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## Effect of moisture on the adhesion of CFRP-to-steel bonded joints using peel tests

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### Abstract

In this work, an experimental study has been carried out to evaluate the effect of salt water condition on the long-term adhesion of composite-to-metal bonded joints using peel tests. A new test configuration is tested via fully and partially immersed specimens with 500 mm length. Fracture surfaces of non-aged samples exhibited a cohesive failure within the adhesive layer, which indicates a good adhesion of the joint. Results revealed a significant difference in the interface adhesion between aged and non-aged condition after 150 days of immersion. Partially immersed specimens allow to evaluate the adhesion performance of the joint under dry and wet condition in single specimen using a simple peel test.

### Keywords

Adhesively bonded joints; peel tests; moisture; CFRP; salt water

## 1 Introduction

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Nowadays, composite materials are widely used for repair of metallic components in offshore, onshore, oil and gas industries. Adhesive bonding is the most efficient joining technology in terms of weight and performance to join these two materials. Therefore, composite-to-metal bonded joints are nowadays a common practice in such maritime industries. The effect of multi-material bonding process on joint strength has been investigated by many researchers and found a positive influence on the performance of bonded structure [1-3]. However, limited research is found on the performance of such hybrid joints under the harsh maritime environments. One of the most critical aspects for the long term durability of bonded joints is the adhesion at the interfaces. With the increasing use of composite-to-metal bonded joints, attention should be paid on the adhesion quality in the presence of environmental effect in order to assure long term durability of the multimaterial bonded joints.

During service, metal-structure can be exposed to extreme environmental condition, which impact their performance. This can be especially severe in offshore and onshore unit, where the metal pipe is always in contact with the salt water [4-5]. Several works were carried out in order to assess the influence of environmental parameters on the durability of bonded structures [5-9]. There are many studies carried out on the ageing effect, using the composite-composite bonded joints (mode I, mode II and mixed mode) and only very few carried out with the composite-metal bonded joints [10-14]. Most of the results found a detrimental effect of moisture on the performance of the bonded joints and show that moisture is one of the most problematic factors, when it comes to the durability of adhesive joints with FRP and metallic adherents [15-18]. Salt water immersion is one of the worst ageing conditions to affect the joint performance, because

1 not only the adhesion is affected by the moisture but the metal gets corrode easily. So, it is  
2 of prime importance to assess the interface adhesion between the metal-composite joint in  
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4 the presence of different environmental conditions.  
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7 Interface adhesion is one of the most important parameters for assuring the integrity of  
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9 composite-metal bonded joints. Peel test is a suitable destructive test method to examine  
10 the interface adhesion properties, while others test methods such as Single Leg Bending  
11 (SLB) and Double Lap Joint (DLJ) examine the mechanical properties of the joint rather  
12 than the interface adhesion. The floating roller peel test is one of the most commonly used  
13 in metal bonding because it is a simple, fast and reliable test method to assess adhesion.  
14 [19-23]. For metal substrates, the peel test is well developed and documented.  
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16 Nevertheless, few research has been done on this type of test when applied to dissimilar  
17 substrate joints [24, 25]. Some researchers [26-28] performed peel tests on composite-  
18 metal bonded joints and they successfully assessed the interface adhesion between  
19 composite-metal joints. A simple and straightforward test coupon is needed to assess the  
20 adhesion of metal composite bonding and evaluates the adhesion interface of interest.  
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36 In this study, floating roller peel tests are used to assess the adhesion properties of the  
37 composite-metal bonded joints. A new method of analysis is proposed in which long peel  
38 specimens are used to evaluate the effect of ageing and non ageing in the same specimen.  
39 This work aims to evaluate the effect of salt water on the adhesion of Carbon Fibre  
40 Reinforced Polymer (CFRP)-to-carbon steel bonded joints using peel tests and to evaluate  
41 the new method of analysis proposed.  
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3 **2 EXPERIMENTAL METHODS**  
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7 **2.1 Materials**  
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10 Normally, steel pipes are used to carry fluid over a long distance because of its good  
11 mechanical properties [29-30]. To assess the interface adhesion between the existing  
12 steel structure and the CFRP repair, peel test specimens were manufactured with carbon  
13 steel and CFRP substrates. The peel specimens consisted of a CFRP rigid adherend  
14 bonded to a carbon steel flexible adherend. A NVT201E® epoxy resin (Novatec Ltd., Rio  
15 de Janeiro, Brazil) was used as the adhesive to join the substrates. Carbon fiber fabrics  
16 with a density of 430 g/m<sup>2</sup> were selected to make the composite adherend. Each fabric is  
17 composed of two unidirectional laminas of continuous fibers oriented perpendicula  
18 [0/90]. PIPEFIX® epoxy resin (Novatec Ltd., Rio de Janeiro, Brazil) was selected for  
19 impregnation of the composite laminate. A 0.4 mm thick carbon steel ASTM A36 plate  
20 was used as a flexible adherend.  
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38 **2.2 Sample Preparation**  
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41 The composite-to-metal bonded joint test specimens were manufactured by the same  
42 process used in composite patch repairs of oil and gas production platforms [29-30]. The  
43 carbon steel plate was blasted with G-40 steel shot and then degreased using acetone. The  
44 final surface roughness measured was 12.26 μm root mean square deviation (Rq) and  
45 76.12 μm total height of the roughness profile (Rt). The epoxy resin NVT201E® was then  
46 applied over the treated steel plate surface. Afterwards, the lamination process started by  
47 alternating application of carbon fiber fabric layers and the impregnation resin PIPEFIX®,  
48 as shown in Figure 1. Six plies of carbon fiber were used to produce the composite with a  
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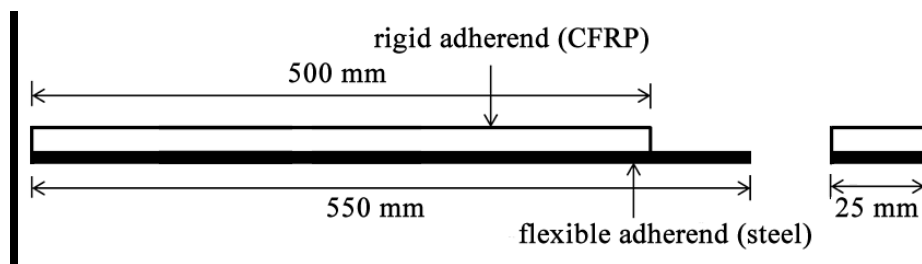
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final layup of  $[0/90]_6$ . This means that the CFRP is oriented alternatively at the specimen longitudinal and transverse direction. The curing process was performed at room temperature for 2 hrs. One layer of glass fiber dry chopped strand mat ( $300 \text{ g/m}^2$ ) was used between epoxy adhesive and carbon fiber fabric layer to avoid direct contact between carbon fiber and steel plate. The mechanical properties of the composite laminate and adhesive were determined in a previous work [28].



