineering fields such as aircraft, space, automotive, sport, marine industries, and military applications due to their excellent performance in terms of high specific strength and stiffness, good static and dynamic properties, good corrosion resistance, adjustable properties, competitive cost, and fast manufacture [1–4]. Carbon fibres, for example, have great strength and hardness, as well as excellent temperature resistance, chemical resistance, and low thermal expansion. They are ideal candidates for use in the aerospace/aeronautical, automotive, construction, military, and sports industries due to their advantages [1,3,5–9].

Numerous studies have been conducted to increase interfacial adhesion with the matrix [10–14], as a consequence of the low surface energy and chemically inert surface of some fibres. On the other hand, literature reports that the addition of low concentrations of nanoparticles to the matrix is an excellent solution to improve the mechanical performance of composite laminates without compromising their density, toughness or manufacturing process [4,15,16]. Nowadays, for reasons of environmental sustainability, composites incorporating natural reinforcements have gained popularity due to their low density, abundance, abrasiveness during processing, low cost, renewable, and biodegradable properties [17]. In this context, cork has unique characteristics, and its powder, which is a by-product of agriculture, is a viable option for use in polymeric composites. For example,

literature reports that, compared to laminates with neat matrices, polymer composites containing cork powder have higher absorbed impact energy and glass transition temperature, promote less damage areas and have the best elastic recovery performance. At the same time, it became clear that cork powder increases the penetration threshold [17–19]. Therefore, high tolerance to damage from impact loads, wear and fire resistance, low thermal conductivity, and excellent damping are important characteristics for the use of cork in composites used in harsh environments [20–23].

The open literature reports several studies on the effect of hostile environments on the mechanical properties of composite materials. For example, the effects of alkaline (NaOH) and acid (HCl) solutions in fibre-reinforced polymer composites were studied by Amaro et al. [24] and, according to the authors, alkaline solutions proved to be more harmful than acid solutions, resulting in lower flexural strength and modulus. Another study showed that flexural strength is insensitive up to 30 days after immersion in HCl, after which there is a 10% decrease [25]. Kawada and Srivastava [26] studied the exposure of a composite laminate to a corrosive environment and observed that sharp cracks begin to develop and spread when the acid weakens the fibres, resulting in very low strength and highly flat fracture with low failure stress. The resins used as matrix in laminated composites, according to Banna et al. [27], have a considerable influence on the final mechanical performance. When subjected to higher temperatures or for longer periods of time, authors conclude that polyester resin has a lower modulus than bisphenol A epoxy vinyl ester. Due to the importance of the matrix to the composite structural integrity, the epoxy resin's ability to deal with progressively adverse conditions will determine the composite's suitability for specific applications [27].

According to the literature, composite laminates are strong in the in-plane loading direction, but very weak in the out-of-plane loading direction [28]. Impact damage is believed to be the main source of composite delamination in service, which is quite dangerous because it significantly affects the performance of these materials [8,9,29] and, simultaneously, is difficult to identify visually [30]. However, there is a lack of research that incorporates impact loads and harsh environmental conditions, in particular involving composites reinforced with cork. Therefore, the main goal of this work is to investigate the benefit of cork powder on low velocity impact strength in carbon/epoxy composites after immersion into hydrochloric acid (HCl), sodium hydroxide (NaOH), sulphuric acid (H₂SO₄), diesel, distilled water, and seawater.

2. Material and Experimental Procedure

Nine ply laminates of carbon fibre bidirectional plain weave fabric (taffeta weave with 160 g/cm²), all in the same direction, and an Ampreg 22 epoxy resin with an Ampreg 22 hardener standard, both supplied by Gurit, were used to produce composite laminates. Plates with overall dimensions of 330 × 330 × 2.4 ± 0.1 (mm³) were produced by the hand lay-up process. This system was placed inside a vacuum bag and a load of 2.5 kN was applied for 48 h to maintain a constant fibre volume fraction and a uniform laminate thickness. During the first 10 h the bag remained attached to a vacuum pump to eliminate any air bubbles existing in the composite. The post-cure was carried out in an oven at 45 °C for 48 h.

Using the same manufacturing process, composite laminates with epoxy matrix filled with cork powder were also produced. The bulk density of the cork powder used is about 0.11 g·cm⁻³ and the particles' size, in terms of percentile, is d(0.1) = 18.6 µm, d(0.5) = 78.9 µm and d(0.9) = 208.3 µm. More details can be found in [17]. The cork powder was dried in an oven (Heraeus, model UT 6060) at about 120 °C during 2 h and dried in a desiccator until use. Epoxy resin and cork powder were mixed at 900 rpm for 2 h and, at same time, subjected to ultrasonic bath sonicator. Finally, the mixture was degassed in a vacuum oven, followed by addition of the hardener with special care to avoid the presence of air bubbles. The filler content was 3 wt.% of the epoxy resin-hardener mixture as reported in [19].

The samples used in this study were cut from these plates to square specimens with 100 mm side, which were completely submerged into different solutions and different immersion times, both summarized in Table 1. All solutions have a concentration of 10% by weight (wt.%), which corresponds to a pH of 13.0 for NaOH and 1.5 for acids. Except for seawater at 60 °C, all the other solutions in which the specimens were immersed are at room temperature. Finally, the samples were washed with clean water and dried at room temperature.

Table 1. Different solutions and immersion times used in this study.

Solutions	Immersion Time (Days)
Diesel	15, 30 and 45
Sulphuric acid (H ₂ SO ₄), pH = 1.5	10, 20 and 30
Hydrochloric acid (HCl), pH = 1.5	10, 20 and 30
Sodium hydroxide (NaOH), pH = 13.0	10, 20 and 30
Distilled water	15, 30, 60 and 90
Seawater at room temperature	15, 30, 60 and 90
Seawater at 60 °C	15, 30 and 45

Low-velocity impact tests were performed using a drop weight testing machine IMATEK-IM10. More details of the impact machine can be found in [31]. An impactor diameter of 20 mm with a mass of 3.005 kg was used. The tests were performed on square section samples of dimensions 75 × 75 mm and the impactor stroke at the centre of the samples obtained by centrally supporting the 100 × 100 mm specimens. Impact energies of 2, 4, 8, 10, 12, 16, 20 and 24 J were used to analyse the impact strength and the effect of cork powder on the impact strength. The effect of hostile solutions on the impact strength was evaluated for the energy of 12 J. For each condition/environment, five specimens were tested, and the results presented in terms of average values.

3. Results

The benefits obtained with cork powder were evaluated by impact tests carried out for different impact energies. Figure 1 shows typical load and energy versus time curves of control samples and laminates with resin filled with 3% of cork powder tested for an impact energy of 2 J.

