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Chemical Etching for Boosting Adhesive Bond Strength of CFRP–Al Joints: A Lap Shear Tensile Test Study

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ABSTRACT

Carbon fiber–reinforced polymers (CFRPs) have gained widespread use across the aerospace, automotive, and energy sectors due to their high strength-to-weight ratio. Many structural applications require joining CFRPs to aluminium alloys, with adhesive bonding offering several advantages over mechanical joining methods, including uniform stress distribution and compatibility with CFRPs. Among various strategies to improve bond strength, surface preparation plays a critical role. This study investigates the effect of chemical etching on CFRP using sulfuric acid solution to enhance bond strength in single-lap joints with aluminium alloys when tested in lap shear test. Experimental results show that optimised chemical etching can triple the lap shear strength compared with untreated samples. Acid concentration significantly influences joint performance; furthermore, the soaking method of etching can yield stronger bonds than the pipette method. Additionally, chemically etched CFRP–Al joints can outperform those treated with conventional sandpaper abrasion. These findings demonstrate the effectiveness of controlled chemical etching in improving the structural integrity of CFRP–metal adhesive joints and provide insights for designing more robust hybrid structures for lightweight applications.

1 | Introduction

Carbon fiber–reinforced polymers (CFRPs) saw a large growth in the industry around the 1970s and 1980s, and ever since, they have been dominating the global market. However, during the 1970s, the usage of CFRP mainly adhered to application in sport equipment [1].

Very soon, these materials found application in the aerospace industry, but initially, they were used only for secondary aircraft structures. Later, the aircraft industry started using them for building aircraft bodies around the 1990s and early 2000s. This growth and shift in application was due to the need for lighter planes with lesser fuel consumption and lower CO₂ emission. Ever since then, CFRPs have found applications also in the non-aerospace related sectors like energy, automotive, rail transport, and so on. The constant research in the field of CFRP has led

to a reduction in their cost, while increasing their strength to weight ratio [2, 3].

Many applications of CFRP often need them to be joined to metal alloys to obtain components with high strength, low weight, and reduced cost [4]. Present technologies allow various methodologies for joining CFRP with metal alloys, especially with aluminium alloys for applications in aerospace and automotive industries; among them, adhesive bonding, diffusion bonding, riveting, welding, and bolting [5, 6].

Each of these processes has advantages and disadvantages: riveting, welding, and bolting may cause stress concentrations, which aid in formation of cracks [7, 8], whereas welding—*suitable for thermoplastic matrix CFRP only*—causes several porosities at the CFRP–alloy interface, causing joint failure at the porous zones [9, 10].

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On the other hand, adhesive bonding poses several advantages over these processes since adhesives can be used at temperatures that are more compatible with the CFRP, with stress distribution being more uniform [11, 12].

Considering adhesive bonding, it is vital to focus on the different strategies to increase the bond strength to achieve maximum results and avoid bond failure under high loads. Multiple techniques exist for enhancement of the joint strength like selecting proper bond line thickness [13–15], selection of adhesives that are compatible with the adherend [16], and surface preparation [17–20].

Surface preparation techniques that are well-known in the field of joining: they allow to alter the surface energy and morphology of the specimen, thus ensuring enhanced bond strength; the main options are mechanical abrasion and chemical etching [21–23].

The objective of the present work is to enhance adhesive bond strength between CFRP joined with aluminium alloy through chemical etching using sulfuric acid solutions. Joined samples are tested in tensile mode by a lap shear test; etching effect on joint strength will be compared with what is obtained by mechanical abrasion and untreated case.

2 | Materials

CFRP plates C380T7002X2TX3-150 (Figure 1a) are produced by Microtex Composites S.r.l (Pistoia, Italy). The prepreg is made up of carbon fibers of T700 type, and the aerial weight of the fabric is 380 gsm (gram per square meter). The weave pattern of this particular model is 2 × 2 twill weave, which is known for its stability and strength. The number of layers of the weave pattern were three.

