



NAZARBAYEV
UNIVERSITY

School of Engineering and Digital Sciences

Bachelor of Engineering in
Mechanical and Aerospace Engineering

**Eco-Friendly Manufacturing Process for
Lightweight High-Performance Fiber
Metal Laminates (FML)**

(Final Report for Capstone Project)

by

Danial Zhumashev
Anuar Myrzamuratov
Alikhan Madali

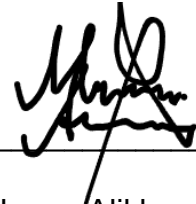
Principal Supervisor: Associate Prof. Sherif Araby
Gouda

Co-supervisor: Dr. Umut Bakhbergen

April 2026

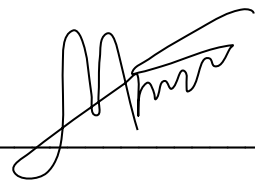
Declaration

We hereby declare that this report, entitled "Eco-Friendly Manufacturing Process for Lightweight and High-Performance Fiber Metal Laminates (FML)" is the result of our own project work except for quotations and citations that have been duly acknowledged. We also declare that it has not been previously or concurrently submitted for any other degree at Nazarbayev University.



Name: Alikhan Madali

Date: April 30, 2026



Name: Anuar Myrzamuratov

Date: April 30, 2026



Name: Danial Zhumashev

Date: April 30, 2026

Acknowledgements

We would like to thank our supervisors, Associate Prof. Sherif Araby and Dr. Umut Bakhbergen, for their support and guidance throughout the semester. Additionally, we would like to thank graduate students Sadyr Sabitov, Madina Moldabayeva, and Gaukhar Mukhambetkaliyeva for their advice and shared experiences.

Abstract

Fiber Metal Laminates (FML) is a hybrid material that consists of alternating fiber-reinforced polymer (FRP) and metal layers. Currently, FML is being used frequently in industry fields such as aerospace and automotive engineering. The combination of excellent strength, stiffness, and fatigue resistance of FRP and outstanding impact absorption of metals makes it an efficient replacement for monolithic metals. The interfacial adhesion between the dissimilar metal and composite layer is the critical factor in structural integrity of FMLs due to high risk of delamination. In order to ensure strong interface bonding, pre-treatment of the metal surface has become an essential part of the manufacturing process. Currently, the surface treatment procedures of FML involve the use of highly toxic acids, such as sulphuric and nitric acids. As environmental concerns rise, there is a need to make the FML production process more sustainable without compromising structural integrity. This study proposes: 1) Finding more sustainable FML manufacturing methods; 2) Quantifying a structure-property relationship between metal surface and interfacial adhesion; 3) A comprehensive mechanical testing and fracture analysis of laminates. We developed a novel, less toxic potassium chloride (KCl) anodizing surface treatment method that resulted in a 64% improvement in the flexural strength of FML compared with conventional acidic treatments.

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Engineering Ethics and Standards

All experiments and tests in this report were carried out closely following engineering ethics principles and applicable testing standards.

Engineering ethics

- All experiments were conducted using proper Personal Protective Equipment (PPE) and safe laboratory practices to protect students.
- Hazardous chemical use and waste generation were minimized to reduce environmental impact.
- Experimental results and sustainability claims were reported honestly without exaggeration.

ASTM International standards

The following ASTM International standards were followed for mechanical and durability testing done in this project:

- ASTM D3039 – Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials.
- ASTM D790 – Standard Test Method for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials.
- ASTM D1002 – Standard Test Method for Apparent Shear Strength of Single-Lap-Joint Adhesively Bonded Metal Specimens by Tension Loading.
- ASTM D3330 – Standard Test Method for Peel Adhesion of Pressure-Sensitive Tape.
- ASTM B117 – Standard Practice for Operating Salt Spray Apparatus.

