Accepted Manuscript

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N. Mortas[1], O. Er[2], P.N.B. Reis[3], J.A.M. Ferreira[4]

[1] Depart. Mechatronic Engineering, Bozok University, Yozgat, Turkey

[2] Depart. Electric & Electronic Engineering, Bozok University, Yozgat, Turkey

[3] Depart. Electromechanical Engineering, University of Beira Interior, Covilhã, Portugal;
e-mail: preis@ubi.pt; telephone: +351 275 329 725; fax: +351 275 329 972

[4] CEMUC, Depart. Mechanical Engineering, University of Coimbra, Coimbra, Portugal

Abstract: In recent years, there has been a rapid growth in the use of fibre reinforced composite materials in engineering applications and this phenomenon will be continuing. In this context, composite structures can be exposed to a range of corrosive environments during their in-service life, which causes degradation in terms of material properties. Some works can be found in open literature, but the studies presented are not sufficient to establish a full knowledge about this subject. Therefore, the aim of this work is study the low velocity impact response of Kevlar/epoxy laminates and carbon/epoxy laminates, after immersion into hydrochloric acid (HCl) and sodium hydroxide (NaOH). The aggressive solutions affect significantly the impact strength, but their effects are strongly dependent of the concentration. On the other hand, a significant effect of the temperature can be found, independently of the aggressive solution, on the impact performance and residual bending strength.

Key words: Corrosive solutions; Polymer-matrix composites (PMCs); Impact behaviour; Mechanical testing

1. Introduction

In recent years, there has been a rapid growth in the use of fibre reinforced composite materials in engineering applications and there is a clear indication that this will be continuing. In this context, it is becoming common the components manufactured by composite materials for applications at highly corrosive environments, as an alternative to the stainless or coated steels. However, the studies presented on the open literature are not sufficient to establish a full knowledge about the effect of hostile solutions on the PMC mechanical properties.

Stamenovic *et al* [1] compared, for glass/polyester composites, the effect of alkaline and acid solutions on their tensile properties, concluding that the alkaline solution decreases the ultimate tensile strength and modulus. However, the acid solution increases the tensile properties and this phenomenon was more relevant when the pH value decreases. On the other hand, for both solutions, Stamenovic *et al* [1] concluded that the changes observed on the tensile properties are proportional with the number of days into immersion. The period of exposure was studied by Mahmoud *et al* [2] and significant influence of this parameter on the flexural strength, hardness and Charpy impact resistance was found by these authors. The flexural strength of the glass/polyester composite is insensitive until 30 days of immersion in HCl and, after this period, a decrease around 10% was observed. In terms of hardness, they showed that the Barcol hardness drops around 15% after 90 days of exposure. A slight decrease of the Charpy impact resistance (about 5%) was observed during the first 60 days of immersion but, between 60 and 90 days, a significant drop of 10% was found. Composites with different resins were immersed into two aqueous acid solutions and, according with Banna *et al* [3], their final mechanical properties showed to be very dependents of the resins used. Amaro *et al* [4] developed a systematic study about the effects of alkaline (NaOH) and acid (HCl) solutions on glass/epoxy composites. These authors found that, independently of

the solution, the flexural strength and the flexural modulus decreases with the exposure time. On the other hand, the alkaline solution (NaOH) showed to be more aggressive than the acid solution (HCl), promoting the lowest flexural properties. The flexural strength, after 36 days of exposure, decreased around 22% for the NaOH solution and 16.2% for the HCl solution. Similar tendency was observed for the flexural modulus with values around 26.9% and 22.3%, respectively. Complementary tests were carried out and the ultramicroindentation showed a decreasing of the matrix mechanical properties. The roughness increased with the exposure time and was higher for the samples immersed in alkaline solutions. The effect of different acid solutions was studied, in a similar work, by the same authors [5]. In this case, a hydrochloric acid (HCl) and a sulphuric acid (H_2SO_4) were used and the flexural properties were affected significantly by these solutions. For example, after 36 days of exposition, the flexural strength decreased around 16.2% for the HCl solution and 11.6% for the H_2SO_4 solution. Similar tendency was observed for the flexural modulus with values around 22.4% and 17.6%, respectively. It is evident that the hydrochloric acid was responsible by the worst results.

It is well known that the composites are very strong in the plane, but with very low impact performance through the thickness. Various types of damages can occur, which are very dangerous because they are not easily detected visually [6, 7] and they can affect significantly the residual properties and structural integrity of those materials [8-12]. However, low velocity impact associated with highly corrosive environments, is reported in bibliography only by Amaro *et al* [4, 5] on glass/epoxy composites. According with these authors, the resistance of the glass/epoxy laminates to repeated low velocity impacts is very dependent of the corrosive environment and the exposure time. The alkaline solution shows to be more aggressive than the acid solution, promoting the lowest impact resistance. On the

other hand, comparing the hydrochloric acid (HCl) with the sulphuric acid (H_2SO_4), the first one is responsible by the lowest impact performance.