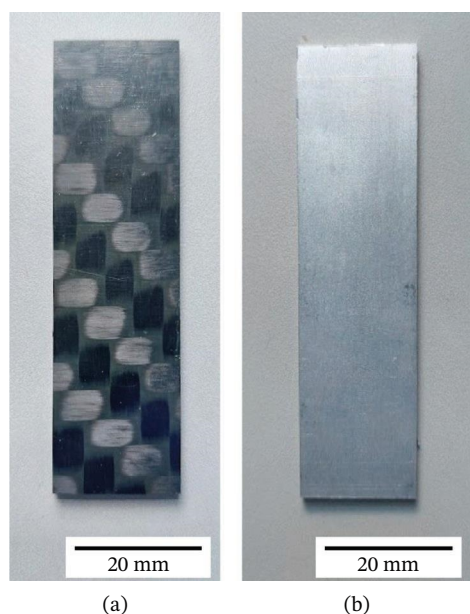


FIGURE 1 | Materials used in the study: (a) carbon fiber-reinforced polymer (CFRP)—Microtex C380T7002X2TX3-150, a twill weave carbon fabric; and (b) aluminium alloy—Al–Mg–Si 6016 T4 Grade A, commonly used in automotive applications.

The aluminium alloy Al–Mg–Si 6016 T4 Grade A (Figure 1b) belongs to the 6000 series where the primary alloying elements are aluminium, magnesium, and silicon. The tempering of this material was done by solution heat treatment and natural aging.

Both the CFRP and aluminium plates were cut into smaller slabs of 80 × 20 × 2 mm (l × w × t) using an automatic cutting machine equipped with an aluminium oxide cutting wheel (QATM, Mammelzen, Germany) for single-lap shear testing. The aluminium alloy was cleaned using distilled water, isopropyl alcohol, and heptane. The chemical etching of the CFRP slab was performed using sulfuric acid (Honeywell Fluka, United States) solution with volume concentrations of 30%, 60%, and 97%. The sulfuric acid supplied by the manufacturer had a volume concentration of 97% and was used directly for the highest concentration etching condition. The lower concentration solutions (30% and 60%) were prepared by diluting the 97% sulfuric acid with distilled water according to standard dilution equation:

$$C_1 V_1 = C_2 V_2$$

where C_1 is the initial acid concentration (97%), C_2 is target concentration (30% or 60%), V_1 is required volume of initial acid concentration, and V_2 is the final volume of the diluted solution. For all chemical etching processes, a diluted volume of 30 mL was prepared and used.

To adhesively bond the two specimens in a single-lap joint, a two-component adhesive—Betamate 2098 (DuPont, Delaware, United States) was used; this adhesive is widely used in the automotive sector for adhesively bonding aluminium parts for durability and crash performance. The adhesive is a bicomponent one, with an epoxy-based part (A) to be mixed with an amine-based one (B). The A:B mixing ratio is 2:1.

3 | Experimental Methods

3.1 | Etching Procedure

The CFRP slabs were cleaned with distilled water in an ultrasonic bath at a temperature of 40°C for 10 min to ensure removal of any dirt from the surface. The specimens were then rinsed with acetone. Following cleaning, the CFRP samples were dried using compressed air and masked with glass fiber tape to cover nontarget areas to prevent acid seepage to unwanted faces of the specimen. These steps comprise the pre-etching procedure.

Chemical etching is controlled by three key parameters, that is, time, temperature, and acid concentration: the time and temperature optimization were done through trial-and-error method, based on results in [21], where sulfuric acid etching of CFRP was examined over wide range of acid concentrations (33–96 vol.%), temperature ranging between (80°C and 150°C) and etching time between (5 and 20 min), and where excessive matrix removal was observed under overly aggressive conditions. Based on these findings, a targeted subset of etching conditions was investigated in this study to achieve controlled surface modification while avoiding composite damage. Specifically, the sulfuric acid concentration was varied (30%, 60%, and 97% by volume), whereas the etching time and temperature were maintained at fixed values to

isolate the effect of acid concentration. The complete set of etching parameters examined in this study is summarized in Table 1, providing a clear overview of the experimental conditions used.

The etching treatment was performed on a hot plate at 60°C. The acid of desired concentration was kept on the hot plate, and the CFRP area to be etched (25 × 20 mm) was either dipped in the acid, thus comprising the soaking method (Figure 2a), or 100 µL of acid was dispensed through a micropipette on the surface of the CFRP (the pipette method) (Figure 2b). In both cases, the acid remains in contact with the CFRP for 5 min.

At the end of the etching treatment, the CFRP (Figure 2c) was immediately dipped in a beaker filled with distilled water for 1 min to stop any further chemical reaction. Then, the glass fiber tape was removed, and the CFRP was submerged in fresh distilled water in an ultrasonic bath for 10 min at 40°C. The specimen was then dried in an oven at 80°C for 1 h to remove excess humidity on or inside the CFRP specimen. These steps comprise the postetching procedure.

