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Chemical Surface Treatment and Seawater Aging Effects on AA6082-T6 Adhesive Joints

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ABSTRACT: The mechanical strength and durability of the bonds between structural components is a major contributor to the performance of an engineered structure. Due to the potential for weight savings while preserving excellent mechanical characteristics and providing even load distribution without stress concentrations or peak stresses, adhesively bonded joints are being widely adopted in engineering. Therefore, they represent an attractive alternative to other fastening methods, particularly in structural applications where high loads are present. In the current research effort, the effects of a chemical surface treatment and seawater aging on aluminum alloys (AA6082-T6) were investigated. Epoxy resins were chosen as the adhesives for this study because of their excellent mechanical behavior and suitability for various applications. Specifically, two types of epoxy adhesives were evaluated: flexible (Araldite 2015) and rigid (Araldite AV 138/HV 998) adhesives. All specimens were made using single lap joints as defined by ASTM D 1002. To enhance the bond strength between the adhesive and the substrates, three various surface treatments were employed. Specimens were then aged in seawater at room temperature for varying lengths of time. Mechanical testing (tensile tests) was conducted to determine the effects of aging and the type of surface treatment on the mechanical strength and durability of the bonded joints. The results clearly indicate that both the surface treatment and aging duration had significant effects on the performance of bonded joints. Compared to the rigid adhesive, the flexible adhesive exhibited superior tensile strengths and greater resistance to degradation. The surface treatment that provided the best bonding and durability was identified. As expected, tensile strength decreased with increasing aging time due to moisture absorption into the substrate and interfacial degradation. The current research demonstrates the importance of appropriate surface preparation and adhesive choice when designing and implementing adhesive-bonded aluminium alloys for long term use in marine environments.

1. INTRODUCTION:

The use of structural adhesives has been greatly enhanced through technological advancements in manufacturing technology, which has increased both the reliability and performance of structural adhesives. In this context, the number of engineering applications that utilize structural adhesively bonded joints is increasing, because they offer an effective means of transferring loads efficiently with minimal added weight to the structure. Compared to other forms of joining (i.e., welding and mechanical fastening), adhesive bonding has significant advantages when it comes to the transfer of loads. Specifically, adhesive bonded joints exhibit the potential for a more uniform distribution of stress than do welded or mechanically fastened joints, a reduced concentration of stress at critical points within the joint, longer fatigue lives compared to welded or mechanically fastened joints, and a less complex manufacturing process [5-7]. However, like all types of structural connections, adhesive bonded joints also possess inherent disadvantages. For example, issues related to poor adhesion to particular substrate materials, limitations in the resistance of adhesive bonded joints to peel and cleavage stresses, the effects of environmental degradation and the economic performance trade-off associated with adhesive bonding can affect their overall performance and efficiency.

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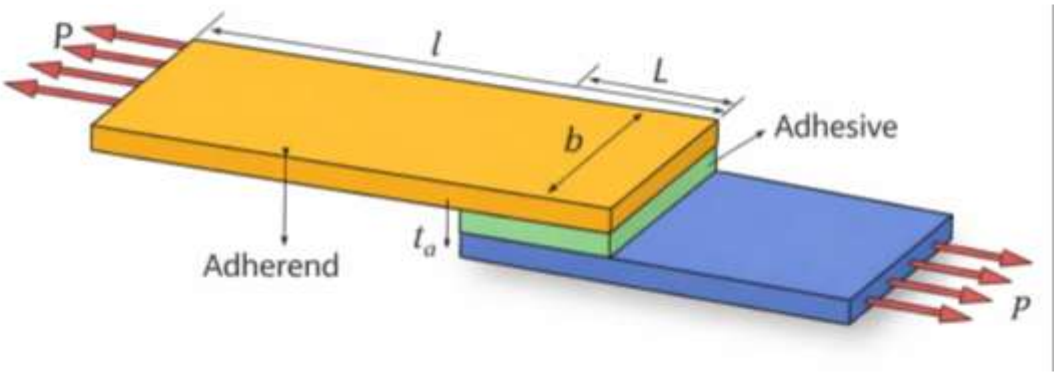

Figure1 :- Single lap Joint.