Therefore, the aim of this work is study the low velocity impact response of Kevlar/epoxy laminates and carbon/epoxy laminates, after immersion into hydrochloric acid (HCl) and sodium hydroxide (NaOH). These solutions were selected because, according with Amaro *et al* [4, 5], they promoted the lowest impact performance. The effect of concentration and temperature of the solutions on the impact strength was analysed and the residual bending strength was compared with the control samples. In fact, composites reinforced by carbon or Kevlar fibres have several application fields, but the open literature does not report any study about the effect of corrosive environments on their impact strength.

2. Material and experimental procedure

Two different composite laminates were manufactured by hand lay-up and the overall dimensions of the plates obtained were 330x330x3 [mm]. An Ampreg 22 epoxy resin and an Ampreg 22 hardener standard, supplied by Gurit, was used with nine layers, all in the same direction, of woven bi-directional Kevlar 170-1000P (170 g/m^2) and woven bi-directional carbon 195-1000 (195 g/m^2) to produce each laminate. The system was placed inside a vacuum bag and a load of 2.5 kN was applied for 48 hours in order to maintain a constant fibre volume fraction and an uniform laminate thickness. During the first 10 hours 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 ºC for 48 hours.

The samples used in the experiments were cut from these plates to square specimens with 100 mm side and 3 mm thickness (100x100x3 mm). Afterwards, they were completely submerged into hydrochloric acid (HCl) and sodium hydroxide (NaOH), at room temperature,

during 20 days. Both solutions presented a concentration of 5, 15, 25 and 35% in weight (wt.%). The effect of the temperature was also analysed and, for this study, the specimens were submerged, during 20 days, at 40ºC and 60ºC into both solutions with a concentration of 15% in weight (wt.%). Finally, the samples were washed with clean water and dried at room temperature.

Low-velocity impact tests were performed using a drop weight-testing machine IMATEK-IM10. More details of the impact machine can be found in [13]. 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 75x75 mm and the impactor stroke at the centre of the samples obtained by centrally supporting the 100x100 mm specimens. The impact energy used was 10 J which corresponds to an impact velocity of 2.6 ms^{-1}.

After impact tests, the specimens were submitted to three point bending (3PB) tests. The specimens were tested with a span of 50 mm using a Shimadzu AG-10 universal testing machine equipped with a 5kN load cell and TRAPEZIUM software at a displacement rate of 5 mm/min.

3. Results and discussion

Impact tests were carried out on different materials after exposure to the HCl and NaOH solutions with several concentrations. Figure 1 and 2 shows the force-time and force-displacement curves, respectively, for both composite laminates (Kevlar/epoxy and carbon/epoxy laminates). These diagrams represent a typical behaviour occurred for all specimens and agree with the bibliography [14-19]. The curves contain oscillations that result from the elastic wave and are created by the vibrations of the samples [18-20]. It depends on

the stiffness and the mass of the specimen and impactor being excited by the rapid variation of the cinematic magnitudes at the collision moment [18, 19, 21].

It is possible to observe that the force increases up to a maximum value, P_{max}, followed by a drop after the peak load. The impact energy was not high enough to infiltrate full penetration, because the impactor sticks into specimens and rebound always. Therefore, non-perforating impact occurred for all laminates. However, when the concentration of the solutions increases there are higher displacements and contact time, which means that major damage occurs.

Table 1 shows the effect of the aggressive environments on maximum load (P_{max}), maximum displacement and contact time in terms of average values and respective standard deviation. These parameters are significantly affected by the corrosive solutions. In terms of Kevlar/epoxy laminates, and comparing the control samples with the samples immersed into 5 wt% of NaOH, the load decreases around 16.3%, while the displacement and contact time increases about 0.4% and 1%, respectively. However, these values are dramatically affected when the samples are immersed into 35 wt% of NaOH, with differences of 26.8%, 7.3% and 4.1%, respectively. Similar tendency can be found for the samples immersed into HCl solution, where, for the same comparison (control samples versus samples immersed into 35 wt% of HCl), the load decreases around 18.8% but the displacement and contact time increases about 9% and 11.5%, respectively. The results described before can be replicated for the carbon/epoxy laminates. In this case, comparing with the control samples, the maximum load observed for the samples immersed into 35 wt% of NaOH decreases around 36.7%, while the displacement and the contact time increases about 16.6% and 20.4%, respectively. On the other hand, the same comparison for 35 wt% of HCl presents values about 33.3%, 19.7% and 26.1%, respectively. Therefore, it is possible to conclude that the results agree with the study developed by Amaro *et al* [4], where the effect of NaOH solution,

compared to the HCl solution, promotes the lowest maximum load, displacement and contact time.

Figure 3 shows the effect of the aggressive solutions, and respective concentration, on elastic recovery (elastic recuperation). For this purpose, the elastic energy was calculated as the difference between the absorbed energy and the energy at peak load [18, 19]. For both laminates, it is possible to observe that the corrosive environment decreases the elastic energy and its effect is highly dependent with the concentration of the solution. The alkaline solution promotes the lowest elastic energies, because these solutions are highly corrosives [1, 4, 22]. For example, in terms of Kevlar/epoxy laminates, a decrease about 20.8% occurs for 35 wt% of HCl and around 25.9% for 35 wt% of NaOH solution, when compared with the control samples. In terms of carbon/epoxy laminates, these values are around 12.6% and 18.3%, respectively.