3.2 | Joining and Mechanical Testing

The joint area (same as the etched area) was adhesively bonded between the CFRP and aluminium specimen in a single-lap

TABLE 1 | Summary of sulfuric acid etching parameters investigated in the present study for CFRP.

H ₂ SO ₄ concentration (vol.%)	Temperature (°C)	Etching time (min)
30	60	5
60	60	5
97	60	5

joint. A copper weight of 300 g was put on the joined area to prevent the samples from moving during the curing process and to prevent any gaps or bubbles in the adhesive or between the adhesive and the specimen while also ensuring same pressure during curing. The weight also provides a uniform bond-line thickness. A spacer was kept below the higher specimen in the lap joint to avoid sliding of CFRP and aluminium specimen against each other. After this, the samples were cured in the oven at 60°C for 2 h in air.

Successive to the curing period, the specimens were tested for shear load through lap shear test under tensile mode (Figure 3a) using universal tensile/compression machine Z050 (ZwickRoell S.r.l., Genova, Italy). Tabs of the same thickness as the specific substrate were glued in the clamped region so that the joint would be in the centerline of the applied load, as represented in Figure 3b. A test speed of 2 mm/min was used for each test. The tensile strength was calculated dividing the max load at failure by the overall target area (25 × 20 mm = 500 mm²), adapted from DIN EN1465 standards. Joint cross sections were observed by benchtop scanning electron microscope (JCM-6000Plus, JEOL Ltd, Tokyo, Japan) to analyze the joined specimen.

A comparison was made among different chemically etched specimens, mechanically abraded specimens, sanded using sandpaper grit P320, and a base case of untreated specimens. This comparison of joint strength helps assess the benefit of chemical etching, and its advantage over mechanical abrasion since sandpaper treatment in particular is known to produce higher shear strength for single-lap joints, especially when abraded in random direction [23]. In total, eight different cases were mechanically tested and compared. Each test was repeated twice.

4 | Results and Discussion

The Figure 4 below shows the lap shear test results for single-joints created between CFRP–Al specimen for different

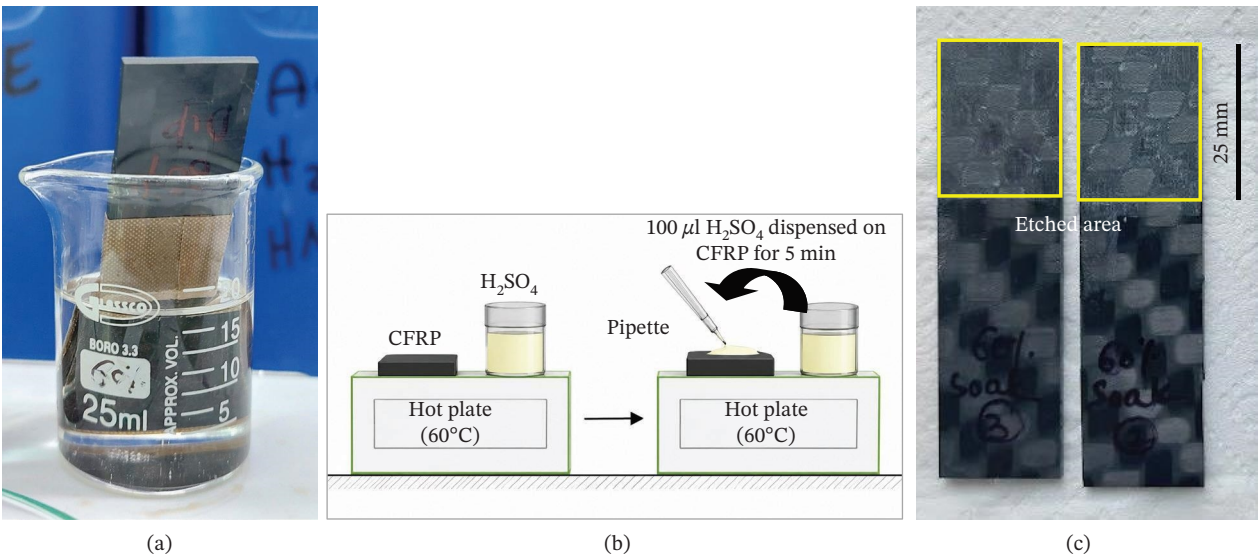


FIGURE 2 | Chemical etching methods and resulting surface morphology of CFRP: (a) Soaking method, where the specimen target area is fully immersed in the sulfuric acid; (b) pipette method, involving localized application of the etchant using a pipette; and (c) representative image showing the etched area on CFRP.