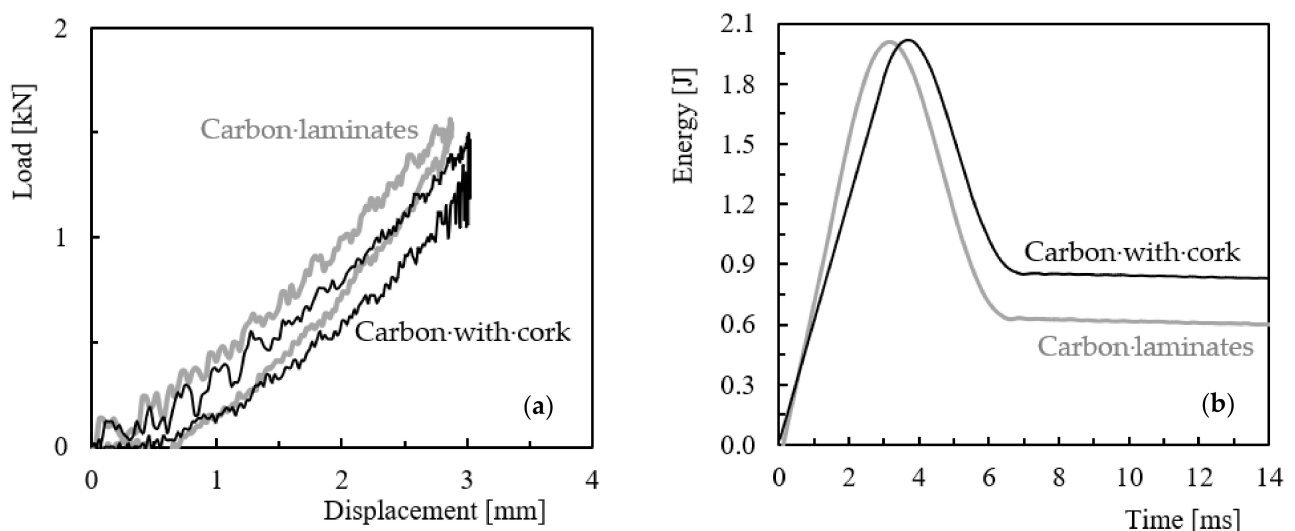


Figure 1. For an impact energy of 2 J, typical: (a) load versus time curves; (b) energy versus time curves.

Both curves shown in Figure 1 represent the typical profile of all tests and are in good agreement with the literature [8,32–34]. The oscillations in the load-displacement curves (Figure 1a) are caused by the elastic wave and the vibrations of the samples [35]. In detail, the load-displacement curves show that the load rises until it reaches a maximum value, and then drops abruptly after reaching its maximum value. A non-perforating impact was identified because the impact energy was insufficient to completely penetrate the sample. In fact, the impactor stuck into the sample and always rebounded. Therefore, the beginning of the plateau in the energy-time curves (Figure 1b) corresponds to the loss of contact between the striker and the specimen [19,36], so this energy is the one absorbed by the specimen. Finally, in both figures it is possible to observe the influence of the resin filled with cork powder on the impact behaviour of the composite. For example, Figure 1a shows an increase in displacement with the presence of cork powder, while Figure 1b shows less absorbed energy and longer contact time.

In detail, the effect of the cork powder is shown in Figure 2 in terms of maximum load, maximum displacement, and restored energy for all impact energies. Symbols represent average values. This figure shows how these parameters evolve with impact energy, while Table 2 summarizes all average values and their respective statistical variations in terms of standard deviation.

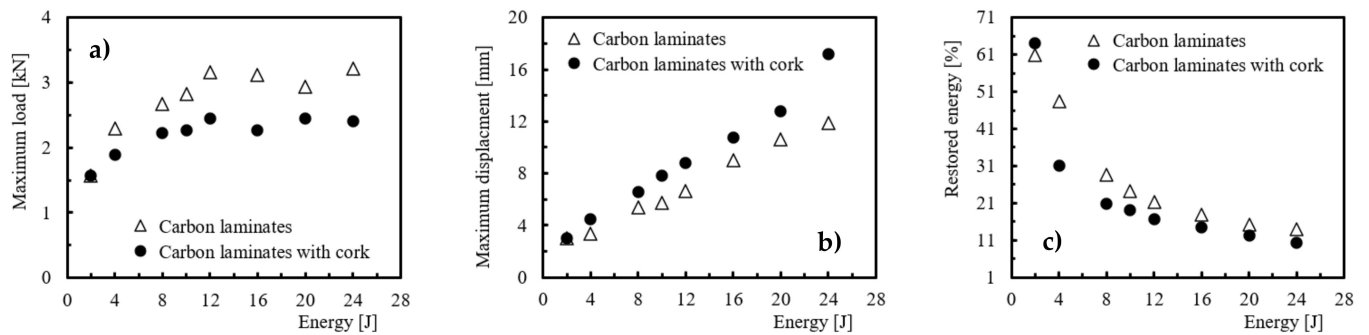


Figure 2. For different impact energies: (a) maximum load, (b) maximum displacement, (c) restored energy.

Table 2. Summary of all parameters obtained from the impact tests and respective standard deviation.

Impact Energy (J)	Maximum Load (kN)		Maximum Load (mm)		Contact Time (ms)		Restored Energy (%)	
	Average	Std Dev	Average	Std Dev	Average	Std Dev	Average	Std Dev
Carbon Laminates								
2	1.56	0.21	3.0	0.3	7.24	0.29	60.8	2.1
4	2.29	0.18	3.3	0.5	7.07	0.46	48.5	2.3
8	2.67	0.20	5.4	0.3	7.73	0.35	28.6	1.4
10	2.82	0.22	5.7	0.6	7.93	0.36	24.3	1.9
12	3.16	0.23	6.6	0.3	8.04	0.31	21.4	1.2
16	3.12	0.19	9.0	0.3	8.55	0.35	17.8	1.6
20	2.93	0.17	10.6	0.7	9.77	0.64	15.1	1.3
24	3.21	0.24	11.9	0.4	10.49	0.67	13.9	1.1
Carbon Laminates with Cork								
2	1.56	0.21	3.0	0.2	7.55	0.32	64.2	3.2
4	1.89	0.23	4.5	0.2	8.51	0.29	31.0	3.9
8	2.22	0.19	6.6	0.3	9.31	0.37	20.7	3.5
10	2.27	0.22	7.8	0.3	10.38	0.24	19.1	2.7
12	2.44	0.19	8.8	0.5	10.17	0.43	16.6	2.8
16	2.27	0.17	10.8	0.6	10.95	0.39	14.4	3.6
20	2.44	0.19	12.8	0.2	11.89	0.31	12.2	3.1
24	2.41	0.24	17.2	0.3	15.68	0.37	10.3	3.0

Regarding the maximum load (Figure 2a and Table 2), and regardless of the impact energy, it is possible to observe higher values for composites with neat resin than for composites with resin filled with cork powder. On the other hand, for both laminates, higher impact energies promoted higher maximum loads up to 12 J, after which the maximum impact load seems to remain constant. While the maximum load increased around 56.4% between 2 J and 12 J for composites with cork powder, this value was about 102.6% (around twice higher) for composite laminates with neat resin.