**Fig. 1** Carbon fiber lamination process

Peel test specimens were manufactured with 550 mm length flexible substrate, 500 mm length rigid substrate and 25 mm width. The rigid substrate is made of carbon fiber laminated composite, and the flexible substrate is made of carbon steel, as shown in Figure 2.



**Fig. 2** Peel test specimen geometry

The salt water ageing process was employed in order to evaluate the long-term effect of environmental exposure on the joint. The specimens were kept inside the salt water

1 tank (5% NaCl) at room temperature for 40 days and 150 days. The ageing process  
2 simulates the effect of environmental conditions wherein the bonded repairs are exposed  
3 to marine environment. Specimens were exposed to ageing in two different conditions:  
4 partially immersed in salt water, i.e. 50% of bonding length was in salt water and  
5 remaining in atmospheric conditions; and fully immersed in salt water. Some reference  
6 specimens were tested without any ageing. Figure 3 shows the specimens in the salt water  
7 tank and Table 1 provides the ageing conditions tested and the number of specimens for  
8 each condition.  
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33 **Fig. 3** Specimens in salt water tank  
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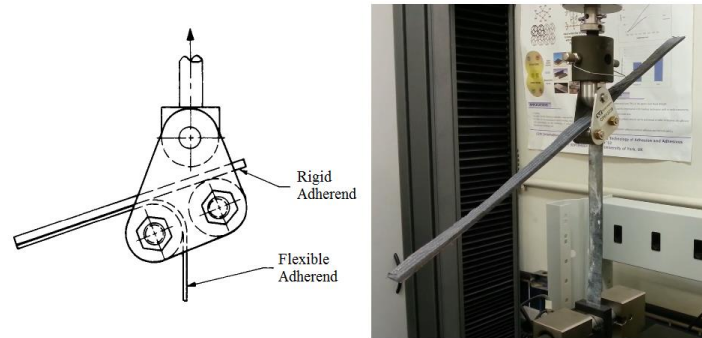
37 **Table 1** Specimens tested  
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Condition	Number of Specimens
non-aged	3
40 days fully aged	3
40 days partially aged	3
150 days fully aged	3
150 partially aged	3

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## 2.3 Floating Roller Peel Tests

The floating roller peel test allows determining the peel strength between a rigid and a flexible adherend. Tests were performed on the non-aged and aged CFRP-to-steel specimens at room conditions, by means of an electro mechanic Instron machine (Series 5966) with a maximum capacity of 20 kN, coupled with a load cell of 1 kN. The testing speed was 152 mm/min., according to ASTM D-3167 for metal substrates [31]. During the test, the flexible adherend is peeled off from the rigid adherend. Figure 4 shows the test set up. Load-displacement curves were recorded during testing.

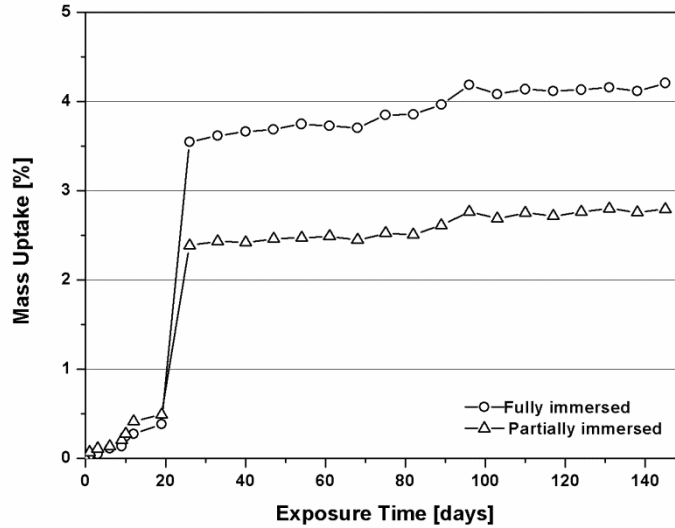


**Fig. 4** Floating roller peel test set up.

## 3 RESULTS AND DISCUSSION

### 3.1 Moisture absorption

The specimens were immersed in salt water for different duration in order to attain different moisture levels in the joints. The specimens were periodically removed from the tank, wipe with tissue paper and then weighed. Weighing was performed on a weight scale with an accuracy of 0.1 mg. Representative curves of the moisture absorption of fully and partially immersed specimen are shown in Figure 5.