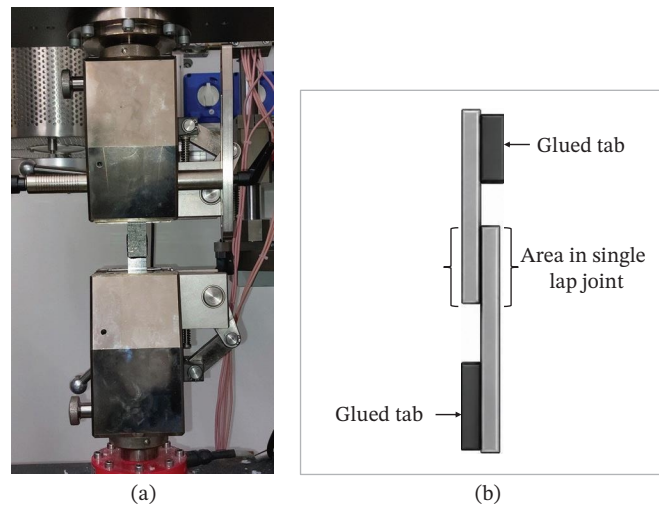


FIGURE 3 | Mechanical testing setup: (a) Lap shear test conducted under tensile mode to evaluate joint strength; and (b) application of tabs to ensure uniform load distribution along the centerline of the bonded joint.

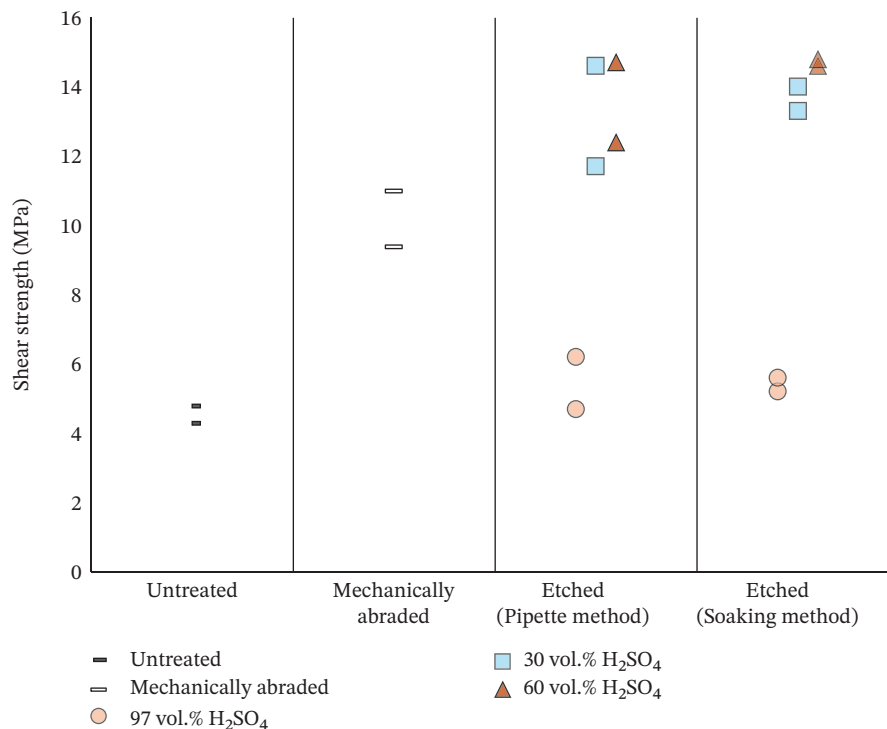


FIGURE 4 | Lap shear strength of CFRP–Al joints for different surface treatments. Untreated and mechanically abraded specimens are compared with chemically etched specimens prepared using pipette and soaking methods are shown for sulfuric acid concentrations of 97%, 60%, and 30%, distinguished by marker style. Each data point represents an individual mechanical test.

treatment conditions of CFRP, namely—untreated, mechanically abraded, and chemically etched.