All samples were inspected by C-Scan technique in a square area of 50x40 mm, containing the impact zone. For the ultrasonic analysis a 25 MHz broadband immersion piezocomposite transducer was used in the pulse-echo mode. More details about the technique and equipment can be found on [7]. The samples were inspected by the opposite side of the impact. Figure 4 shows the typical images obtained for some laminates, however, they are representative of the other ones. It is noted that the damage can be detected without ambiguity, where the blue colour represents the main damage [7]. The blue area was measured by Image-Pro Plus software. As expected, the damaged area increases with the decreasing of elastic energy and the highest damages occur for samples exposed to the sodium hydroxide (NaOH). These results agree with the studies developed by Amaro *et al* [4].

For all conditions analysed, the damaged area was higher for Kevlar/epoxy laminates. In fact, the aramid fibres do not fail by brittle cracking, as do glass or carbon fibres, but they fail essentially by a series of small fibril failures [18]. According with Aktas *et al* [23] the

main energy absorption mechanism for carbon-fibre reinforced composite is fibre breakage mode, while for Kevlar composites it is essentially by delaminations. On the other hand, for a woven laminate, delamination starts at the centre of impact and propagates to the directions of warp and fills fibres [18]. Additionally, aramid fibres are strongly hygroscopic and often with values higher than the matrices. A maximum moisture content of 6% is reported for Kevlar-49 at room temperature 96% RH [24]. Studies developed by Reis *et al* [25] shown that, after 100 days, the system SR 1500 epoxy resin and SD 2503 hardener presents around 0.9% of moisture content, but when the Kevlar fibres are added to the resin this value increases 2.5 times. In this context, the combined action of water and corrosive fluids leads to matrix expansion, with consequent occurrence of micro-cracks and/or development of microstresses in the composites [26-29]. These micro-cracks, according with some researchers, play an important role at the onset of delaminations, with consequent increase of the damaged area [4, 30]. Mahmoud *et al* [2], for example, associated the drop of mechanical properties with the absorption, penetration and reaction that occur between the solutions and the composite constituents (matrix and fibres). For Hammami and Al-Ghilani [26] the degradation takes place via two stages. In the first stage resin is attacked under the combined action of water diffusion and the presence of H^+. In the second stage the fibre itself is attacked and cracks appear on the fibre surface. Therefore, the poor fibre matrix connection promotes the weakness of the load carrying capacity of the laminate [1].

After impact, residual bending strength was obtained for each condition studied as shows Figure 5. The term "residual bending strength" corresponds to the maximum bending load obtained from the load-displacement curves collected of the three point bending (3PB) tests. In terms of carbon/epoxy laminates, for example, comparing the control samples with samples immersed into 35 wt% of NaOH, the residual bending strength decreases around 9.9%, while for 35 wt% of HCl this value is about 6.5%. However, for the same comparison,

the values obtained for Kevlar/epoxy laminates drops significantly to 34.0% and 21.6%, respectively. As expected, for both laminates, the alkaline solution promotes the lowest residual bending strength. On the other hand, the lowest residual bending strength occurred for Kevlar/epoxy laminates, as consequence of the highest damaged areas (see Figure 4).

According with Amaro *et al* [4], the decreasing observed on the flexural properties can be explained, also, by the matrix/fibre interface degradation and by the Young's modulus of the matrix. This property is significantly affected when the polymeric matrixes are exposed to the aggressive solutions [4]. To identify the influence of the Young's modulus on the residual bending strength, and on the impact parameters, this property was evaluated by ultramicroindentation using Fisherscope H100 equipment and a load of 500 mN. The testes were performed according with Antunes *et al* [31] and Figure 6 shows the results obtained.

The results agree with the study developed by Amaro *et al* [4]. It is possible to observe that the Young's modulus decreases, independently of the solutions, but the decreasing observed is more expressive in terms of solutions' concentration. On the other hand, and comparing both materials studied, the Kevlar/epoxy laminates are responsible by the lowest Young's modulus. This phenomenon can be explained by the strong hygroscopic behaviour of the aramid fibres, which promotes higher wetting of the matrix.

The effect of the temperature was also analysed and Table 2 presents the results obtained. The specimens were submerged, during 20 days, at room temperature (22ºC), 40ºC and 60ºC into both solutions with a concentration of 15wt%.

It is possible to observe that, independently of the solution, higher temperatures induce a decreasing of the maximum loads, but the opposite can be found for the displacement and the contact time. In terms of Kevlar/epoxy laminates, for example, the sodium hydroxide (NaOH) promotes a decreasing of the maximum loads around 9.3%, while the displacement and the contact time increases about 10.1% and 2.3%, respectively, when the room

temperature is compared with the temperature of 60ºC. Similar tendency can be found for the samples immersed into hydrochloric acid (HCl) with values around 8.9%, 10.6% and 6.7%, respectively. On the other hand, for the carbon/epoxy laminates, the alkaline solution (NaOH) promotes a decreasing of the maximum loads around 9.9%, while the displacement and the contact time increases about 11.5% and 6.3%, respectively. For the acid solution (HCl), these values are around 8.3%, 12.1% and 6%, respectively.