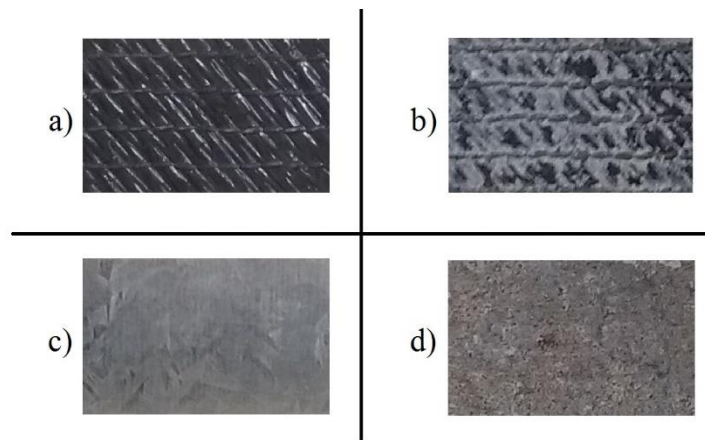
**Fig. 5** Moisture absorption by fully and partially immersed specimens

Mass uptakes of 3.7% and 4.2% were recorded in the fully immersed specimen after 40 days and 150 days of ageing, respectively. In case of partially immersed specimens, moisture uptake of 2.4% and 2.8% were recorded for the same periods. As it can be seen from Figure 5, both curves show similar behaviour of fully and partially immersed specimens. The moisture absorption is higher compared to similar studies of carbon-epoxy laminates [12, 17] and most possible reason for this is the manufacture process of the composite used in this work (hand lay-up may produce voids inside the composite that promotes moisture penetration). Furthermore, most of the moisture absorption occurred in the measurement range between 20 and 27 days in both cases. The presence of moisture causes plasticization, swelling, and degradation of the polymer matrix and deteriorate the fibre/matrix interface [32, 33]. These effects may have weakened the composite properties during the first weeks. This combined with the presence of voids in the material might have caused the steep mass uptake observed.

Figure 6 shows the ageing effect on the composite and steel adherend materials. The composite adherend is shown before immersion (Figure 6a) and after 150 days of



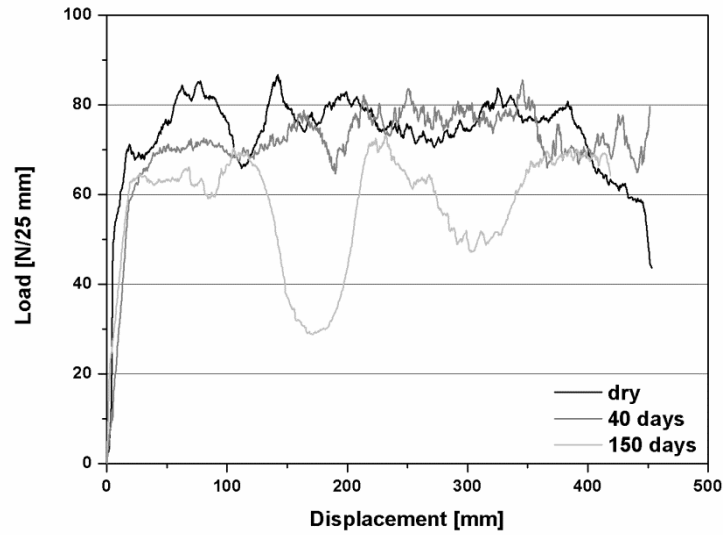
1 exposure to salt water (Figure 6b). Carbon steel is also shown before immersion (Figure  
2 6c) and after the same period of exposure (Figure 6d). The effect of corrosion along the  
3 steel surface can be visualized when comparing the carbon steel surface before and after  
4 conditioning. Otherwise, the composite surface was merely covered with salt in the same  
5 period. This exemplifies one of the main advantages of composite materials in relation to  
6 metals on harsh environment.  
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33 **Fig. 6** Ageing effect on composite (a and b, before and after immersion, respectively) and  
34 metal adherends (c and d, before and after immersion, respectively)  
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### 39 **3.2 Peel Loads**