To facilitate a direct numerical comparison of different surface treatments, the lap shear strength values obtained for each condition have been summarized in Table 2.

As summarized in Table 2, chemically etched specimens, particularly those treated using the soaking method at 60vol.% acid concentrations, exhibited higher lap shear strengths. The

corresponding fracture surfaces below (Figure 5) elucidate the underlying failure mechanisms.

Two different chemical etching approaches were implemented—soaking and pipette method. Both the methods result in a higher lap shear strength compared with the untreated CFRP–Al joints, as shown by the individual test results in Figure 4 and summarized numerically in Table 2. Among all the examined conditions, the highest lap shear strength was obtained for specimens etched using the soaking

TABLE 2 | Summary of lap shear strength values for CFRP–Al joints subjected to different surface treatments. For each condition, individual lap shear strength values obtained from duplicate mechanical tests are reported. Acid concentration is given for chemically etched specimens.

Treatment	Acid conc. (vol.%)	Lap shear strength (MPa)	
		Repetition 1	Repetition 2
Untreated	—	4.3	4.8
Mechanically abraded	—	9.4	11
Etched (pipette method)	30%	11.7	14.6
Etched (pipette method)	60%	12.4	14.7
Etched (pipette method)	97%	4.8	6.2
Etched (soaking method)	30%	14	13.3
Etched (soaking method)	60%	14.6	14.8
Etched (soaking method)	97%	5.2	5.6

method with sulfuric acid solution concentration of 60 vol.%, yielding values of 14.8 and 14.6 MPa. These closely clustered results indicate good repeatability and are attributed to more uniform etching achieved when the CFRP is fully immersed in the acid during the soaking process—leading to cohesive failure (Figure 5h).

The pipette method at the same acid concentration (60 vol.%) also produced relatively higher lap shear strengths (14.7 and 12.4 MPa); however, a wider spread between the repeated tests was observed. This variability is attributed to the nonuniform spread of acid on the CFRP surface during the pipette method of chemical etching—localized acid accumulation or acid spillage occurs, leading to a mixed failure (Figure 5g). At lower acid concentrations (30 vol.%), the pipette method shows a pronounced scatter in lap shear strength values (14.6 and 11.7 MPa), suggesting inconsistent surface modification under these conditions. Its respective fracture surface is shown in Figure 5f. At the same acid concentration, the soaking method produces significantly more consistent results, with variability up to four times lower than that of the pipette method (Figure 4; Table 2). The failure mode remains unchanged (Figure 5e). In contrast, the pipette method shows higher variability across all tested concentrations, which is attributed to the nonuniform distribution of acid on the CFRP surface due to localized accumulation.

Mechanical abrasion of CFRP using sandpaper led to substantial improvement in lap shear strength compared with the untreated condition, with values of 11 and 9.4 MPa, representing more than a twofold increase compared with the untreated case (4.8 and 4.3 MPa). Nevertheless, mechanically abraded specimens do not achieve the lap shear strengths obtained for chemically etched specimens at 60 vol.% acid concentration. The comparatively lower performance of the mechanically abraded joints is consistent with the fracture surface observed in Figure 5d, where adhesive failure along with some delamination of CFRP is observed.

Lower lap shear strengths were also recorded for both pipette and soaking etching methods at the highest acid concentration (97 vol.%) with values of 6.2 and 4.8 MPa for pipette method and 5.6 and 5.2 MPa for soaking method. The corresponding fracture surfaces (Figure 5b,c) reveal fiber detachment from the CFRP owing to aggressive acid attack, which weakens the CFRP surface and adversely affects the joint strength.

The relatively low lap shear strength obtained for the untreated CFRP–Al joints in the present study is consistent with the behavior reported in [21], where untreated CFRP joints exhibited limited load-bearing capacity due to poor interfacial interaction between the adhesive and the composite surface (Figure 5a). In that work, controlled sulfuric acid etching was shown to significantly enhance joint strength by selectively removing the polymer matrix and promoting adhesive infiltration around the carbon fibers, resulting in up to a two-fold increase in lap shear strength compared with untreated specimens.

Although the absolute lap shear strength values differ between the two studies due to differences in adherend materials, adhesive systems, and joint configurations, the overall trend observed here—namely, the inferior performance of untreated CFRP joints and the substantial improvement achieved through appropriately controlled chemical etching—is in good qualitative agreement with [21]. This agreement further supports the conclusion that surface modification of CFRP plays a critical role in enhancing adhesive joint performance.