Figure 7 shows the effect of the temperature on the elastic recovery (elastic recuperation). Independently of the temperature, the lowest elastic recovery occurs for samples exposed to the sodium hydroxide (NaOH). The temperature promotes a significant drop of the elastic energy and, for the Kevlar/epoxy laminates, a decreasing of 21.1% can be found for the samples immersed into 15 wt% of HCl at 60ºC and around 24.9% for 15 wt% of NaOH at 60ºC, when compared with the same solutions at room temperature. For the carbon/epoxy laminates these values are around 16.4% and 19.6%, respectively. One more time, in terms of elastic recovery, the Kevlar/epoxy laminates are responsible by the highest drop, which is a consequence of the highest damaged areas. Figure 8 shows the residual bending strength and can prove the last sentence.

A significant effect of the temperature can be found, independently of the aggressive solution, on the residual bending strength. However, as expected, the alkaline solution promotes the lowest residual bending strength. In this context, and comparing the results obtained at 60ºC with the other ones at room temperature, the residual bending strength decreases around 21.7% for the carbon/epoxy laminates and about 25.1% for the Kevlar/epoxy laminates. In terms of acid solution (HCl) these values are 14.9% and 19.3%, respectively. The aggressive solutions are absorbed mainly by the resins and the temperature affects its absorption [32]. As diffusion is a thermally activated process, an increase in temperature accelerates short-term diffusion and increases the diffusion coefficient On the

other hand, different coefficients of thermal expansion between the fibre and the polymeric matrix develops residual stresses at the interface, which can result in microvoids or cracks [33]. In this context, the aggressive solution creates a hydrostatic pressure at the crack tips and hastens the crack propagation and damage in the matrix [34].

4. Conclusions

This work studied the low velocity impact response of different laminates after immersion in hydrochloric acid (HCl) and sodium hydroxide (NaOH). Special focus was taken in terms of concentration and temperature of these aggressive solutions.

For both laminates, it was possible to conclude that the corrosive environments affect significantly the impact response, but their effects are strongly dependent of the solutions' concentration. The alkaline solution shows to be more aggressive than the acid solution, promoting the lowest impact performance and residual bending strength. For Kevlar/epoxy laminates, the elastic recovery decreased about 20.8% for 35 wt% of HCl and around 25.9% for 35 wt% of NaOH solution, when compared with the control samples. Regarding to carbon/epoxy laminates, these values were around 12.6% and 18.3%, respectively. Comparing the control samples with samples immersed into 35 wt% of NaOH, the residual bending strength decreased around 9.9%, while for 35 wt% of HCl this value was about 6.5%. On the other hand, the values obtained for Kevlar/epoxy laminates drops significantly to 34.0% and 21.6%, respectively. Consequently, for all conditions analysed, the damaged area was higher for Kevlar/epoxy laminates.