The strength and durability of an adhesive joint depends upon many things such as; Joint Geometry, Service Conditions, Temperature, Adhesive Type and Surface Condition of the Adherends [1,10]. The surface characteristics of Aluminium Alloys have been found to be significant in determining their adhesion performance. When a proper surface treatment is performed, it results in improved wettability, removal of contaminants, enhanced mechanical interlocking which in turn contributes to increased bond strength [10-12]. Additionally, the type of adhesive has a significant impact on joint behavior. The flexible adhesives can deform and absorb stresses, which reduces the stress concentrations at the joint, whereas the stiff adhesives provide increased stiffness to the joint but are also more likely to fail under brittle conditions [2,6,13]. Therefore, to improve the joint performance, the effects of both surface treatment and adhesive type should be understood. This paper focuses on evaluating the surface treatment of AA6082-T6 Aluminum Alloy. The effects of surface treatment on joint performance will be examined. Two epoxy adhesive systems with different mechanical properties are evaluated using Araldite brittle AV138/HV998 and ductile Araldite 2015 types of adhesive and [1,13]. In this study, a test program was implemented using Single Lap Joints (SLJ). The test specimens were produced according to ASTM D1002. SLJ one of the most commonly used configurations in structural applications because of its simplicity in terms of manufacturing, ease of testing and similarity to engineering design practices. The main concept of this to investigate how different surface chemical treatments affect the mechanical behavior, failure mode and joint strength of AI-AI joints adhesive that utilize two different types of adhesives.

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2. THEORETICAL BACKGROUND

When analyzing single lap joints (SLJ) theoretically or numerically, it is commonly assumed that there is no gap between the adherends and the adhesive layer, and therefore, failure will occur within the adhesive layer. Although this assumption may be accurate in some theoretical analyses, it does not accurately represent real-world applications. Interfacial failures have been reported as occurring when the bonding conditions are less than ideal. As a means of reducing these types of problems, aluminum surfaces prior to bonding are often treated. Surface treatment increases the cleanliness of the surface, the surface roughness, and the chemical activity of the surface, thereby improving adhesion [10-12,15]. Additionally, the application of a

Natural Resources for
Human Health
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primer or adhesion promoter improves bonding by providing an intermediate layer between the adhesive and the substrate [11,15]. Ideally, failure would occur within the adhesive layer, indicating good adhesion at the interface [1,6,13]. Commonly observed failure modes in adhesive joints include those listed below in figure 2 [5,7]: Cohesive failure occurs within the adhesive layer. This failure mode indicates that the adhesive-adherent bond is stronger than the adhesive itself and is typically considered acceptable [1,6]. This failure mode is most likely due to inadequate surface preparation and/or contamination, resulting in weak adhesion [10-12]. A mixed failure occurs where cohesive and adhesive failure occur concurrently. This failure mode indicates that the adhesion between the adhesive and the adherend is not entirely satisfactory and that the interface has partially degraded [5,9]. Finally, adherent failure occurs when the adherend fails rather than the adhesive layer. This failure mode indicates that the joint has sufficient strength to withstand the load applied to the adherend [2,7].