In fact, literature reports that the maximum load increases with increasing impact energy [16,19,31,37], and this trend can be observed in this study up to 12 J for both laminates. According to Gustin et al. [38] the differences observed in the maximum loads are a consequence of the different failure modes introduced in the laminate and, in this context, it is possible to note that for impact energies higher than 12 J the severity of the damage is so significant that the effect is not visible in terms of maximum load. Similar behaviour was observed by Reis et al. [37], as well as the non-linearity also observed in Figure 2a and that according to Hosur et al. [39], the maximum load should increase almost linearly with the increase of the impact energy. This parameter is controlled by the impact energy and reflects the maximum load that the composite laminate can tolerate before severe damage occurs [37].

The benefits obtained with the cork powder and respective influence of impact energy on the displacement is shown in Figure 2b and Table 2. Independently of the impact energy, the average results show that laminates with cork powder have the highest displacements. For the studied energy range, for example, displacements increased around 298% and 476% for laminates with neat resin and laminates with epoxy filled by cork powder, respectively. Consequently, as shown in Table 2, the contact time is higher for laminates with cork powder. Under compressive loading during the impact, when cork deforms, the cell walls bend and buckle and can undergo large strain deformation. Therefore, this explains the higher displacements and the lowest maximum loads observed in composites that incorporate cork powder. On the other hand, when cell walls bend and buckle, they can absorb large amounts of energy with high viscoelastic return. This means that, after an impact, the capacity of the cork to continue to absorb energy is almost unchanged due to its elastic deformation [40,41]. The benefits reported are expressed in Figure 2c, where the lower restored energy to laminates with cork powder is a consequence of the higher energy absorption capacity. For carbon/epoxy laminates, it is possible to observe a decrease in the elastic recovery around 53%, between 2 and 8 J, but this value drops drastically to 77% between 2 and 24 J. When the cork powder is added to the resin, the behaviour is similar, but in this case with values around 68% and 84%, respectively.

From Figure 2c it is possible to observe that the elastic energy is never equal to zero, which means that the absorbed energy is never equal to the impact energy. Therefore, the penetration threshold was not reached because the excess energy is used to rebound the impactor [42,43]. In this context, if the elastic energy versus impact energy is plotted and the data fitted by polynomial equations, the penetration thresholds can be determined [19,43]. Figure 3 shows the methodology used to obtain the penetration threshold for all laminates, and values of 34.5 J and 37.6 J were found for control laminates and cork-filled laminates, respectively.

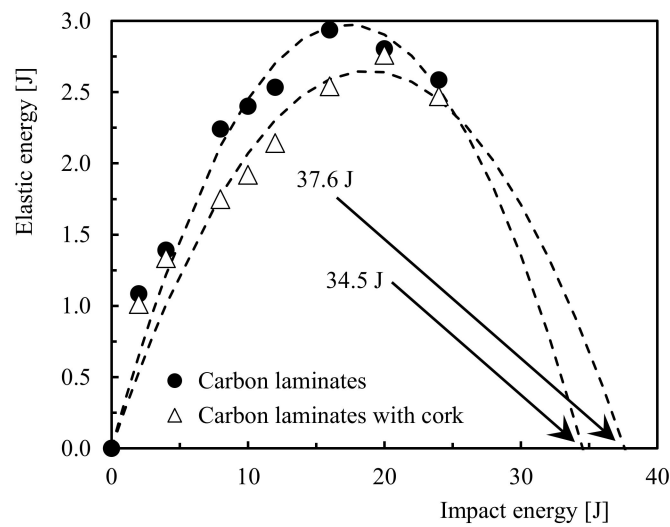


Figure 3. Penetration threshold for laminates with neat resin and matrix filled with cork powder.

The effect of hostile solutions on the impact strength was evaluated for the energy of 12 J. For each condition, five specimens were used, and the results presented in terms of average values. The influence of the exposure time on the maximum load, maximum displacement and restored energy were analysed and the results shown in Figures 4–6, respectively. In these representations, each result was dimensionless/divided by the respective value obtained with the control specimens (without immersion in any solution). The average results and respective standard deviations will be summarized in form of tables.

Therefore, the benefit achieved with the introduction of cork powder in the resin is notorious, which promotes an increase in the penetration threshold about 9% higher than that of the control samples. This is a consequence of the significant amount of energy absorbed by cork associated with the high viscoelastic return [40,41]. A similar benefit was obtained by Reis et al. [19] for Kevlar laminates, when the epoxy matrix was filled with cork powder. In this case, the penetration threshold was around 30.9 J for the control samples, while for laminates with cork powder this value was about 34.8 J, promoting, in this case, a benefit around 12.6%. However, the greatest benefit observed by the authors in relation to that obtained in the present study is due to Kevlar fibres being more tolerant to damage than carbon fibres.

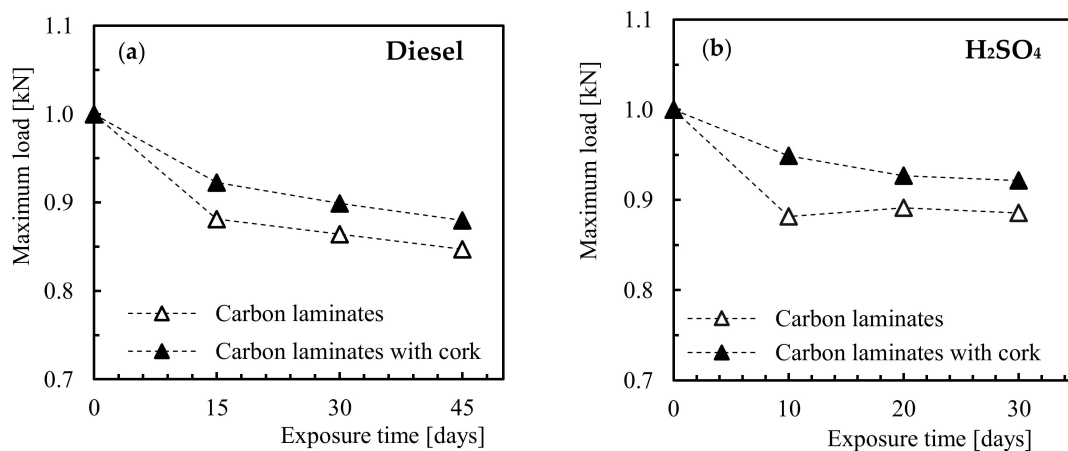


Figure 4. Cont.