1 Introduction and Problem Statement

From the very beginning of human civilization, developments in engineering have been closely associated with the discovery of new materials. Their inherent bulk properties limit the functionality of the engineering materials. Currently, to improve energy efficiency, industries such as aerospace demand materials that are both lightweight and strong [1]. To address this problem, researchers have developed a new class of materials called composites, which combine several materials with different properties. Fiber Metal Laminates (FML) is a sandwich structure composite consisting of Fiber Reinforced Polymers (FRP) and metal sheet layers. FMLs unite the excellent fracture and fatigue resistance of composites with the outstanding impact absorption of metal alloys. Hence, the combination of two distinct materials addresses their separate limitations such as low fatigue resistance of metals and energy absorption of polymer composites. The lightweight structure, superior fatigue resistance, and corrosion resistance make FMLs an efficient replacement for monolithic metals in the aerospace and automotive industries [2]. For example, global car manufacturer BMW uses FML to manufacture chassis parts under high load, as shown in Figure 1(b) [3]. Various classifications of FMLs are based on the type of metal, fiber, matrix, and stacking sequence. Synthetic fibers are common reinforcing materials for FMLs due to their high strength, low weight, and corrosion resistance. They include CARALL, GLARE, and ARALL, fabricated using carbon, carbon and aramid fibers, respectively [4].

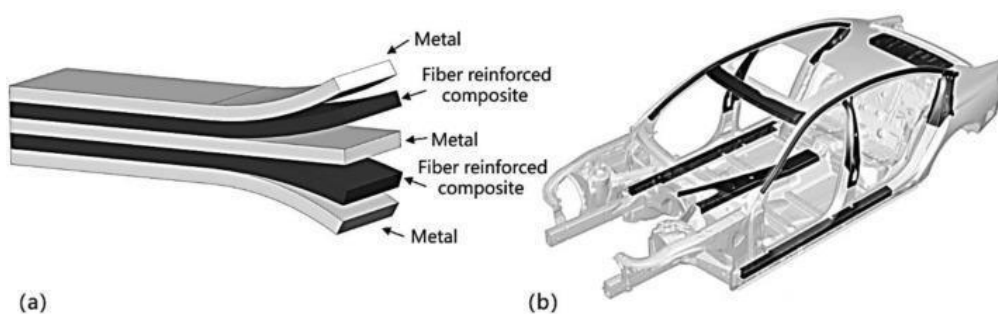


Figure 1. Schematic diagram of FMLs; (b) Application of FMLs
in BMW 7 series sedan [3]

Despite outstanding structural integrity, the manufacturing process of two joint materials with different mechanical properties poses several challenges. It is important to ensure strong interfacial adhesion between the two dissimilar materials. There are two types of interfaces present in FMLs: fiber-polymer and metal-polymer interfaces. The latter is considered to be a weaker interface. The adhesion at the metal-polymer interface directly affects the stress transfer efficiency between metal and fiber layers [5]. Therefore, the failure mode and overall performance of the FML is highly dependent on it. It can be stated that ensuring a strong metal-polymer interface is the most critical aspect of manufacturing high-performance FMLs. However, it is difficult to achieve high adhesion between polymer and metal due to their substantial difference in mechanical properties [6]. In order to address this issue, researchers have developed numerous adhesion theories that are based on polymer science, fracture mechanics, material mechanics, rheology and other disciplines.

Currently, there are seven widely recognized interfacial adhesion theories which include mechanical interlocking, physical adsorption, chemical bonding, diffusion, electrostatic theories, rheological model and weak boundary layer theories. Even though each of these theories explain the adhesion phenomenon to a certain extent, there is no single unified theory that explains all the underlying mechanisms between adhesive and a substrate [7]. However, we already possess some basic understanding of why layers stick to each other and why they sometimes debond.

Mechanical interlocking is considered to be the oldest and the most well-known adhesion theory. It states that bonding occurs when adhesives penetrate into the high amount of pores on a rough surface [8]. Since no surface is perfectly smooth, these microscopic pores increase the contact area between metal and polymer. After the polymer resin in pores is cured, layers are mechanically interlocked. Therefore, surface roughness and resin wettability plays an important role in achieving reliable adhesion. The rougher the surface, the more contact surface for a resin to infiltrate. The porosity and roughness of the metal can be usually

enriched through mechanical or chemical treatments. It is also very important for a resin to have high wettability and low viscosity to penetrate into microscopic pores [9].