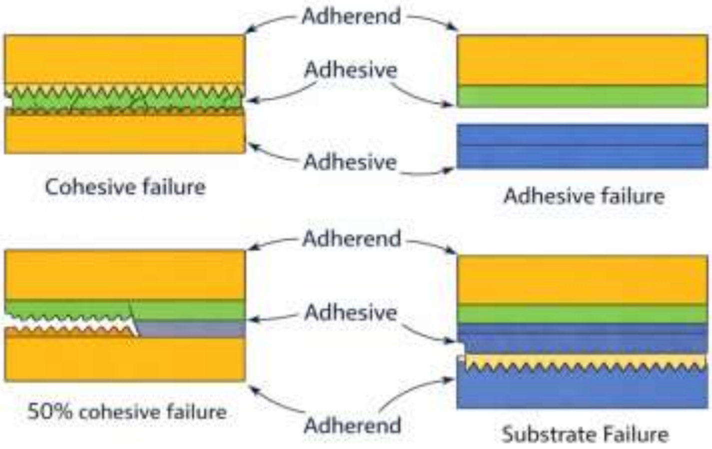

Figure 2: - Types of adhesive failure mechanism

Chemical surface treatments are also very important when it comes to preparing surfaces for adhesive bonding. Chemical surface treatments have been shown to enhance the mechanical interlocking and increase the surface energy at the metal-adhesive interface. Chemical surface treatments can be very effective at cleaning the surface of contaminants and creating a uniform and controlled surface chemistry. As with all surface treatment methods, there are some limitations to using chemical surface treatments. For example, some chemical surface treatments may require special handling, equipment, and/or safety precautions. Additionally, some chemical surface treatments may not work well with certain types of adhesives, so care should be taken when choosing a chemical surface treatment to ensure that it will meet the needs of specific project.

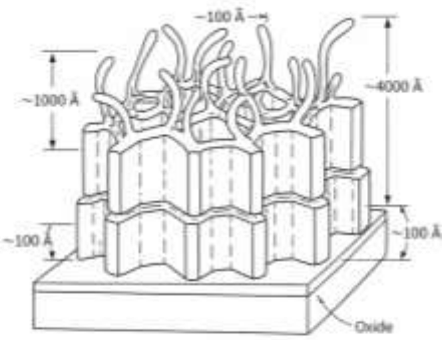

Figure 3: - Diagram showing the microscopic oxide layer formed after phosphoric acid anodizing.

2.1 Anodizing using sulfuric acid (SAA)

Sulfuric acid anodising is among the most common ways of improving the ability of aluminium to bond to other materials particularly in wet conditions [11,16]. The anodising process is performed by submerging an aluminium specimen in a sulphuric acid-based electrolyte with an applied electric current. The current generates a It forms a very thin, porous oxide layer on the surface that enhances of the surface to bond by providing better mechanical lock and improved wetting [11,16,17].

2.2 Boric–Sulfuric Acid Anodizing Surface Treatment (BSAA)

Boric-sulphuric acid anodising is viewed as a safer method of anodising than traditional anodising processes [16,18]. Boric-sulphuric acid anodising can produce a stable oxide film with good adhesion and durability. As a result, it has become more commonly adopted as a replacement for many hazardous chemical anodising treatments, including phosphoric acid anodising (PAA) and chromic acid anodising (CAA). Although these treatments have been effective they are being replaced by less toxic methods to reduce potential environmental and health impacts [16,18,19].

2.3 Alkaline Surface Etching (ASE)

Alkaline surface etching is a simple pre-treatment used to enhance adhesion on aluminum surfaces.. The specimens are submerged into an alkaline solution, which typically includes sodium hydroxide (NaOH). The treatment will remove the normal oxide layer from the surface of the material, and also remove contaminants. The surface of the specimen will become rough, allowing for increased surface area, therefore improving mechanical interlock with the adhesive [11,15]. The primary chemical reaction during the alkaline surface etch is:

$$2Al + 2NaOH + 2H_2O \longrightarrow 2NaAlO_2 + 3H_2$$

Following etching, the surface typically has a neutralization step using a mild acid (i.e., nitric acid), to clean off any residue left over and stabilize the surface:

$$NaAlO_2 + HNO_3 \longrightarrow Al(OH)_3 + NaNO_3$$

This method is economical, and is easy to apply for laboratory scale and industrial applications. The alkaline surface etch provides excellent adhesion performance; however, the corrosion resistance will be less than that of anodic treatments.