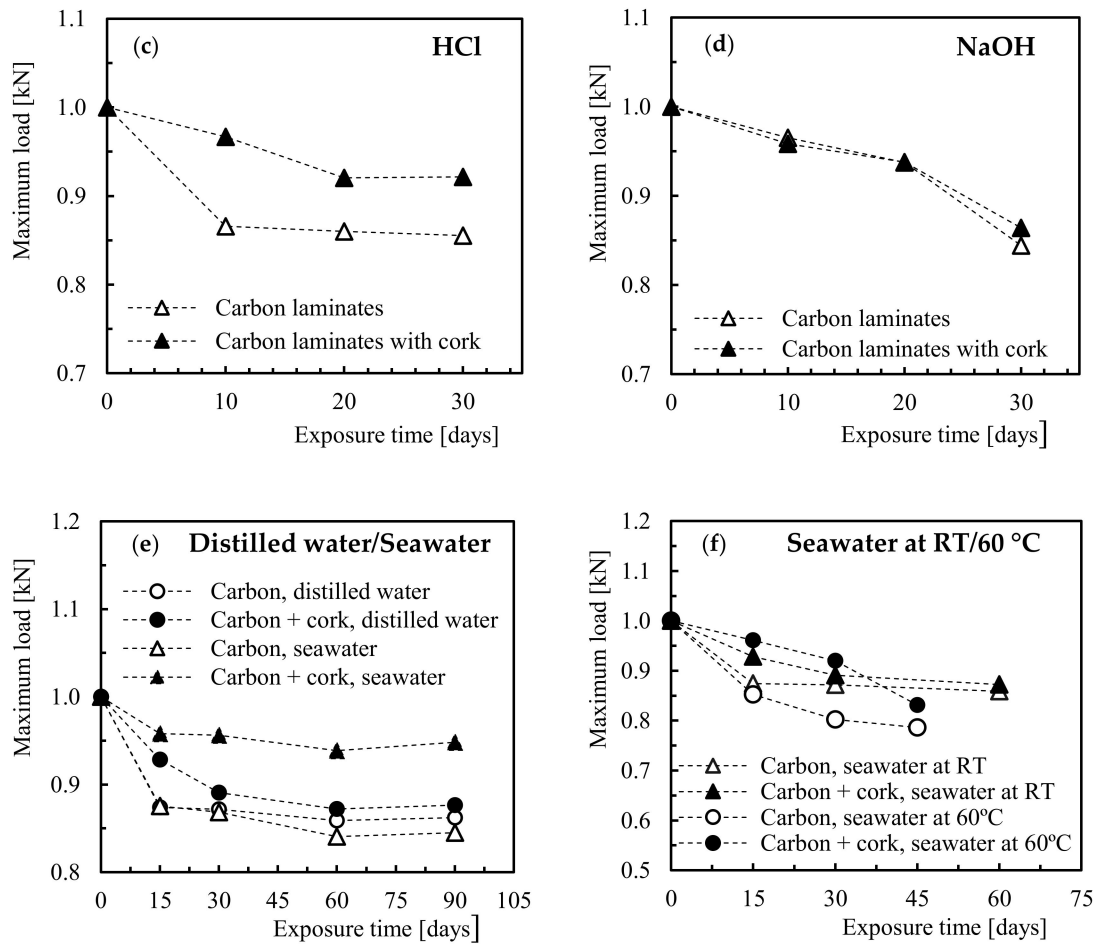


Figure 4. Influence of solution type and exposure time on the maximum impact load after immersion into: (a) diesel; (b) H_2SO_4 , (c) HCl, (d) NaOH, (e) distilled water and seawater; (f) seawater at room temperature and at 60 °C.

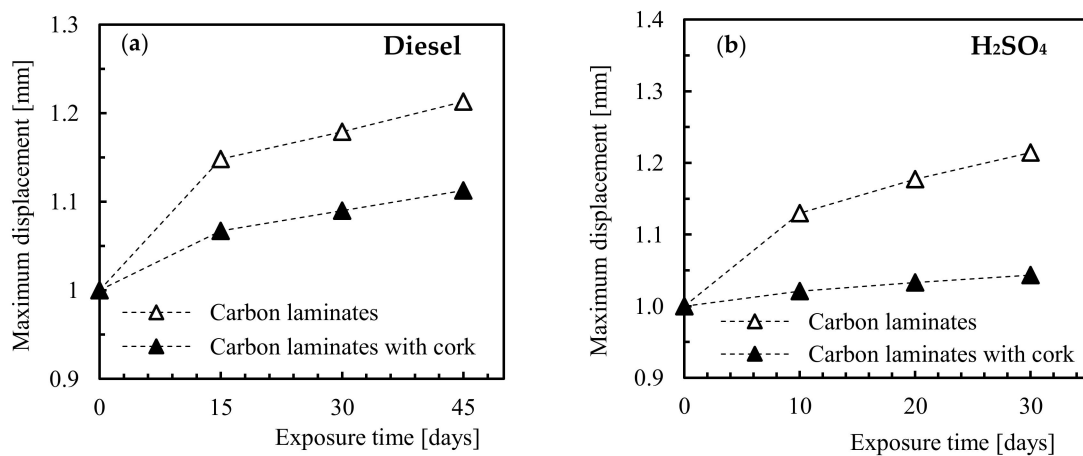


Figure 5. Cont.