Further insight into the observed lap shear strength trends is provided by the SEM images of the adhesively bonded CFRP–Al cross-sections shown in Figure 6. The SEM images are shown for the untreated CFRP specimen (Figure 6c), the soaking-etched CFRP specimen at 60 vol.% H_2SO_4 , which yielded the highest lap shear strength (Figure 6a), and the soaking-etched CFRP specimen at 97 vol.% H_2SO_4 , which resulted in one of the lowest shear strengths among the etched conditions (Figure 6b).

For the specimens etched at 97 vol.% H_2SO_4 by soaking method, the reduced lap shear strength is associated with the presence of microcracks within the etched surface region of the CFRP. These microcracks indicate excessive matrix degradation caused by the aggressive acid concentration. Moreover, the adhesive was observed to be unable to effectively infiltrate these microcracked regions during the bonding process (Figure 6a), suggesting that the surface fibers were insufficiently supported by the surrounding matrix. As a consequence, the joint exhibited

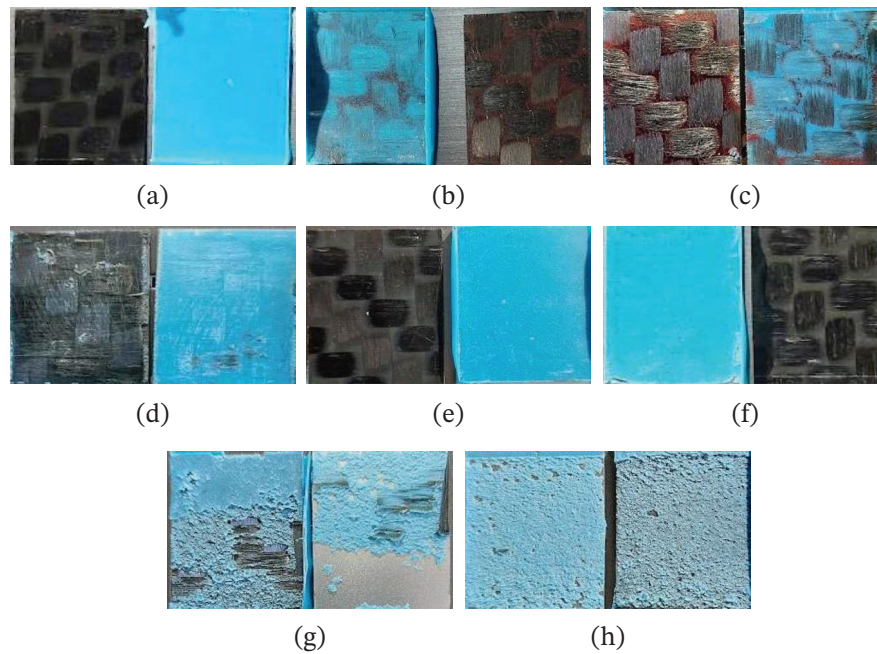


FIGURE 5 | Comparison of fracture surfaces after mechanical test: (a) Untreated, showing adhesive failure; (b) soaking etch: 60°C-5 min-97% acid concentration, showing fiber-tear-type failure; (c) pipette etch: 60°C-5 min-97% acid concentration, showing fiber-tear-type failure; (d) mechanical abrasion: sandpaper P320, showing adhesive failure with some delamination on CFRP; (e) soaking etch: 60°C-5 min-30% acid concentration, showing adhesive failure; (f) pipette etch: 60°C-5 min-30% acid concentration, showing adhesive failure; (g) pipette etch: 60°C-5 min-60% acid concentration, showing mixed failure; and (h) soaking etch: 60°C-5 min-60% acid concentration, showing cohesive failure.