3. MATERIALS

The Aluminum 6082-T6 plates that were 1.5 mm thick were chosen for the adhesive substrates since they have many structural applications [18,20]. Lap joints were created for testing single lap joints of 25 mm. In addition to meeting ASTM E8M-09 specifications [21], the two structural adhesives provided by Huntsman International India Pvt. Ltd. were tested on different sets of specimens. The first set of specimens were made using ductile Araldite 2015 and the second set of specimens were made using brittle Araldite AV138/HV998 [1,13]. The geometric and dimensional configurations of the specimen can be seen in figure one and the material characteristics can be found in tables 1 and 2.

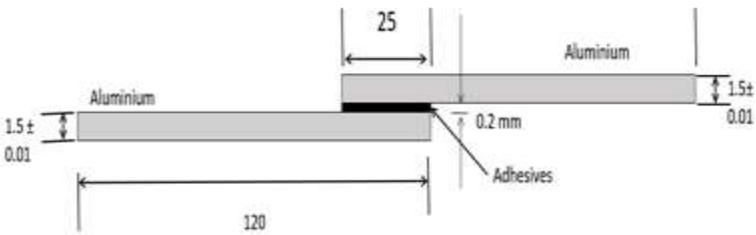

Figure 4 :- Geometry of single lap adhesive joint with 25 mm width (dimensions in mm).

Table 1: Mechanical Characteristics of AA6082-T6 Alloy [17].

Parameter	**Magnitude**
Elastic modulus, E (GPa)	69.5
Failure strain, ε_f (%)	16.5
Modulus of rigidity, G (GPa)	25.3
Poisson's ratio	0.35
Yield strength, σ_y (MPa)	245.1
Ultimate tensile strength, σ_{UTS} (MPa)	305.6

Table 2 – Shear Behavior of Selected Epoxy Adhesives [18].

Property	Araldite® 2015	Araldite® AV138/HV998
Shear modulus (MPa)	487 ± 77	1559 ± 11
Shear yield stress (MPa)	17.9 ± 1.8	25.0 ± 0.55
Maximum shear strength (MPa)	17.9 ± 1.8	30.2 ± 0.4
Shear strain at failure (%)	43.9 ± 3.4	5.5 ± 0.44

3.1 Experimental Method

A full factorial DOE was used to evaluate the influence of three variables on the mechanical properties of bonded jointed specimens. Variables were adhesive type, surface treatment, and aging duration; the three variables were combined to create 2 x 3 x 5 = 30 combinations of variable levels. For each combination of variables, there were 3 replicate specimens; these were subjected to a 60-day seawater immersion exposure to simulate marine conditions. The detailed experimental matrix is shown in Table 3. As a result, a total of 90 specimens were tested.

Table 3 : DOE Matrix for Tensile Testing

Exp. No.	Adhesive	Treatment	Aging (Days)	Replicates	Total Samples
1	2015	T1	8	3	3
2	2015	T1	16	3	3
3	2015	T1	24	3	3
4	2015	T1	48	3	3
5	2015	T1	60	3	3
6	2015	T2	8	3	3
7	2015	T2	16	3	3
8	2015	T2	24	3	3
9	2015	T2	48	3	3
10	2015	T2	60	3	3
11	2015	T3	8	3	3
12	2015	T3	16	3	3
13	2015	T3	24	3	3
14	2015	T3	48	3	3
15	2015	T3	60	3	3
16	AV138	T1	8	3	3
17	AV138	T1	16	3	3
18	AV138	T1	24	3	3
19	AV138	T1	48	3	3

20	AV138	T1	60	3	3
21	AV138	T2	8	3	3
22	AV138	T2	16	3	3
23	AV138	T2	24	3	3
24	AV138	T2	48	3	3
25	AV138	T2	60	3	3
26	AV138	T3	8	3	3
27	AV138	T3	16	3	3
28	AV138	T3	24	3	3
29	AV138	T3	48	3	3
30	AV138	T3	60	3	3

2015 = Araldite 2015 (ductile adhesive), AV138 = Araldite AV138/HV998 (brittle adhesive), T1 (SAA), T2 (BSAA), T3 (ASE) = Surface treatments.