Independently of the solution, higher temperatures induce a decreasing of the maximum loads, but the opposite occurs for the displacement and the contact time. The temperature promotes a significant drop of the elastic energy for both laminates. For
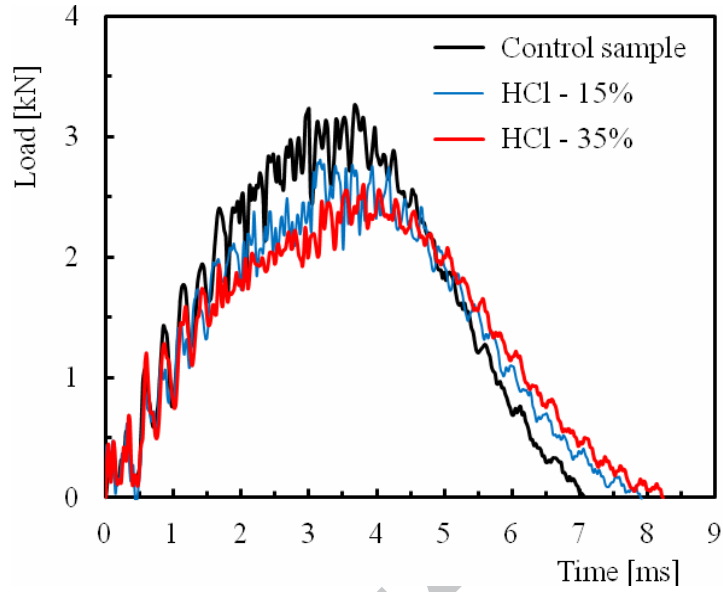
Kevlar/epoxy laminates, a decreasing of 21.1% was found for the samples immersed into 15 wt% of HCl at 60ºC and around 24.9% for 15 wt% of NaOH at 60ºC, when compared with the same solutions at room temperature. Relatively to the carbon/epoxy laminates these values were around 16.4% and 19.6%, respectively. A significant effect of the temperature on the residual bending strength was also found, independently of the aggressive solution. For NaOH a decreasing around 21.7% for the carbon/epoxy laminates and about 25.1% for the Kevlar/epoxy laminates was obtained. In terms of acid solution (HCl) these values were 14.9% and 19.3%, respectively.
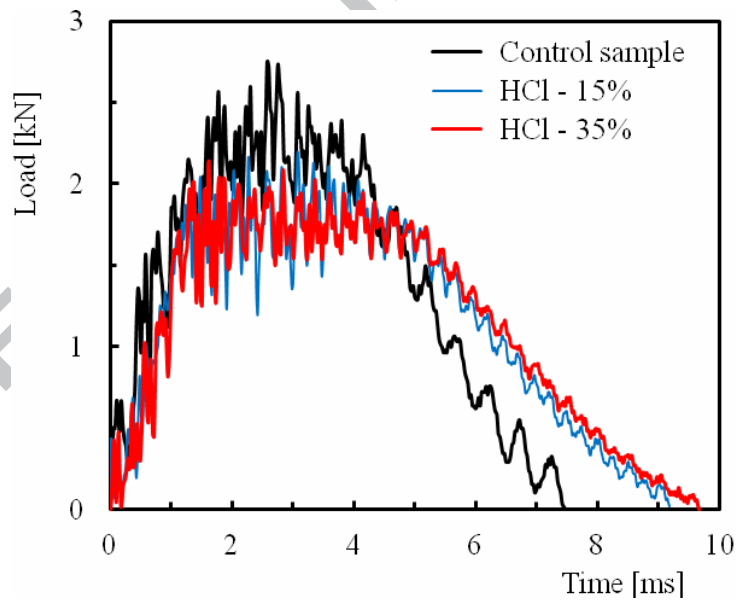
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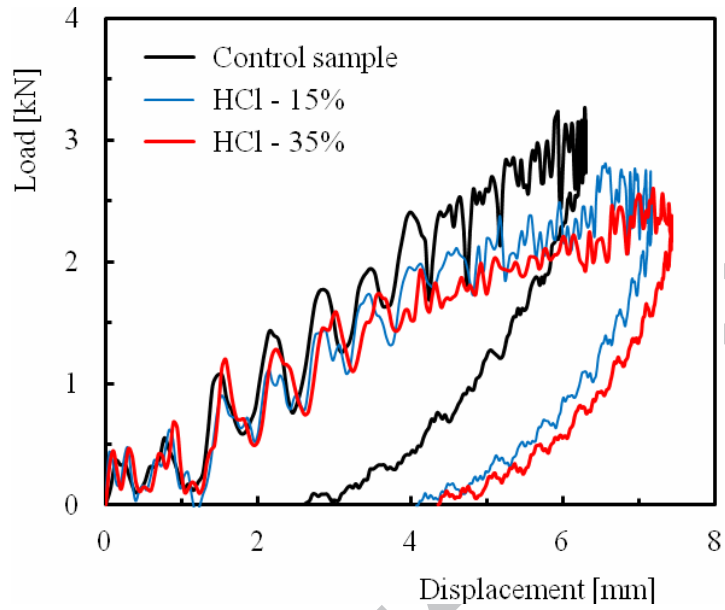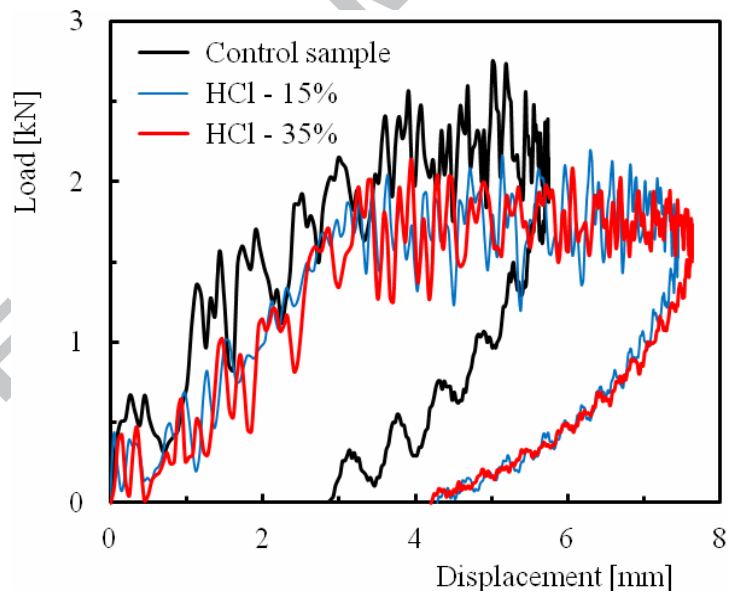
a)

b)

Figure 1 – Typical diagrams load versus time obtained, at room temperature, for:

a) Kevlar/epoxy laminates; b) Carbon/epoxy laminates.

a)

b)