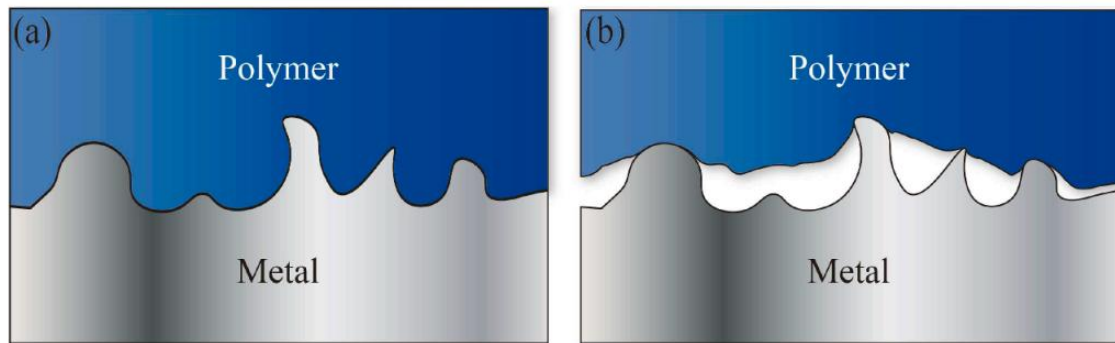


Figure 2. Illustration of metal-polymer interface with (a) good wetting and (b) poor wetting

In order to increase the surface wettability, metal sheets are chemically treated to create pores in micro and nano length scales. Initially, the surface is cleaned with ethanol or acetone to remove all the contaminants. Then, one of two chemical treatments are commonly performed; etching and anodizing. Etching is a process of removing native aluminum layers by using acidic solutions. As a result, the original aluminium oxide layer is removed and roughness is increased [10]. In comparison, anodizing is an electrochemical process where aluminum acts as an anode in an electrolytic cell. Anodizing creates an extremely thin porous oxide layer. Similar to etching, highly toxic chemicals such as nitric, phosphoric and sulphuric acids are utilized during anodizing [11-12].

Even though FMLs with chemically modifying metal surfaces have higher metal-polymer adhesion, the usage of toxic acids raises environmental sustainability concerns. For example, main problems include their energy-intensive production, acidic wastewater generation and air emission [13]. Therefore, it is crucial to find alternative manufacturing methods that use eco-friendly chemicals without compromising the structural integrity of the FMLs.

In this project, we propose using Potassium Chloride (KCl) solution for anodization process of FML manufacturing. KCl is considerably less toxic than conventional acids such as HCl, S_2HO_4 , P_2HO_4 . There is no present literature on utilization of KCl anodizing for the epoxy based FMLs. Moreover, comprehensive mechanical testing needs to be conducted in order to verify the structural integrity of the proposed laminates. Even though FML utilized structures (planes, cars) frequently operate in environmentally harsh conditions, there is a lack of studies that investigate the durability of laminates. The proposed methodology has the potential to reduce the environmental harm, production costs and workplace hazards of the manufacturing process.

2 Background

Fiber Metal Laminates (FML) are hybrid composite structures with alternating layers of fiber-reinforced polymer and thin metal sheets, which mostly is aluminum or titanium alloys [14]. The most widely studied FMLs are named by their reinforcement type: CARALL uses carbon fibers, GLARE uses glass fibers, and ARALL uses aramid fibers, all bonded with aluminum sheets [15]. Also, FMLs are commonly divided by their layer configuration, like 2/1, 3/2, and 4/3 stacking sequences, and by their fiber orientation, like unidirectional and cross-ply woven [15]. These classifications have their own advantages and disadvantages, marking their applications. GLARE panels are light and improve fatigue resistance and, therefore, are used in fuselage of Airbus A380, where it achieves between 15 and 30% weight reduction. CARALL has outstanding fatigue fracture resistance and high energy absorption ability, which makes it ideal for structural aerospace applications and helicopter struts [15].