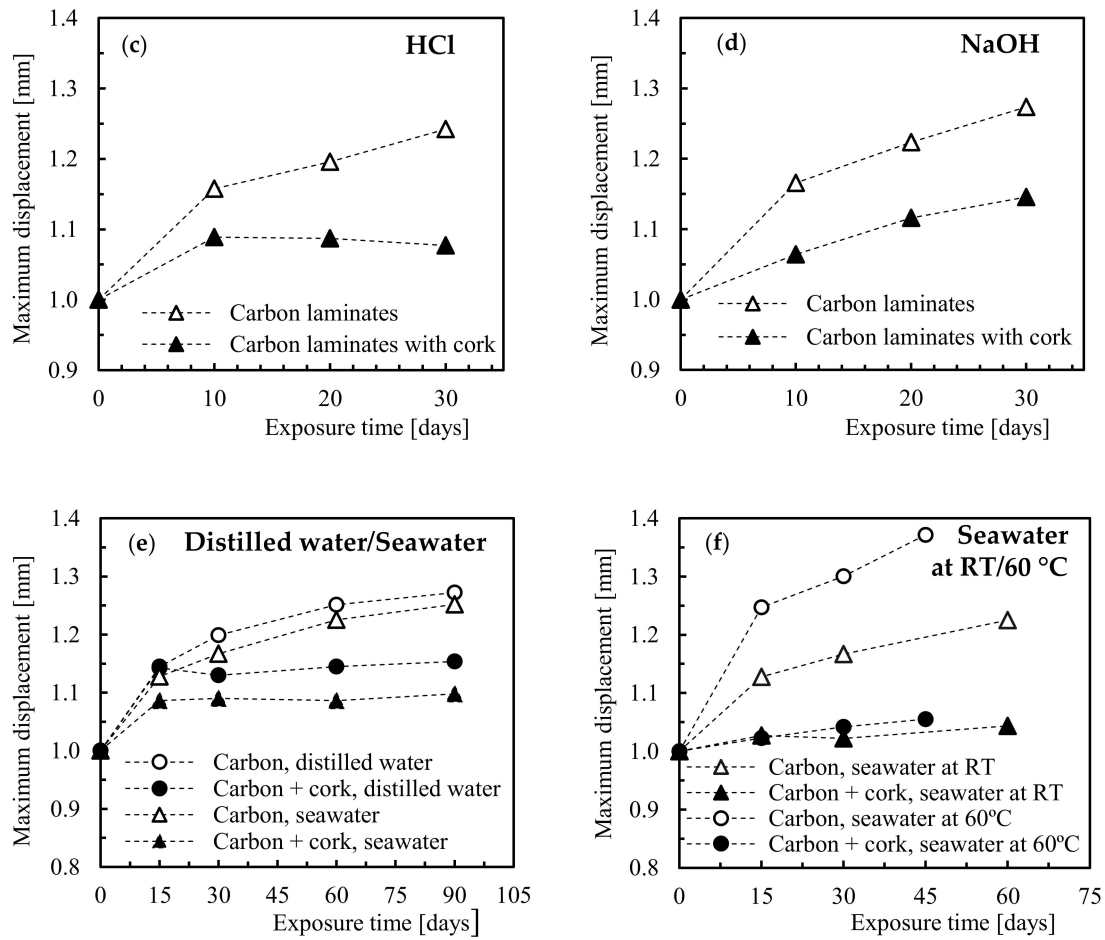


Figure 5. Influence of solution on the displacement after immersion into: (a) diesel; (b) H_2SO_4 , (c) HCl, (d) NaOH, (e) distilled water and seawater; (f) seawater at room temperature and at 60 °C.

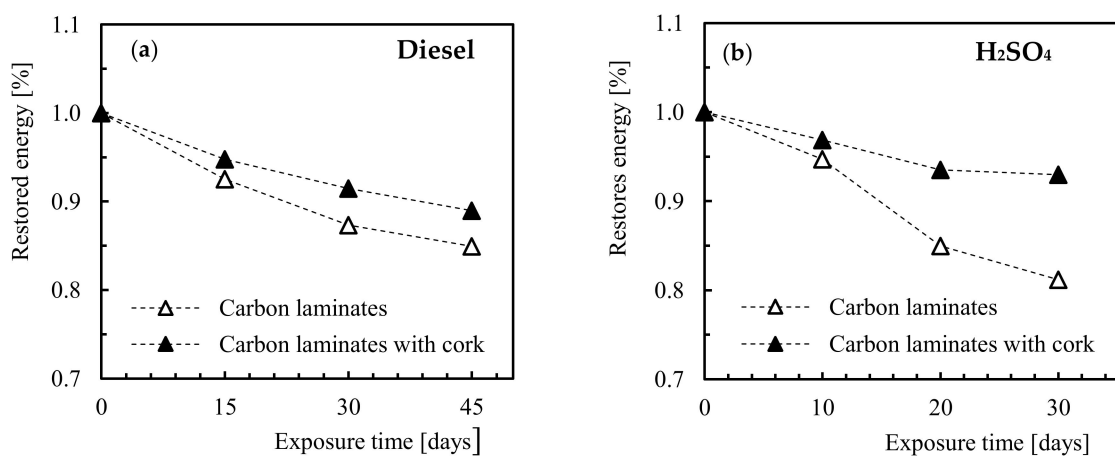


Figure 6. Cont.

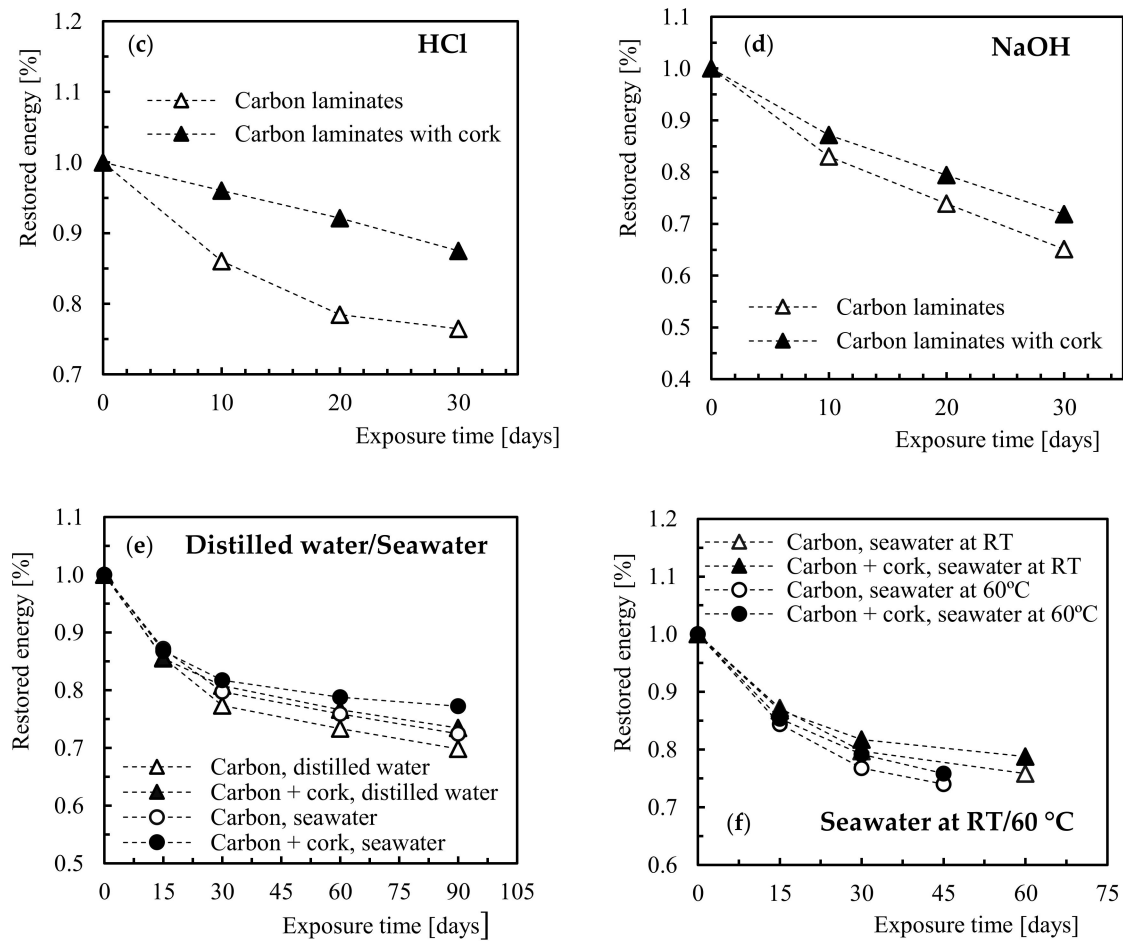


Figure 6. Influence of solution type and exposure time on the restored energy after immersion into: (a) diesel; (b) H_2SO_4 , (c) HCl, (d) NaOH, (e) distilled water and seawater; (f) seawater at room temperature and at 60 °C.