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42 Representative curves of the test results on fully immersed specimens and non-aged  
43 representative specimens (dry) are shown in Figure 7. The average peel loads ( $F_{ave}$ ) of  
44 fully immersed specimens were determined over the range of 25 to 400 mm of  
45 displacement. The values are shown in Table 2 as average and standard deviation of the  
46 three tests performed in each condition. The large dispersion in the results, shown by the  
47 high value of standard deviation, makes it difficult to analyse the ageing in 500 mm  
48 length joints.  
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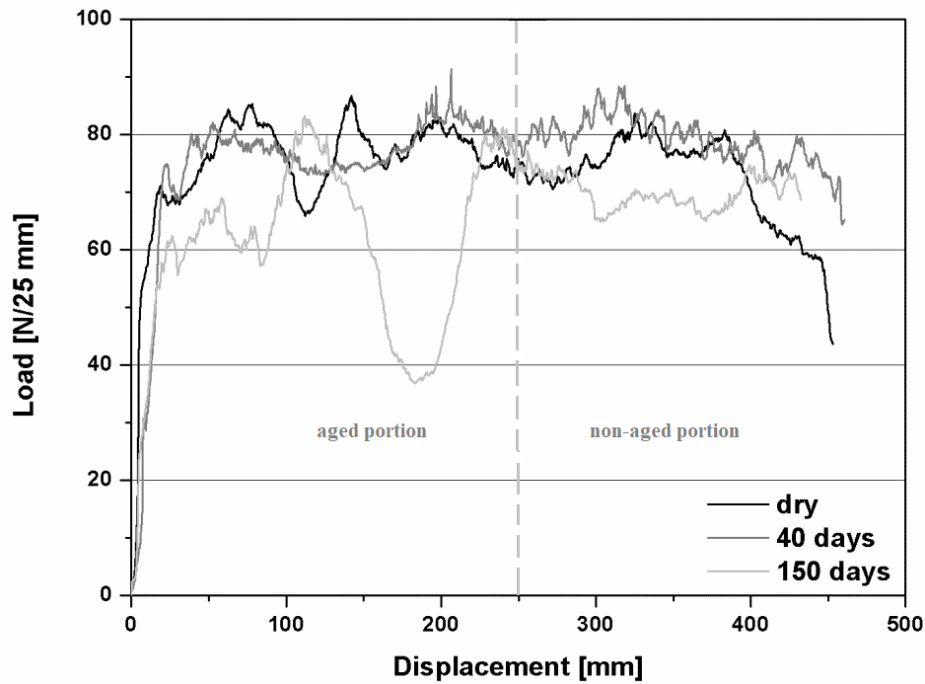


**Fig. 7** Representative load –displacement curves of dry and fully aged specimens (40 days and 150 days)

**Table 2** Average peel loads of fully immersed specimens

Specimen	$F_{ave}$ (N/25 mm)
non-aged	$63.2 \pm 11.8$
40 days aged	$69.9 \pm 3.5$
150 days aged	$63.6 \pm 14.1$

Representative load-displacement curves of the test on partially immersed specimens are shown in Figure 8. The region between 0 and 250 mm displacement corresponds to the aged portion of the specimen and the remaining length correspond to the non-aged portion. The reference non-aged specimen curve (dry) is also shown.



**Fig. 8** Representative curves of dry and partially aged specimens (40 days and 150 days)

The average peel loads of partially immersed specimens were determined over a 150 mm length in two different intervals: 25 to 175 mm and 250 to 400 mm. These intervals were chosen in order to separately analyse the aged and non-aged parts of the specimen. The results are shown in Table 3, as average and standard deviation of the three tests performed in each condition. The average peel loads are lower in the aged part for both 40 and 150 days of ageing, as expected. Significant difference in peel load is observed between 40 days and 150 days for ageing condition. The ageing effect in peel loads is noted on a single partially aged test specimen.

**Table 3** Average peel loads of partially immersed specimens

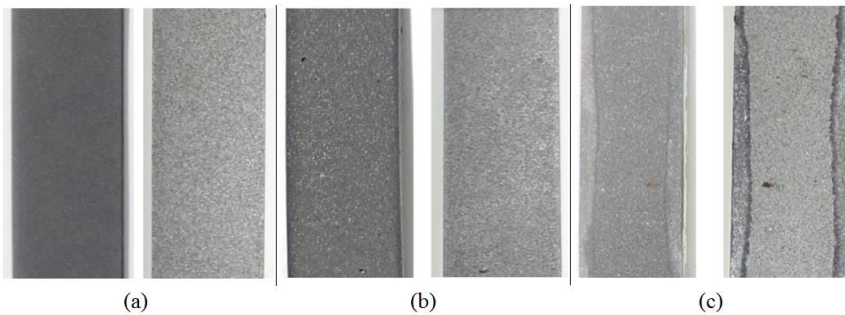
<b>Specimen</b>	<b>F<sub>ave</sub> (N/25 mm)</b>	
40 days aged	non-aged	78.0±6.9
	aged	71.6±8.1
150 days aged	non-aged	64.0±9.2
	aged	62.0±6.1

Load-displacement curves of 40 days aged specimens exhibit the same pattern in both fully and partially aged conditions. It confirms that the influence of moisture on the interface adhesion is insignificant after 40 days immersed in salt water. Otherwise, large decreases in peel loads are observed in fully immersed specimens in salt water for 150 days (Figure 7). The same pattern is noted in the aged portion of partially immersed specimens for the same period (Figure 8). This evidences the effect of ageing on the test results. High deviations on the average peel loads implies in longer ageing periods for more evident results.