4. RESULTS AND STATIC CHARACTERIZATION

This study is based on a comparison of two different types of adhesives. One type is ductile while the other is brittle. Tensile testing has been used to obtain the typical load-extension curve for each type of adhesive as shown in figure 3[1,13].. The ductile adhesive exhibits a nearly straight line response to loading at lower loads. Once the load exceeds the elastic limit of the material (i.e., when it is loaded beyond its elastic limit), there is a deviation from this straight line response. This deviation occurs due to plastic deformation [6] . On the other hand, the brittle adhesive will exhibit a bilinear response to loading. The bilinear response exhibited by the brittle adhesive results from the development of microcracks near the fillet region of the adhesive. These microcracks reduce the stiffness of the joint [5,24].

Initially, joints bonded with the brittle adhesive show higher stiffness than those with the ductile adhesive. However, after the initiation of micro-cracks, the stiffness decreases and becomes lower than that of the ductile joints [5]. In terms of extension at failure, brittle adhesive joints show approximately 35-40% lower values compared to ductile ones. The failure load is higher for ductile adhesive joints, indicating better load-bearing capacity [13]. Figure 5 shows the fracture surfaces after testing. Visual inspection and optical microscopy indicate that failure is mainly cohesive for both adhesives [1,6]. A thin layer of adhesive remaining on the substrate confirms that failure occurs within the adhesive layer, typically near the interface. This suggests that failure is controlled by the maximum strain capacity of the adhesive [1,7].

Figure 5:- Surface morphology of failure in Araldite® 2015 adhesive under tensile conditions.

Natural Resources for
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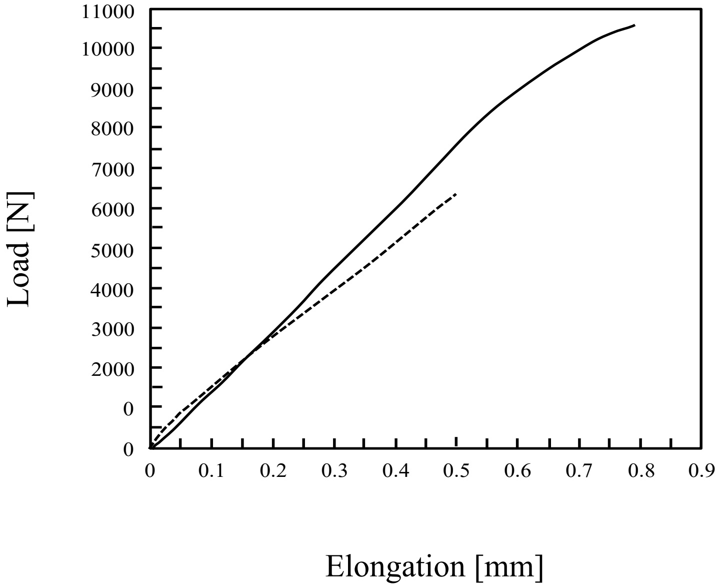

Figure 6:- Variation of load with extension observed during tensile testing

Figure 7 shows the variation of tensile strength with aging time for Araldite 2015 under different surface treatments. A gradual decrease in strength is observed as the aging duration increases for all cases. This indicates that exposure to seawater has a negative effect on joint performance. Surface treatments are critical for developing high quality joints. Of the three surface treatments studied in this research project, T2 has consistently produced the highest tensile strengths for every aging duration studied. Therefore, T2 provided superior surface preparations as well as superior interfacial bond properties. These advantages resulted in a longer time before moisture penetrated through the joint and resulted in improved durability. T1 showed moderate tensile strengths while T3 exhibited the poorest tensile strengths. Therefore, the weakest bonds were developed with T3 and, therefore, the fastest rate of degradation was observed.