From Figure 4, it is possible to observe that, for all harsh environments, laminates with neat resin are much more sensitive to exposure to such solutions than carbon laminates with matrix filled with cork. It is also noticed that the maximum load decreases when the samples are exposed to different environments and this tendency is highly dependent on the exposure time. This evidence agrees with the studies reported in the open literature [24,44,45]. Although the values are always lower than those observed in specimens not exposed to hostile environments, Table 3 also shows that the behaviour of laminates with matrix filled with cork powder always revealed a maximum impact load lower than that observed in control laminates.

For example, comparing the maximum impact load obtained for 30 days of immersion into different solutions and the value obtained for the respective laminates without any degradation (no immersion in such solutions), it is possible to assess the severity of harsh environments in this parameter (maximum impact load). This effect is summarized in Table 4, in which the decrease observed for the different solutions is presented in percentage terms.

Table 3. Maximum load obtained for different harsh solutions and immersion time (values in kN).

Immersion Solution	Immersion Time (Days)							
	0	10	15	20	30	45	60	90
Carbon Laminates								
Diesel	3.16 (0.12)	-	2.79 (0.12)	-	2.73 (0.13)	2.68 (0.11)	-	-
H ₂ SO	3.16 (0.12)	2.79 (0.18)	-	2.82 (0.13)	2.80 (0.11)	-	-	-
HCl	3.16 (0.12)	2.74 (0.15)	-	2.72 (0.13)	2.71 (0.14)	-	-	-
NaOH	3.16 (0.12)	3.06 (0.13)	-	2.97 (0.11)	2.67 (0.13)	-	-	-
Distilled water	3.16 (0.12)	-	2.77 (0.14)	-	2.75 (0.15)	-	2.66 (0.14)	2.67 (0.18)
Seawater	3.16 (0.12)	-	2.76 (0.13)	-	2.76 (0.16)	-	2.72 (0.18)	2.73 (0.17)
Seawater at 60 °C	3.16 (0.12)	-	2.70 (0.16)	-	2.54 (0.14)	2.49 (0.18)	-	-
Carbon Laminates with Cork								
Diesel	2.44 (0.19)	-	2.25 (0.12)	-	2.19 (0.11)	2.15 (0.14)	-	-
H ₂ SO	2.44 (0.19)	2.32 (0.11)	-	2.26 (0.12)	2.25 (0.15)	-	-	-
HCl	2.44 (0.19)	2.36 (0.13)	-	2.25 (0.11)	2.21 (0.14)	-	-	-
NaOH	2.44 (0.19)	2.34 (0.12)	-	2.29 (0.13)	2.11 (0.11)	-	-	-
Distilled water	2.44 (0.19)	-	2.27 (0.16)	-	2.17 (0.15)	-	2.13 (0.12)	2.14 (0.17)
Seawater	2.44 (0.19)	-	2.34 (0.14)	-	2.33 (0.13)	-	2.29 (0.15)	2.31 (0.14)
Seawater at 60 °C	2.44 (0.19)	-	2.35 (0.17)	-	2.25 (0.20)	2.03 (0.19)	-	-

Average value (Standard deviation).

Table 4. Difference in the maximum impact load obtained for 30 days of immersion into different harsh solutions compared to non-immersed specimens.

Solutions	Matrix with Cork Powder (%)	Neat Resin (%)
Diesel	10.2	13.6
Sulphuric acid (H ₂ SO ₄)	7.8	10.8
Hydrochloric acid (HCl)	9.4	14.2
Sodium hydroxide (NaOH)	13.5	15.5
Distilled water	11.1	13.0
Sea water at room temperature	4.5	12.7
Sea water at 60 °C	7.8	19.6

According to Table 4, it is possible to conclude that the highest decreases were observed for laminates with neat resin. In this case, it is noted that the highest decrease in the maximum impact load occurs when the laminates are immersed into seawater at 60 °C (19.6%), followed by immersion in NaOH (15.5%) and HCl (14.2%). This observed trend agrees with the studies developed by Amaro et al. [24,44], where the effect of NaOH solution, compared to the HCl solution, promotes the lowest maximum load. However, the hydrochloric acid promotes the worst results compared to the sulfuric acid.

From the global point of view of all results, they can be explained by the literature due to absorption, penetration and reaction that occurred between solutions and composite constituents [25]. For example, in terms of matrix, the solution penetrates through the resin and separates it in micro-cracks [26]. Regarding the fibres, their interfaces with the matrix degrade due to dehydration of the matrix and penetration of solutions through the micro-cracks [46,47], crazes or voids in the matrix [26]. Nevertheless, according to Stamenovic et al. [47] the major influence is related to the fibre–matrix interface strength and consequent weakness of the load carrying capacity of the composite laminate. Furthermore, the highest decrease in the maximum impact load was observed when temperatures above room temperature were involved. In fact, because diffusion is a thermally activated process,

any increase in temperature accelerates short-term diffusion and increases the diffusion coefficient. Simultaneously, due to different coefficients of thermal expansion existing between fibres and matrices, residual stresses occur that are favourable to the occurrence of micro-voids or cracks in the fibre–matrix interface [48]. Under such conditions the aggressive solution creates a hydrostatic pressure at the crack tips and accelerates the crack propagation and matrix damage [49].