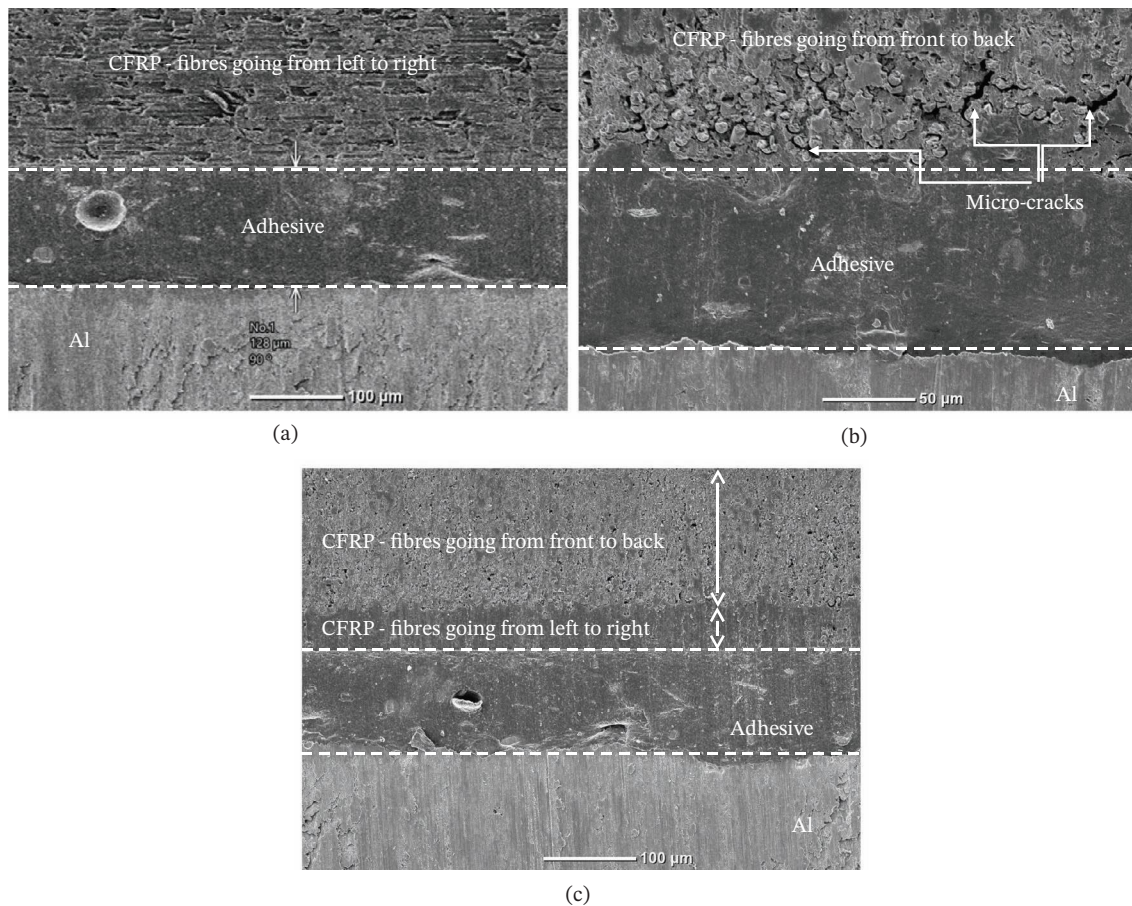


FIGURE 6 | Cross-sectional view of the bonded joint: (a) Etching by soaking: 60°C-5 min-60% acid concentration shows absence of cracks in CFRP or gap in CFRP-adhesive-aluminium junction; (b) etching by soaking: 60°C-5 min-97% acid concentration shows microcracks in the CFRP; and (c) untreated shows absence of cracks in CFRP or gap in CFRP-adhesive-aluminium junction.

fiber-tear-type failure within the CFRP, as also evidenced in the corresponding fracture surface (Figure 5b), ultimately leading to reduced joint strength.

In contrast, CFRP specimen etched by the soaking method at 60 vol.% H_2SO_4 (Figure 6a) showed no visible microcracking or surface damage. The CFRP–adhesive–aluminium interface appeared continuous and free from gaps or voids, indicating effective adhesive wetting and infiltration. This uniform interfacial morphology is consistent with the high lap shear strengths measured for this condition and confirms that controlled chemical etching at this concentration promotes an optimal balance between surface activation and structural integrity of the CFRP.

The untreated CFRP specimen (Figure 6c) similarly showed an interface free of obvious cracks or voids; however, it produced the lowest lap shear strength among all conditions examined. This behavior is attributed to the absence of surface activation and limited removal of surface contaminants, resulting in poor interfacial bonding between the CFRP and adhesive. This interpretation is further supported by the fracture surface analysis, where the untreated joints predominantly exhibited adhesive failure (Figure 5a), whereas the specimens etched by soaking at 60 vol.% H_2SO_4 displayed cohesive failure, indicative of a stronger and more effective joint. These fractographic observations provide possible microstructural evidence for the superior joint performance obtained through controlled chemical etching.