Figure 2 – Typical diagrams load versus displacement obtained, at room temperature, for: a) Kevlar/epoxy laminates; b) Carbon/epoxy laminates.
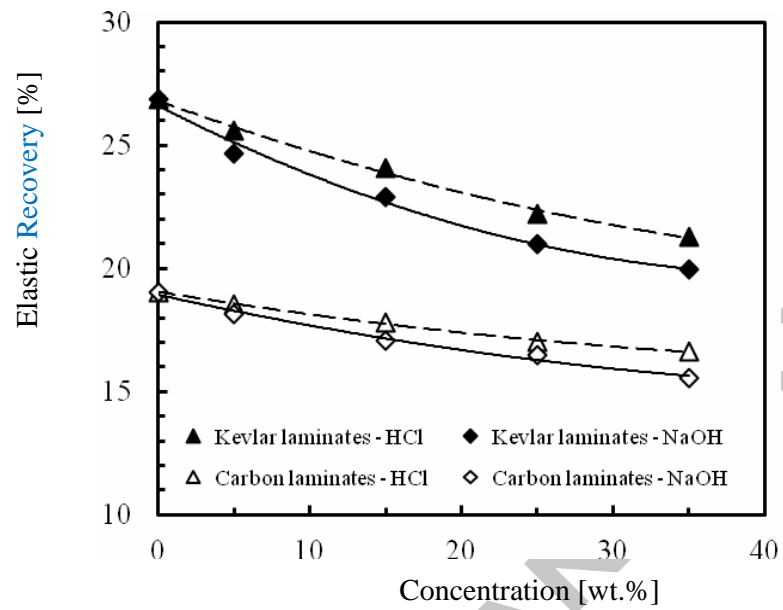
Figure 3 – Elastic recovery, for the different laminates, as a function of the corrosive solution and respective concentration.
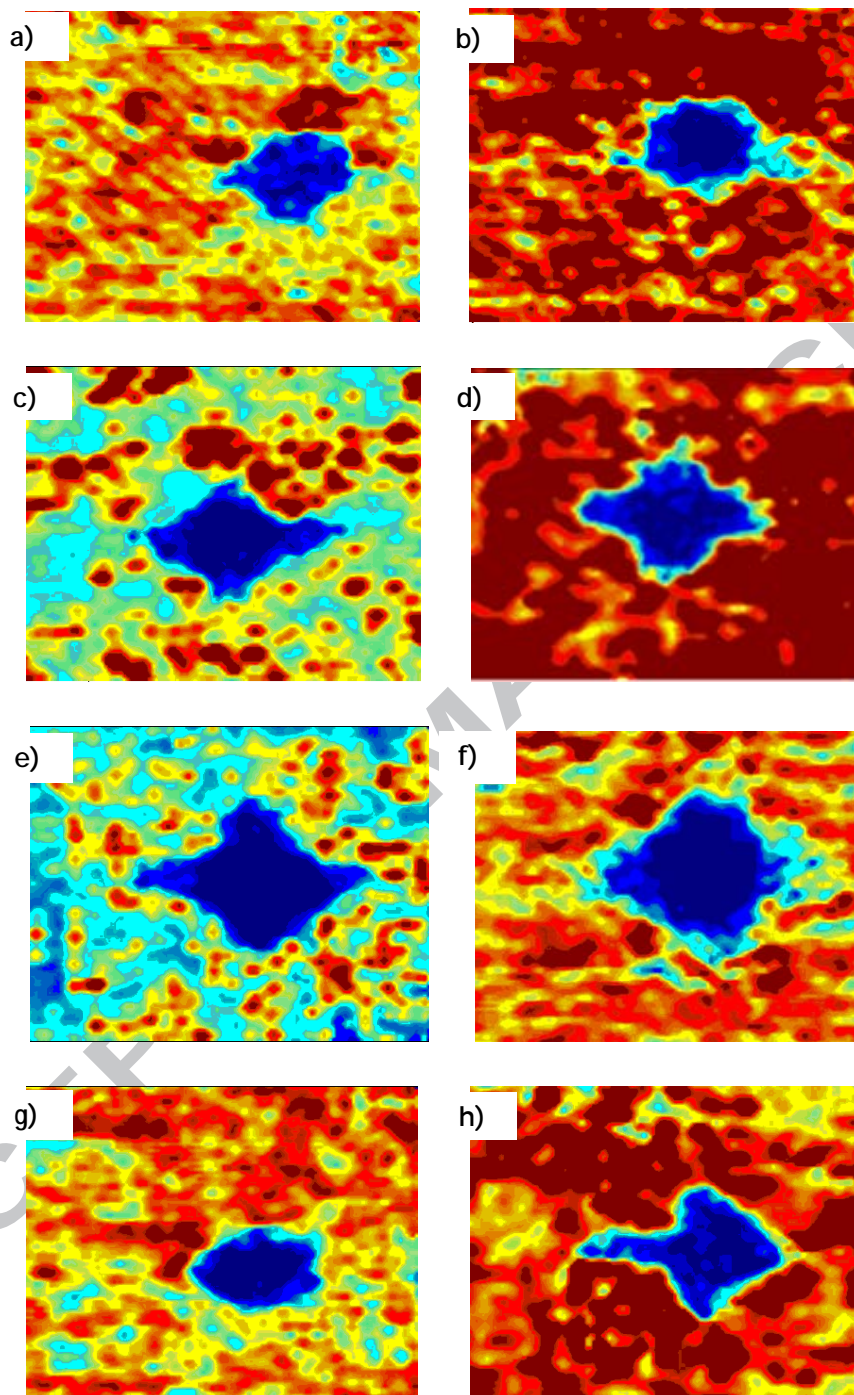
Figure 4 – Typical images of the damages for: a) Carbon laminates (control samples);
b) Kevlar laminates (control samples); c) Carbon laminates immersed into 15% NaOH; d)
kevlar laminates immersed into 15% NaOH; e) Carbon laminates immersed into 35% NaOH;