For example, Pavan et al. [50] developed studies on quasi-isotropic e-glass/epoxy composites immersed into artificial seawater and observed a decrease of 27% in tensile stress due to plasticization, but the failure was mainly governed by fibre failure and debonding of the fibre–matrix interface. Gargano et al. [51] reported that the lower tensile strength observed was a consequence of hydrolysis, cracks due to swelling, debonding of the fibre–matrix interface and plasticization. According to Tual et al. [52], water absorption affects the tensile strength of carbon/epoxy composites due to plasticization of the matrix and degradation of the fibre–matrix interface, but the tensile modulus is not as affected as the tensile strength. Simultaneously, authors also reported that the observed degradation is more influenced by water than by temperature, although the latter accelerates the diffusion. In terms of interlaminar shear strength (ILSS), Tual et al. [52] observed a decrease between 20% and 30%, which is explained by the authors due to matrix plasticization and degradation of the fibre–matrix interface, values that have been confirmed by other studies [53,54]. As consequence of these phenomena, impact strength is also affected [55]. In addition, Table 4 also shows that the impact load values are lower for laminates that have been immersed into distilled water than in seawater. This evidence is in line with the studies developed by Kafodya et al. [54], in which the diffusion coefficient in pultruded carbon/epoxy composites immersed into water was higher than in those immersed into seawater. According to the authors, the dissolved particles into seawater obstruct the osmotic water diffusion. For example, Hu et al. [56] observed that the water absorption of an epoxy resin is considerable higher when immersed into deionized water than salt water, but this difference increases much more for composites due to water diffusion and capillary diffusion along the fibre–matrix interface debonding.

On the other hand, when the matrix is filled with cork powder, exposure to all hostile solutions promoted lower maximum impact loads than those observed for the same conditions but in laminates with pure resin. In fact, cork is impermeable to liquids and practically impermeable to gases, thanks to the suberin and cerin present in the composition of cork cells [23,57,58]. In this context, cork powder acts as an impermeable agent and the diffusion that occurs between the cork/matrix interface is slower due to the longer and tortuous path caused by the presence of cork particles.

Figure 5 shows the effect of exposure to hostile solutions in relation to maximum displacement. Once again, each result was divided by the respective value obtained with the control specimens (no immersion in any solution). Contrary to what was observed in Figure 4, in this case, carbon laminates with neat resin are more sensitive to harsh environments regarding the maximum displacement obtained. This is because they were more degraded by the hostile solutions than those that incorporate cork. As expected by what was previously reported and justified by the open literature, in general, the maximum displacement increases when the samples are exposed to different environments and this tendency is to increase with the exposure time, which agrees with the studies published [24,44,45].

The average results and respective standard deviations are shown in Table 5. According to this table, and contrary to what was observed for the maximum impact load, the maximum displacement is higher for laminates with matrix filled with cork powder than for composites with neat resin.

Table 5. Maximum displacement obtained for different harsh solutions and immersion time (values in mm).

Immersion Solution	Immersion Time (Days)							
	0	10	15	20	30	45	60	90
Carbon Laminates								
Diesel	6.6 (0.1)	-	7.6 (0.2)	-	7.8 (0.1)	8.0 (0.2)	-	-
H ₂ SO	6.6 (0.1)	7.5 (0.3)	-	7.8 (0.2)	8.0 (0.4)	-	-	-
HCl	6.6 (0.1)	7.6 (0.5)	-	7.9 (0.2)	8.2 (0.3)	-	-	-
NaOH	6.6 (0.1)	7.7 (0.1)	-	8.1 (0.1)	8.4 (0.2)	-	-	-
Distilled water	6.6 (0.1)	-	7.6 (0.2)	-	7.9 (0.3)	-	8.3 (0.1)	8.4 (0.5)
Seawater	6.6 (0.1)	-	7.4 (0.1)	-	7.7 (0.4)	-	8.1 (0.1)	8.3 (0.4)
Seawater at 60 °C	6.6 (0.1)	-	8.2 (0.3)	-	8.6 (0.3)	9.1 (0.4)	-	-
Carbon Laminates with Cork								
Diesel	8.8 (0.3)	-	9.4 (0.2)	-	9.6 (0.4)	9.8 (0.3)	-	-
H ₂ SO	8.8 (0.3)	9.0 (0.4)	-	9.1 (0.6)	9.2 (0.5)	-	-	-
HCl	8.8 (0.3)	9.6 (0.3)	-	9.6 (0.2)	9.5 (0.3)	-	-	-
NaOH	8.8 (0.3)	9.4 (0.1)	-	9.8 (0.2)	10.1 (0.2)	-	-	-
Distilled water	8.8 (0.3)	-	10.1 (0.4)	-	10.0 (0.3)	-	10.1 (0.2)	10.2 (0.2)
Seawater	8.8 (0.3)	-	9.1 (0.1)	-	9.0 (0.4)	-	9.2 (0.2)	9.4 (0.2)
Seawater at 60 °C	8.8 (0.3)	-	9.0 (0.2)	-	9.1 (0.3)	9.2 (0.3)	-	-

Average value (standard deviation).

On the other hand, Table 6 shows that the highest increase was observed for laminates with neat resin. For example, the highest increase of the maximum displacement is observed for laminates immersed into seawater at 60 °C (30.3% for neat laminates and 3.4% for laminates with cork), followed by immersion into NaOH (27.3% for neat laminates and 14.8% for laminates with cork) and HCl (24.2% for neat laminates and 8.0% for laminates with cork). This agrees with the studies developed by Amaro et al. [24,44], where the effect of the NaOH solution, compared to the HCl solution, promotes the highest maximum displacement. It is also noted that the hydrochloric acid promotes worse results compared to sulfuric acid.

Table 6. Difference in the maximum displacement obtained for 30 days of immersion into different harsh solutions compared to non-immersed specimens.

Solutions	Matrix with Cork Powder (%)	Neat Resin (%)
Diesel	9.1	18.2
Sulphuric acid (H ₂ SO ₄)	4.5	21.2
Hydrochloric acid (HCl)	8.0	24.2
Sodium hydroxide (NaOH)	14.8	27.3
Distilled water	13.6	19.7
Sea water at room temperature	2.3	16.7
Sea water at 60 °C	3.4	30.3

Similar to what was previously reported, the exposure time proved to be determinant in the degradation of mechanical properties, and, in this case, it promoted higher values of maximum displacement. This parameter is crucial to favour the penetration and reaction that occurs between solutions and composite constituents, especially in terms of matrix cracking and degradation of the fibre–matrix interface strength [26,46,47]. However, for laminates with matrix filled by cork powder, and when subjected to compression during

the impact, the cell walls bend and buckle, which also explains the higher values observed for these laminates.

Finally, based on the previous discussion, and as will be expected, the restored energy decreases as shown in Figure 6. Each result was divided by the respective value obtained with the control specimens (not immersed) and Table 7 summarizes all the average values found and respective standard deviations.

Table 7. Restored energy obtained for the different harsh solutions and immersion time (values in %).