The present results should be interpreted in the broader context of surface preparation strategies reported for CFRP–metal bonding. As shown in the literature, CFRP surfaces inherently exhibit a polymer-rich top layer and contamination from release agents, which adversely affect adhesion and necessitate appropriate surface pretreatment prior to bonding [24]. Consequently, most studies have focused on mechanical (e.g., grinding and grit blasting), peel-ply, plasma, and laser-based treatments to remove this weak boundary layer and enhance surface activation [24]. In contrast, conventional chemical treatments such as acid etching are predominantly applied to metallic substrates, where they modify oxide layers, increase surface roughness, and promote adhesion through improved wettability and mechanical interlocking [25]. For example, sulfuric acid etching of steel has been shown to generate porous surface morphologies that enhance adhesive penetration and increase lap shear strength compared with untreated or mechanically treated surfaces [25]. Similarly, acid pickling of titanium alloys produces roughened surfaces that facilitate mechanical interlocking, although its effectiveness depends strongly on surface morphology and subsequent treatments [26]. However, in these studies, chemical etching is applied exclusively to the metallic adherend, whereas CFRP surfaces are typically treated by mechanical or physical methods (e.g., grinding or plasma), highlighting the limited application of acid-based treatments directly to CFRP substrates [26].

This distinction arises from the fundamentally different nature of CFRP surfaces. Unlike metals, CFRPs consist of a heterogeneous fiber–matrix system, where excessive or uncontrolled

chemical treatment may degrade the polymer matrix or lead to nonuniform surface modification. As a result, the literature on CFRP surface preparation has largely avoided aggressive chemical etching and instead prioritised controlled removal of the matrix layer and surface activation through mechanical or plasma-based approaches [27].

In this context, the present study extends the existing body of work by demonstrating that sulfuric acid–assisted etching can be effectively applied to CFRP surfaces to enhance CFRP–metal joint performance. The observed improvements in lap shear strength and the sensitivity to process parameters (e.g., acid concentration and application method) indicate that controlled chemical etching can modify CFRP surface morphology in a manner analogous to metallic substrates, while maintaining structural integrity. Importantly, the reduced variability associated with the soaking method further highlights the role of uniform surface modification in achieving reliable bonding. Therefore, although acid etching is well established for metals, its systematic application to CFRP surfaces—particularly in the context of CFRP–metal adhesive joints—remains relatively underexplored, and the present findings provide new insights into its potential as a viable surface preparation strategy.

5 | Conclusions

Chemical etching using sulfuric acid solution is effective in increasing the shear strength (lap shear) between these CFRP and aluminium alloy slabs for up to triple the shear strength of untreated counterparts. The chemical etching process can be implemented through two different techniques, that is, through pipette or soaking method. Both these methods *can produce as high as approximately three times the bond strength achieved by untreated CFRP*. However, the soaking method is preferred since it produces uniformly etched specimen with closely clustered lap shear strength values, indicative of a consistent joint performance. The three etching parameters—time, temperature, and acid concentration—are fundamental to the process, and it is imperative to optimize the acid concentration to achieve the best results. Extremely high acid concentration like 97% volume concentration of sulfuric acid forms microcracks in the CFRP, causing weak joints and lower shear strength. Parameter optimization can lead to higher shear strength than for mechanical abrasion (sanding). The best results with the highest shear strength of CFRP–Al joint were obtained at etching parameters kept fixed at 5 min, 60°C, and 60% acid concentration for both soaking and pipette method. However, the lowest shear strength produced by chemical etching (60°C–5 min–97 vol.% H_2SO_4 concentration; soaking method) is still higher than the untreated specimen approximately by 16%–20%.

Overall, the results demonstrate that controlled sulfuric acid etching—particularly when applied via the soaking method—can provide an effective and reproducible approach for enhancing CFRP–Al adhesive joint strength. These findings highlight the importance of etching parameter optimization and support the use of chemical etching as a viable alternative to conventional mechanical surface preparation methods for high-performance composite-metal joints.

Author Contributions

Koshika Pandey: validation, formal analysis, investigation, writing – original draft, visualization. **Alessandro Benelli:** supervision, project administration, writing – review & editing. **Monica Ferraris:** supervision, project administration, funding acquisition, writing – review & editing, methodology.

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Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The data that support the findings of this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

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