f) Kevlar laminates immersed into 35% NaOH; g) Carbon laminates immersed into 15% HCl;
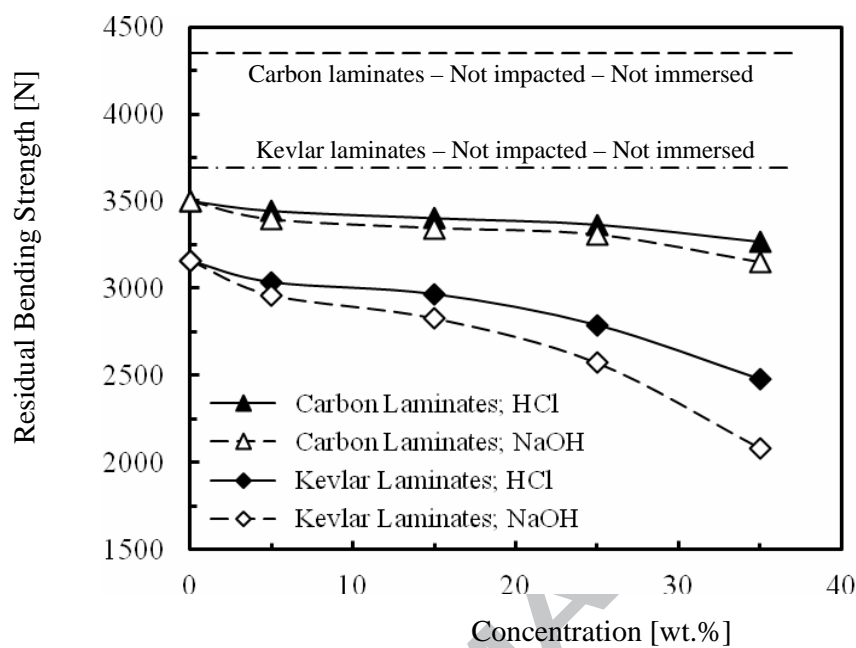
h) Kevlar laminates immersed into 15% HCl.

Figure 5 – Residual bending strength for laminates subjected to different solutions and concentrations.
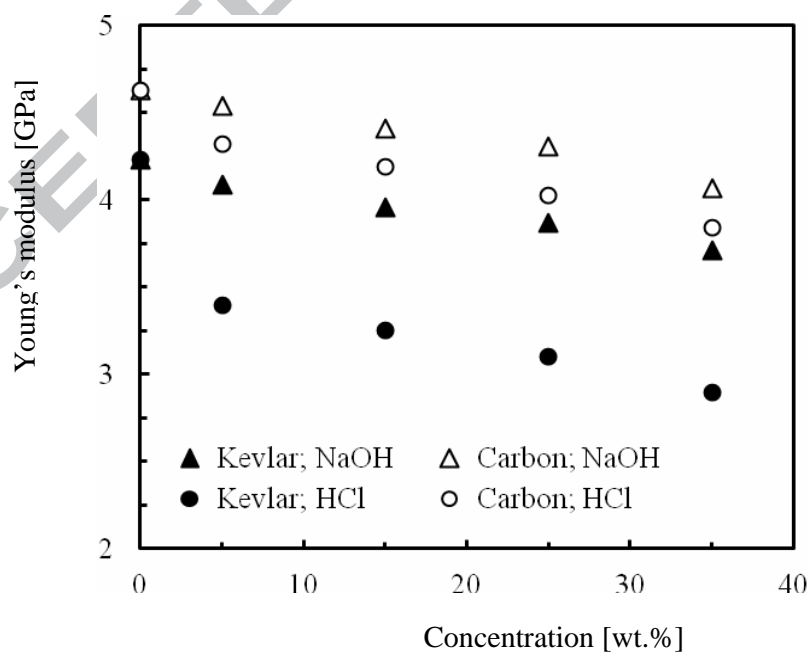
Figure 6 – Young's modulus of the resin after exposition at different solutions and concentrations.
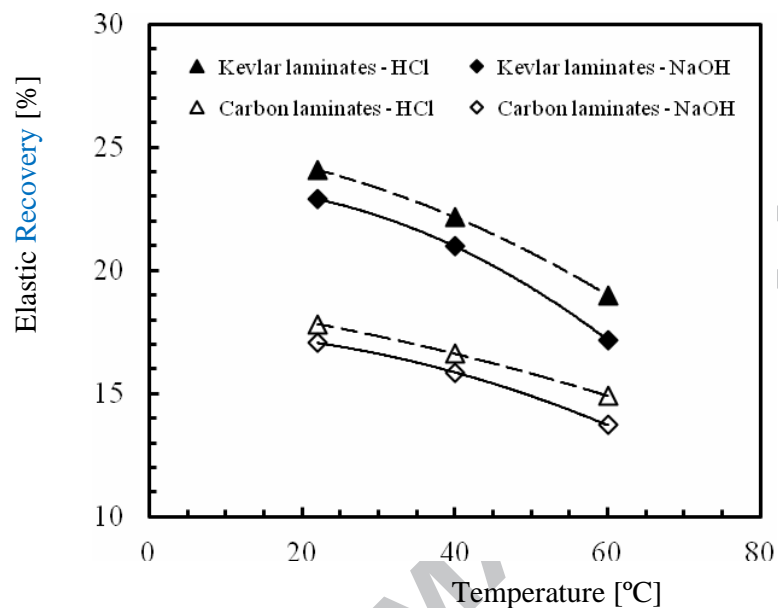
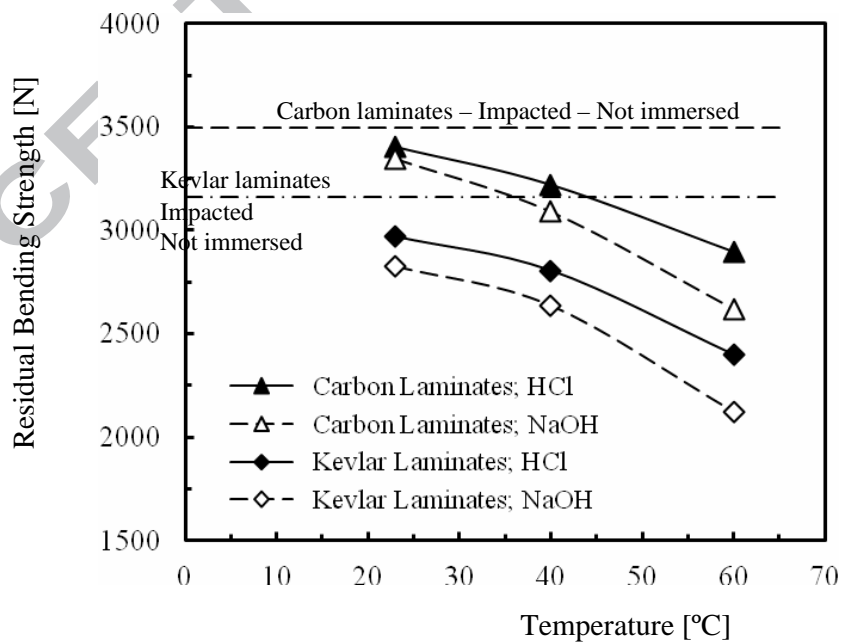Figure 7 – Elastic recovery, for the different laminates and solutions, as a function of the temperature.