Immersion Solution	Immersion Time (Days)							
	0	10	15	20	30	45	60	90
Carbon Laminates								
Diesel	21.3 (1.16)	-	19.7 (0.68)	-	18.6 (1.32)	18.1 (0.49)	-	-
H ₂ SO ₄	21.3 (1.16)	20.2 (1.26)	-	18.1 (1.45)	17.3 (1.36)	-	-	-
HCl	21.3 (1.16)	18.3 (1.23)	-	16.7 (1.17)	16.3 (1.40)	-	-	-
NaOH	21.3 (1.16)	17.7 (1.09)	-	15.8 (1.15)	13.8 (1.30)	-	-	-
Distilled water	21.3 (1.16)	-	18.3 (1.09)	-	16.5 (1.30)	-	15.7 (1.03)	14.9 (1.10)
Seawater	21.3 (1.16)	-	18.6 (1.31)	-	17.0 (1.07)	-	16.2 (1.44)	15.5 (1.32)
Seawater at 60 °C	21.3 (1.16)	-	18.0 (1.49)	-	16.4 (1.72)	15.8 (1.68)	-	-
Carbon Laminates with Cork								
Diesel	16.6 (0.72)	-	15.7 (0.41)	-	15.1 (0.63)	14.7 (0.56)	-	-
H ₂ SO ₄	16.6 (0.72)	16.0 (1.11)	-	15.5 (1.35)	15.4 (0.72)	-	-	-
HCl	16.6 (0.72)	15.9 (0.87)	-	15.3 (0.46)	14.5 (0.71)	-	-	-
NaOH	16.6 (0.72)	14.4 (1.09)	-	13.2 (1.16)	11.9 (1.30)	-	-	-
Distilled water	16.6 (0.72)	-	14.2 (1.28)	-	13.4 (1.00)	-	12.9 (1.40)	12.1 (1.31)
Seawater	16.6 (0.72)	-	14.4 (1.07)	-	13.6 (1.51)	-	13.1 (1.39)	12.8 (1.26)
Seawater at 60 °C	16.6 (0.72)	-	14.2 (1.21)	-	13.1 (1.42)	12.6 (1.33)	-	-

Average value (standard deviation).

Table 8 shows the effect of the different solutions, comparing the restored energy obtained after 30 days of exposure with those found for the control samples.

Table 8. Difference between the restored energy obtained after 30 days of immersion into different harsh solutions compared to that obtained with non-immersed specimens.

Solutions	Matrix with Cork Powder (%)	Neat Resin (%)
Diesel	9.0	12.7
Sulphuric acid (H ₂ SO ₄)	7.2	18.8
Hydrochloric acid (HCl)	12.7	23.5
Sodium hydroxide (NaOH)	28.3	35.2
Distilled water	19.3	22.5
Sea water at room temperature	18.1	20.2
Sea water at 60 °C	21.1	23.0

Both laminates are very sensitive to exposure to all studied solutions, but as noted for maximum impact load, laminates with neat resin show higher sensitivity. As previously reported, cork is impermeable to liquids and practically impermeable to gases [23,57,58] and consequently, when introduced into the matrix, it acts as an impermeable agent and retards the diffusion that occurs between the cork–matrix interface due to a longer and tortuous path caused by the presence of cork particles. This, for example, explains the lower values observed in Table 8 for laminates with matrix filled with cork. Similar benefits

were found by Reis et al. [19] when an epoxy matrix was filled with cork powder. On the other hand, lower values of restored energy are the consequence of higher damaged area. According to Amaro et al. [24], there is an interaction between matrix cracking caused by exposure to hostile solutions and the delamination of composite laminates when subjected to low velocity impact. These cracks interact with delaminations and, in this context, contribute to increase the damaged area (consequently the elastic energy decreases) [24]. Resulting from microcracks produced in the matrix by hostile solutions, Amaro et al. [24] observed an increase in roughness on laminate surfaces with exposure time, which presented the highest values for samples immersed into NaOH solutions. Simultaneously, the ultramicroindentation tests performed by these authors showed a decrease in the mechanical properties of the matrix [24]. For example, the decrease in the matrix Young's modulus promoted by corrosive solutions associated with the degradation of the fibre–matrix interface explained the lower bending properties obtained by the authors. In other studies, an important effect of the defect size on the maximum load was observed (which decreases with increasing the defect size) and the plate deflection is shown to be proportional to the bending stiffness [59,60]. Therefore, according to Stamenovic et al. [47], both the degradation of the matrix–fibre interface and the mechanical properties of the matrix considerably affect the load carrying capacity of the material.

Finally, according to David-West et al. [61], the impact bending stiffness is an important property to assess the damage resistance of a composite and, particularly, delaminations. The slope of the upward section of the load-displacement curve is defined as the impact bending stiffness, however, more details to determine this property can be found in [61]. For comparison purposes, for example, Table 9 shows the observed decrease in impact bending stiffness after 30 days of immersion into different harsh environments compared to the value obtained for the control samples. It should be noted that the average value of impact bending stiffness for laminates with neat resin is about 408 N/mm, with a standard deviation of 15.3 N/mm, while for laminates incorporating cork powder these values are 246.7 N/mm and 34.3 N/mm, respectively.

Table 9. Decrease of the impact bending stiffness after 30 days of immersion into different harsh solutions compared to the value obtained for non-immersed specimens (values in %).

Solutions	Matrix with Cork Powder	Neat Resin
Diesel	9.0	16.6
Sulphuric acid (H ₂ SO ₄)	10.8	19.7
Hydrochloric acid (HCl)	19.7	25.4
Sodium hydroxide (NaOH)	22.6	28.6
Distilled water	13.8	17.4
Sea water at room temperature	7.5	15.1
Sea water at 60 °C	23.8	31.2

Studies developed by Amaro et al. [24] report that the severity of the damage is directly related to the absorbed energy and inversely related to the impact bending stiffness. Therefore, according to Table 9, it is possible to conclude that, after 30 days of exposure to different hostile solutions, damages with different severity/sizes are expected, but the highest ones are obtained for laminates with neat resin. For example, the highest damages are observed for laminates immersed into seawater at 60 °C, followed by immersion into NaOH and HCl, which is in line with the open literature [24,44]. In this context, hydrochloric acid is expected to cause higher damages than immersion into sulfuric acid.

4. Conclusions

This work studied the low velocity impact response of a carbon fibre/epoxy laminate and a similar architecture with matrix filled with cork powder after immersion into diesel, H₂SO₄, HCl, NaOH, distiller water, seawater and seawater at 60 °C. It was possible to conclude that the impact properties are significantly affected by the solutions, with acid

and seawater solutions being the most aggressive. Moreover, the exposure time proves to be determinant in the mechanical behaviour of the composites, where it was noticed that the impact properties clearly decrease with the increase in exposure time. However, the benefit of cork powder on the perforation threshold was noted, with an increase of around 9% compared to the control samples. After exposure to hostile solutions, cork powder also demonstrates less variation in general compared to the control samples, both in maximum load and restored energy.

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