### **3.3 Fracture Surfaces**

Visual inspection of the substrates fracture surfaces provides valuable information about the nature of the failure mode. Figure 9 shows the fracture surfaces of the composite adherend (left) and steel adherend (right) of non-aged and aged specimens. Cohesive failure is identified as the prevailing failure mode for non-ageing specimens (Figure 9a). This indicates a good adhesion quality between the composite and steel substrates. It is also observed that the failure occurs near the interface between the steel (flexible adherend) and the adhesive, suggesting the presence of interfacial failure. The

1 fracture surfaces of a 40 days aged specimen are shown in Figure 9b. In this case,  
2 cohesive failure is also identified as the main failure mode, with a small influence of the  
3 moisture in the adhesive layer (less than 5% of the surface). This shows that the adhesive  
4 layer is not significantly affected after 40 days immersed in salt water. Figure 9c shows  
5 the fracture surfaces of a 150 days aged specimen. It is noted that after 150 days of  
6 exposure the adhesive layer was significantly affected by moisture (about 20% of the  
7 surface). The moisture effect pattern remains at the edges of the surfaces.  
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31 **Fig. 9** Fracture surfaces of (a) non-aged, (b) 40 days aged and (c) 150 days aged  
32 specimens after peel tests  
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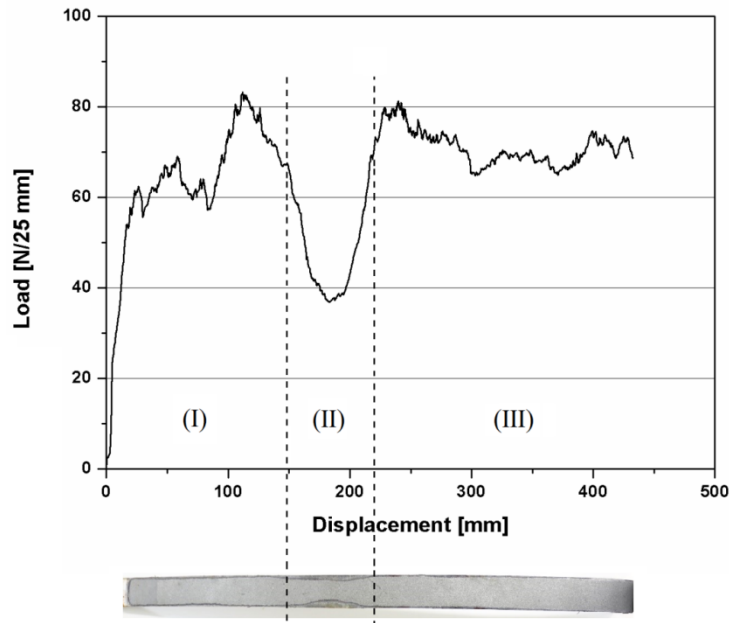
38 Moisture absorption (Figure 5) and fracture surfaces (Figure 9) are now related.  
39 Moisture was mostly absorbed by the composite substrate during the first 40 days of  
40 ageing. The adhesive layer was not significantly affected until this moment. It is also  
41 observed that there was a considerable growth of the region of the fracture surface  
42 affected by moisture between 40 and 150 days of ageing. This shows that most of the  
43 moisture absorption in the adhesive layer occurred during this period. The pattern of  
44 fracture surfaces at different aging times shows that moisture penetrates from the edges  
45 toward the centre of the adhesive layer. It implies that a reduction of the specimen width  
46 can enhance the results related to the conditioning process. A reduced width would  
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1 increase the ratio of affected area on the fracture surface. Finally, the white coloration  
2 presented in areas affected by moisture suggests that there was deposition of salt at the  
3 adhesive layer during ageing times.  
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#### 7 8 **4 DISCUSSION** 9

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11 The average peel load of  $63.2 \pm 11.8$  N/25 mm was measured for non-aged specimens  
12 (500 mm length) tested in the present work. While in previous work [28] similar  
13 specimens with 250 mm length were tested and an average peel load of  $73.6 \pm 1.2$  N/25  
14 mm was determined along 150 mm length. Increasing the length of the specimen  
15 promoted an intensification in the discrepancies of test results. This may be caused by an  
16 increase in defects originated from specimens manufacture. Moreover, the salt spray  
17 ageing process used in [28] generated consistent results in 90 days while the same results  
18 were not reached in 150 days ageing in salt water.  
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31 In order to obtain a better understanding of the ageing effects on the adhesion  
32 performance, peel loads were related to fracture surfaces of 150 days aged joints, as they  
33 presented a more significant influence of moisture on the adhesive layer. Figure 10 shows  
34 a comparison between load-displacement curve and fractured surface of the flexible  
35 substrate in a partially aged tested specimen.  
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**Fig. 10** Comparison between peel load and fracture surface of a 150 days partially aged specimen

Three different regions can be identified by visual inspection along the curve: I, II and III. Regions I and II correspond to the aged part of the specimen while region III is the non-aged part. The moisture penetration at the fracture surface is small and reasonably uniform in the region I (between 25 and 145 mm of displacement) and corresponds to an average peel load of  $62.6 \pm 12.1$  N. This value is close to the average peel load of 150 days fully immersed specimens showed in Table 2. Region II presents a considerable increase of moisture penetration, which is followed by a decrease in peel loads until 36.8 N. Region III (from 220 mm to the end) presents an average peel load of  $71.1 \pm 3.8$  N. This value is close to the average peel load of non-aged specimens (Table 1). There is a 12% reduction in the average peel load between regions I and III. In addition, the minimum peel load occurred in region II is about 48% lower than the average peel load of the non-aged region. These percentages are proportional to the penetration of moisture in

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the fracture surface. This reveals the relationship between ageing effect and reduction of adhesion quality.