Figure 8 – Residual bending strength for laminates subjected to different solutions and temperature.

Table 1 - Effect of corrosive solutions on the impact parameters.

Laminates	Solution	Concentration [wt.%]	Maximum Load		Maximum Displacement		Contact time	
			Average [kN]	St Dev [kN]	Average [mm]	St Dev [mm]	Average [ms]	St Dev [ms]
Kevlar	(*)	-	3.593	0.03	6.75	0.24	7.32	0.14
	NaOH	5	3.008	0.1	6.78	0.11	7.39	0.31
		15	2.896	0.12	6.83	0.24	7.47	0.39
		25	2.887	0.11	7.01	0.19	7.51	0.52
		35	2.631	0.14	7.24	0.16	7.62	0.15
	HCl	5	3.311	0.09	6.94	0.26	7.38	0.27
		15	3.142	0.1	7.01	0.27	7.77	0.13
		25	3.091	0.06	7.11	0.16	7.85	0.16
		35	2.919	0.04	7.36	0.18	8.16	0.28
Carbon	(*)	-	3.163	0.02	6.61	0.07	8.04	0.02
	NaOH	5	2.435	0.07	7.27	0.16	9.39	0.41
		15	2.221	0.13	7.33	0.12	9.47	0.12
		25	2.161	0.11	7.49	0.11	9.59	0.26
		35	2.002	0.11	7.71	0.17	9.68	0.34
	HCl	5	2.646	0.05	7.50	0.12	9.58	0.35
		15	2.346	0.11	7.52	0.08	9.65	0.18
		25	2.295	0.09	7.66	0.09	9.71	0.28
		35	2.111	0.03	7.91	0.11	10.14	0.11

(*) Control samples

Table 2 - Effect of temperature for the different solutions on the impact parameters.

Laminates	Solution	Temperature [°C]	Maximum Load		Maximum Displacement		Contact time	
			Average [kN]	St Dev [kN]	Average [mm]	St Dev [mm]	Average [ms]	St Dev [ms]
Kevlar	NaOH	22	2.896	0.12	6.83	0.24	7.47	0.39
		40	2.798	0.18	7.22	0.23	7.56	0.26
		60	2.627	0.19	7.52	0.18	7.64	0.21
	HCl	22	3.142	0.1	7.01	0.27	7.77	0.13
		40	3.017	0.11	7.53	0.19	7.85	0.15
		60	2.863	0.16	7.75	0.21	8.29	0.11
Carbon	NaOH	22	2.221	0.13	7.33	0.12	9.47	0.12
		40	2.166	0.12	7.89	0.13	9.73	0.19
		60	2.001	0.18	8.17	0.14	10.07	0.18
	HCl	22	2.346	0.11	7.52	0.08	9.65	0.18
		40	2.314	0.13	8.17	0.13	10.11	0.17
		60	2.151	0.17	8.43	0.26	10.23	0.15