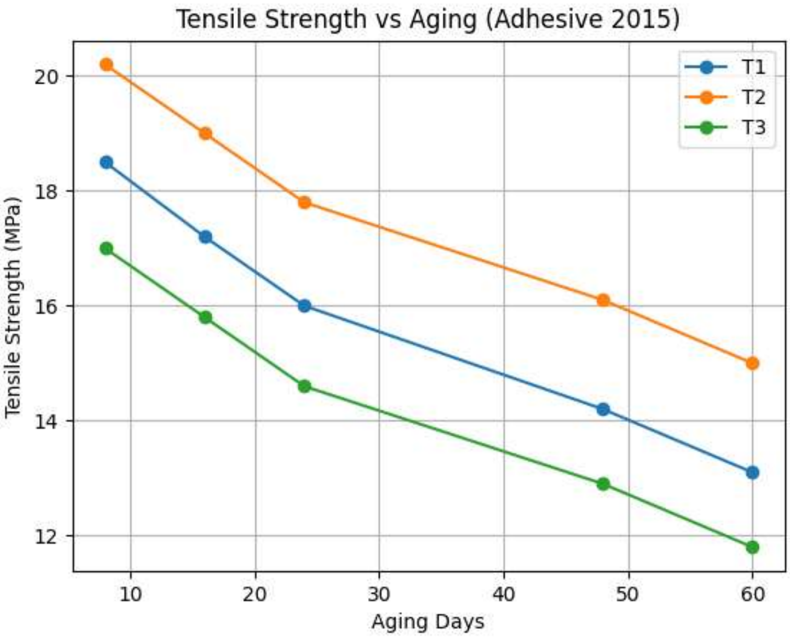

Figure 7:- Variation of tensile strength with aging duration for Araldite 2015 under different surface treatments.

At short aging times (i.e., 8-16 days), the tensile strength decreased by a small amount. Therefore, at these early stages, the adhesive layer and interface were relatively unchanged. However, after 24 days, a greater degree of tensile strength loss began to occur. The primary cause of the strength loss at this point is due to the absorption of moisture into the adhesive and the interface. Once water entered the adhesive and the interface, the adhesive lost some of its ability to adhere to the surfaces that it was bonded to and the adhesive softened. At long aging durations (i.e., 48-60 days), the loss of tensile strength increased even more than at the previous aging durations. This increase in strength loss can be attributed primarily to continuous moisture diffusion, swelling of the adhesive, and damage to the interface. In addition, micro-level defects could begin to develop at this point which would ultimately further degrade the load carrying capabilities of the joints. Despite this degradation, Araldite 2015 maintained a higher tensile strength than the other two adhesives studied. Araldite 2015's ability to maintain a higher tensile strength is due to its ductile nature. When subjected to stress, Araldite 2015 will deform and distribute the stresses across the bond. This ability to deform and distribute stresses results in delayed failures when compared to brittle adhesives.