## 5 CONCLUSIONS

Peel tests were carried out on composite-to-steel bonded joints to study the effect of salt water. A recent developed type of peel test was applied in which a composite rigid adherend is bonded to a flexible metal adherend. New specimen configurations with longer length (500 mm) were fabricated in order to study the wet (ageing) and dry (non-ageing) behavior in the same specimen.

The following conclusions can be drawn:

- There is no significant effect of moisture on the adhesion after 40 days of immersion in salt water. Fracture surface shows a cohesive failure mode as in the non-aged specimens. During this period, the moisture absorption by the adhesive interface is minimal.

- There is a considerable gain in moisture at the interface between the immersion period of 40 days to 150 days. However, the ageing process proved to be time-consuming. For faster and more evident results, either the specimen width should be reduced or a more severe ageing process should be employed (at higher temperatures for example).

- The penetration of moisture at the adhesive interface always occurs from the edges towards the centre of the interface, presenting an irregular pattern. Longer ageing periods produce higher moisture penetrations and therefore lower peel strengths.

- The peel strength of the non-aged specimens changed according to the total length of the specimens. Increasing the length of the specimen intensifies the discrepancies of the test results. This may be caused by an increase in intrinsic manufacture defects of the specimens. However, the new configuration shows a more realistic situation where the composite repairs are applied on larger area.



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- The new specimen configuration (500 mm length) successfully assessed the interface adhesion for ageing and non ageing condition in same specimen using peel test, provided the defect free manufacturing of the composite-metal bonded joints.

### **Acknowledgements**

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### **Conflict of Interest**

The authors declare that they have no conflict of interest.

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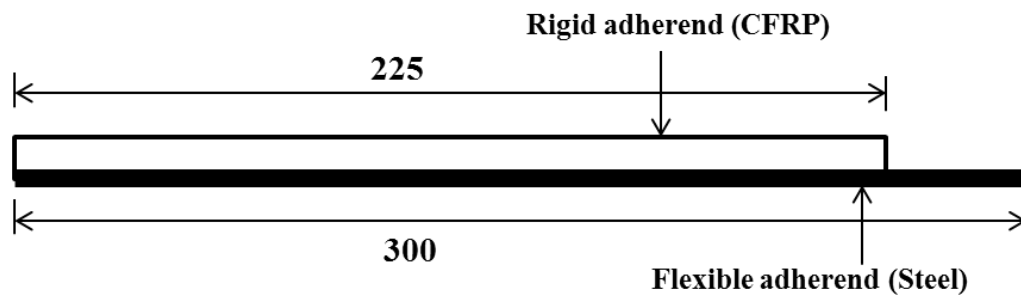
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## Figures



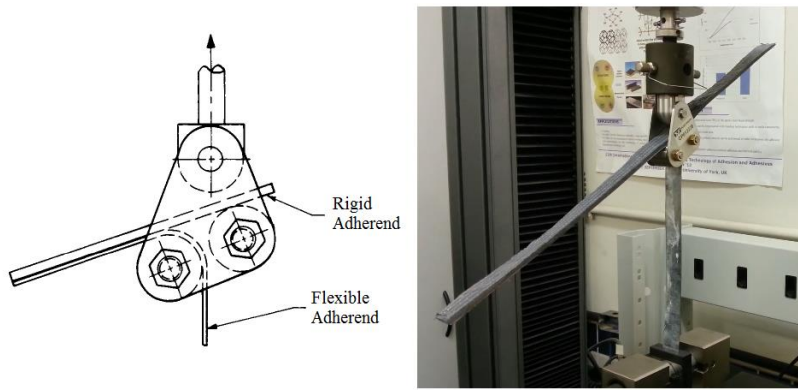
**Fig. 1** Carbon fiber lamination process



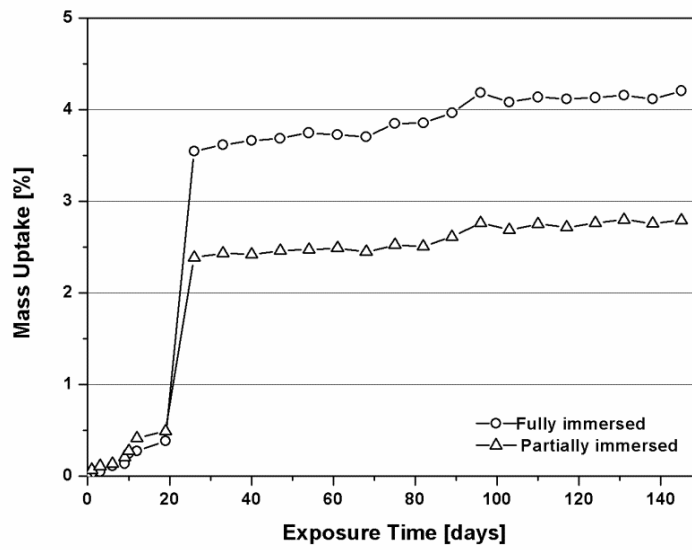
**Fig. 2** Peel test specimen



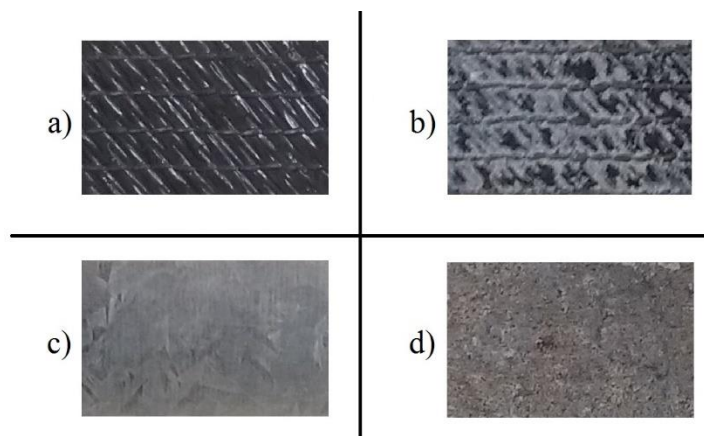
**Fig. 3** Specimens in salt water tank



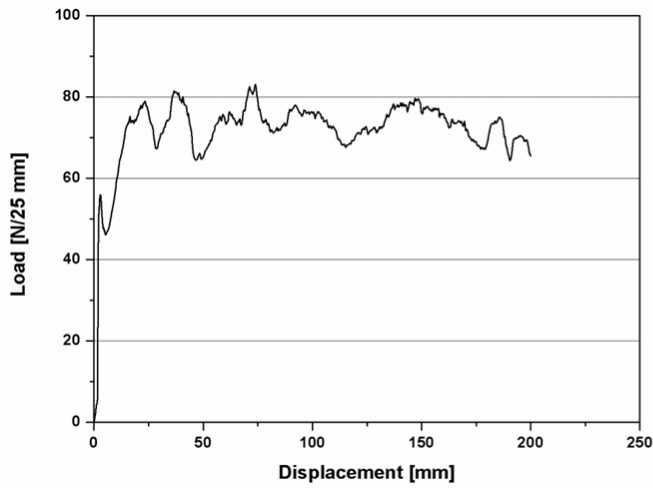
**Fig. 4** Floating roller peel test set up



**Fig. 5** Moisture absorption by the partially and fully immersed specimens



**Fig. 6** Ageing effect on composite (a and b, before and after immersion, respectively) and metal adherends (c and d, before and after immersion, respectively)

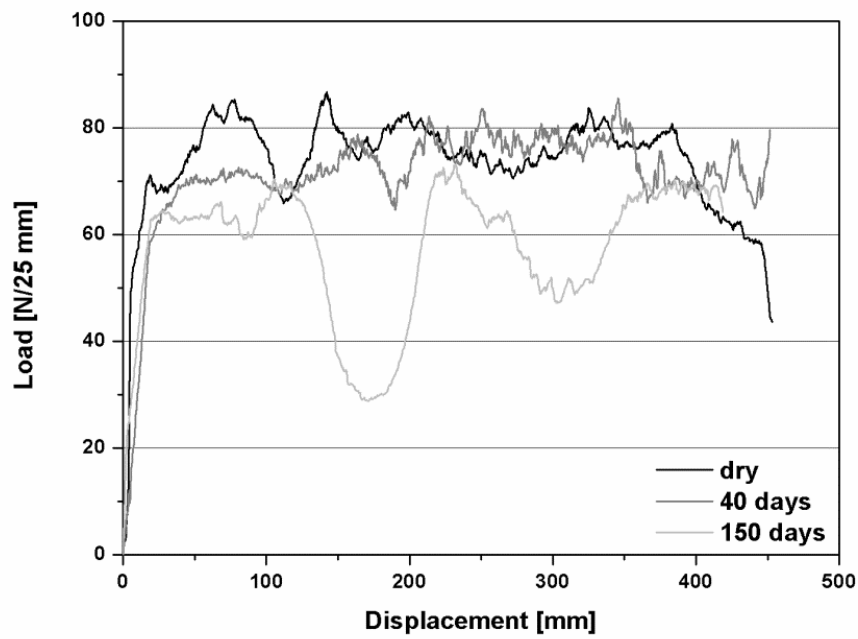


(a)