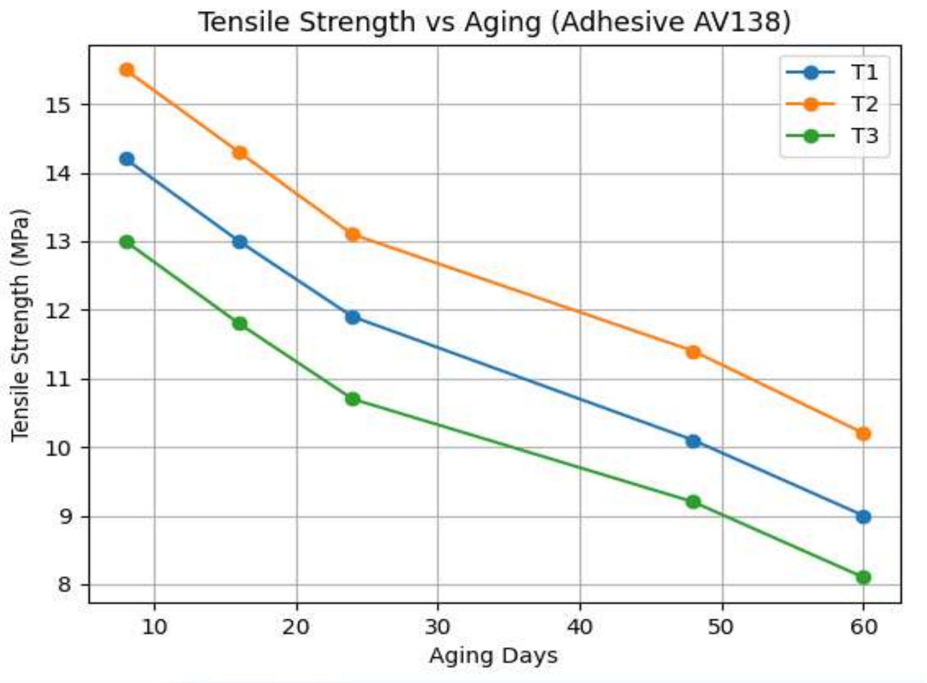

Figure 8:- Morphological features of the failure surface in water-immersed brittle adhesive samples.

The influence on the tensile strength of Araldite AV 138 as a function of the aging time by using various surface treatments has been shown in Fig. 8. The tensile strength continuously diminishes as a function of the aging time. The drop of the tensile strength is much larger than that of a ductile adhesive due to the brittleness of AV 138. In comparison to the other treatments, the highest tensile strength at all times was obtained from treatment T 2. Treatment T 2 leads to higher bonding forces and to an improved moisture resistance. Treatment T 1 results in a moderate behavior; treatment T 3 yields the weakest tensile strength, i.e. the poorest bonding forces and therefore the fastest deterioration. Already at short aging times (from 8 to 16 days) a significant loss of tensile strength occurs. Even at such an early aging state the tensile strengths are, however, lower than those obtained from ductile adhesive systems. Between 24 and 30 days the drop in tensile strength is strongly accelerated. This acceleration is caused primarily by the moisture absorption into the adhesive layer and by the generation of microcracks. For the aging times 48 and 60 days the tensile strength drops very strongly. Due to the brittleness of the adhesive it cannot be able to absorb stresses efficiently. Therefore, the crack growth takes place more quickly and thus already at an earlier point in time the failure of the adhesive joint occurs. Furthermore, the moisture can weaken the adhesive/substrate interface, which leads again to a drop in the tensile strength. Thus, the brittle adhesive AV 138 shows a lower durability when subjected to seawater aging.

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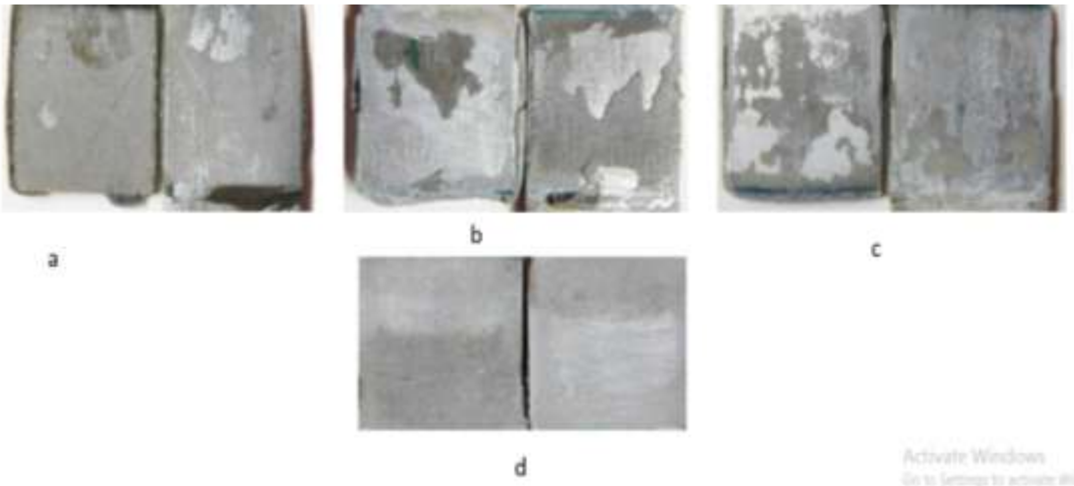