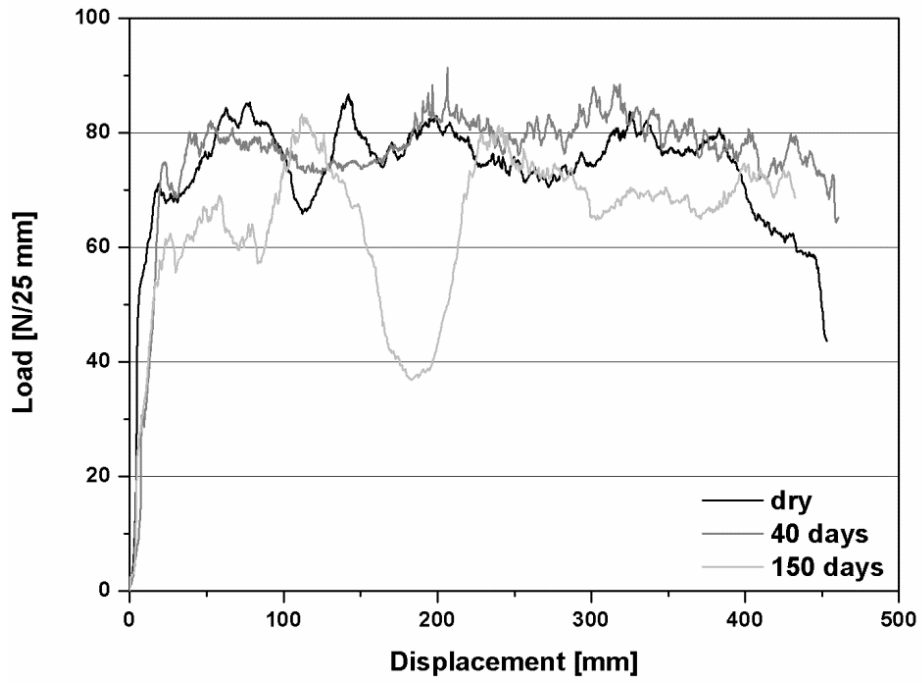


(b)

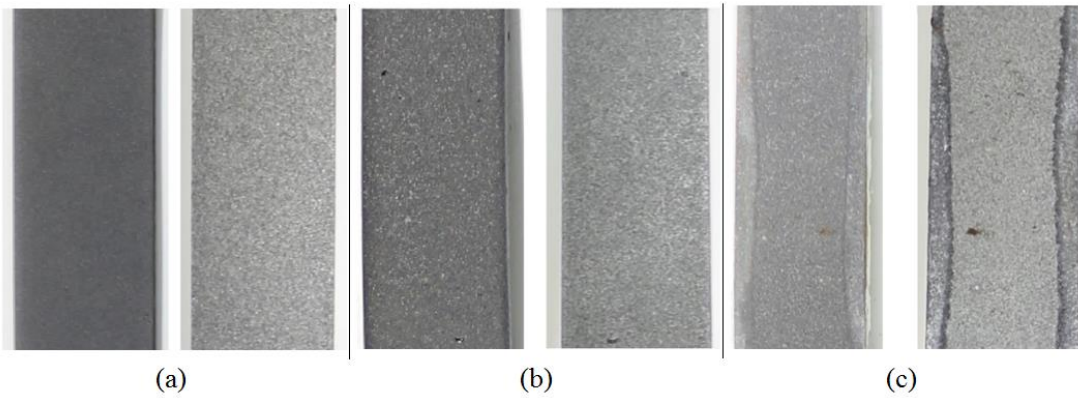
**Fig. 7** Representative curves of dry specimens with: a) 250 mm length; and b) 500 mm length



**Fig. 8** Representative curves of dry and fully aged specimens (40 days and 150 days)

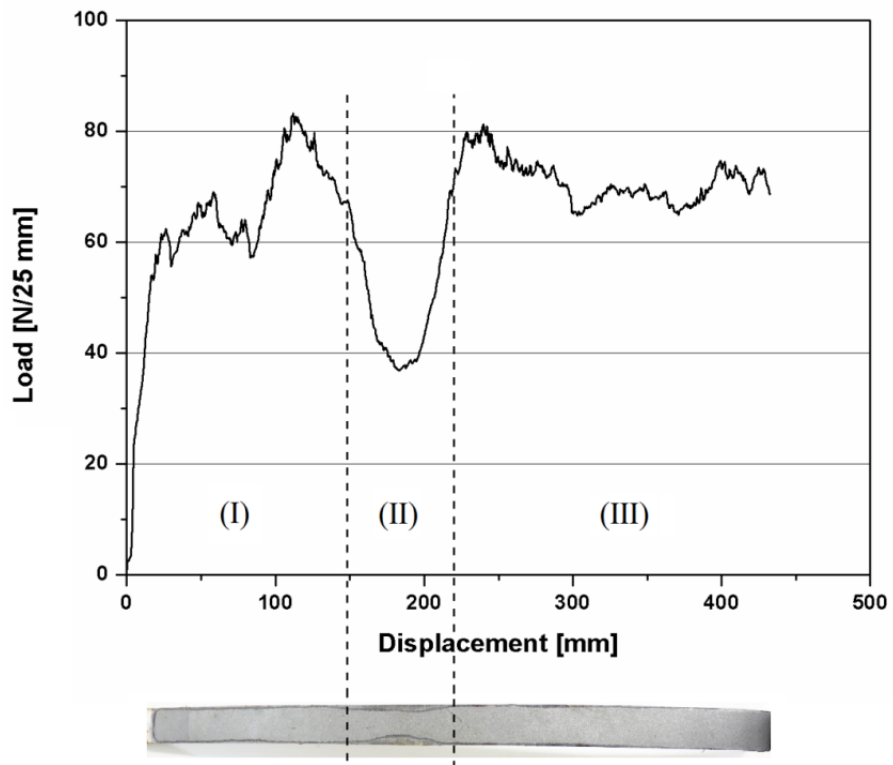


**Fig. 9** Representative curves of dry and partially aged specimens (40 days and 150 days)



**Fig. 10** Fracture surfaces of (a) non-aged, (b) 40 days aged and (c) 150 days aged specimens after peel tests





**Fig. 11** Comparison between peel load and fracture surface of a 150 days partially aged specimen