Figure 9:- Failure surface morphology obtained with the brittle adhesive for samples immersed in water at:

5. FAILURE SURFACE ANALYSIS OF TESTED JOINTS

The surfaces of a few samples that were tested under tension are shown in Fig. 9 (a – d) and illustrate the fracture surface features of the adhesive bonds. The fracture surfaces give an indication of the degree of bond quality as well as of the mechanism(s) of failure of the adhesive joints. As indicated in Fig. (a), the left-hand side of this sample has only partial adhesive coverage while the right-hand side of this sample has no adhesive covering and is completely exposed. Therefore, the failure was a combination of adhesive failure and mechanical failure. It appears that there was a poor bond between the two surfaces; possibly because of the improper surface cleaning before applying the adhesive or possibly because of the effects of water damage occurring early in the application process. In Fig. (b), it can be seen that approximately half of the original area of the sample is still coated with adhesive. However, in certain areas, the base metal is visible through the coating. Thus, the failure was a combination of adhesive failure and mechanical failure. However, as noted above, the area of mechanical failure is less than that which was present in Fig. (a). Also, there appears to be a significant amount of adhesive remaining on the surface indicating that the bonding between the adhesive and the surface of the base metal was significantly stronger than in the case illustrated in Fig. (a). Therefore, this result indicates that the bonding between the two surfaces is becoming more effective and that the load is being transferred more effectively. In Fig. (c), it can be seen that nearly all of the surface of the sample is coated with adhesive and the base metal is only slightly exposed. Therefore, the failure occurred entirely within the adhesive and was therefore a mechanical failure. This indicates that the bonding between the adhesive and the surface of the base metal is quite good and that the strength of the bond is determined by the strength of the adhesive itself. In Fig. (d), a nearly uniform and smooth surface is observed with little if any adhesive remaining on either surface. Therefore, the failure was a complete mechanical failure and occurred entirely along the interface between the two surfaces. This type of failure typically indicates that the surface was contaminated prior to bonding, was not properly treated, or had been severely degraded by the environment.

6. CONCLUSION

The tensile strength of the bonded joints decreased with an increase in the time exposed in seawater. This further supports that the presence of moisture negatively affects the overall performance of the bond. Araldite 2015 (a ductile adhesive) had the highest tensile strength and exhibited better durability than Araldite AV 138 (a brittle adhesive) which showed lower tensile strength. Ductility aids in redistributing stresses within the adhesive layer thus delaying failure. Surface treatments significantly affect the tensile strength of the joints. The highest tensile strengths were observed from specimens treated using T2, followed by those treated with T1, while the lowest tensile strengths were observed from specimens treated with T3. The reduction in tensile strength was gradual during the initial aging times (8 – 16 days), but became more pronounced as the aging time increased (24 – 60 days). These differences are primarily attributed to the progressive diffusion of water into the adhesive layer causing degradation of the adhesive–adherent interface.

Failure surface analyses indicated various failure modes including adhesive, cohesive and mixed failures. A cohesive failure mode demonstrates a high level of adhesion at the adhesive–substrate interface; whereas, adhesive failure indicates a poor adhesive–substrate interface. Adhesive failure and early joint failure occurred in specimens where the surface was poorly treated. In contrast, specimens that were well-treated exhibited cohesive failure and demonstrate good adhesive quality. Degradation of the adhesive substrate interface occurs through moisture absorption, softening of the adhesive, and degradation of the adhesive–substrate interface. Use of a ductile adhesive combined with optimal surface treatment improves the resistance of the adhesive substrate interface to environmental degradation and enhances the reliability of the bonded joint. This research emphasizes the importance of choosing the correct surface preparation method and adhesive material for long term performance in a marine environment.

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