There is a large volume of published studies describing the role of chemical surface treatment on interfacial adhesion of FMLs. For example, Zhu et al [16] has conducted a study comparing the shear strength of chemically anodized FMLs with traditional sand-blasting treated one. According to the results samples with phosphoric acid anodization have demonstrated the shear strength of 51.2 MPa while the reference was 38 MPa. The key difference observed was the size scales of the pores; anodizing has created pores at the nanoscale increasing the surface area. A comprehensive study on effects of various aluminum surface treatments on the basalt FMLs interlaminar adhesion was conducted by H. Aghamohammadi et al. [17] The authors tested the flexural strength of composites with four different surface treatments including mechanical abrasion, alkaline treatment, Forest Products Laboratory etching (FPL-etching) and sulphuric acid anodization (SAA). Moreover, the SAA was conducted at three different time conditions; anodizing for 10, 20 and 30 minutes respectively. Among all methods and conditions, FML with metal sheets anodized for 10 minutes have shown the highest flexural strength of 623 MPa. FPL-etched, SAA

20 min and 30 min have demonstrated the flexural strength of 586, 554, and 547 MPa respectively. On the other hand, composites with mechanical and alkaline treatments were not as adhesively strong with the flexural strength of 396 MPa and 489 MPa.

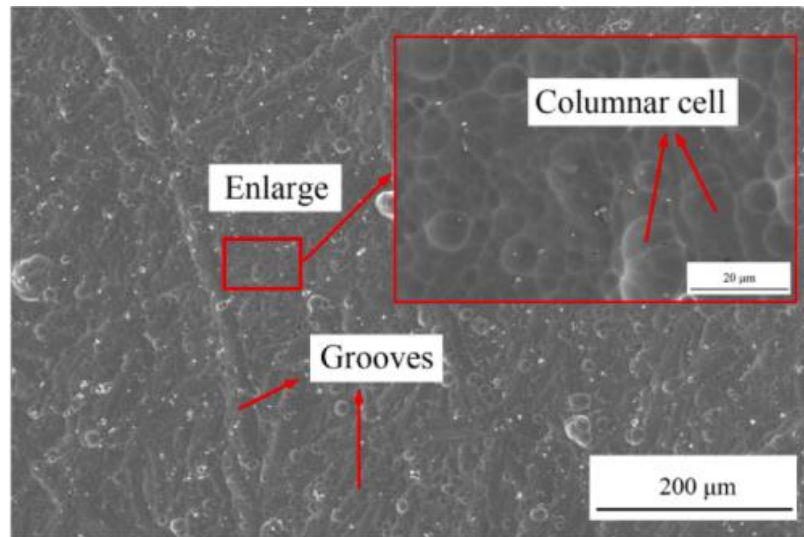


Figure 3. Metal surface of mechanical abrasion.

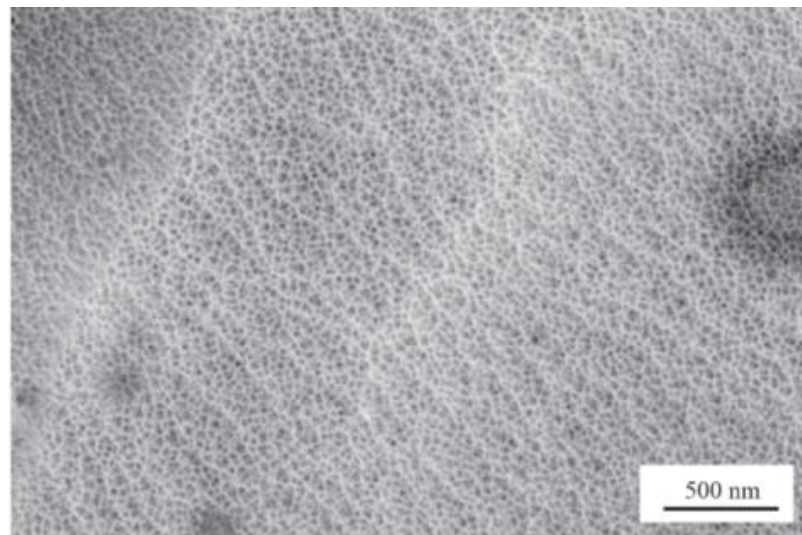


Figure 4. Metal surface of sulphuric anodizing.

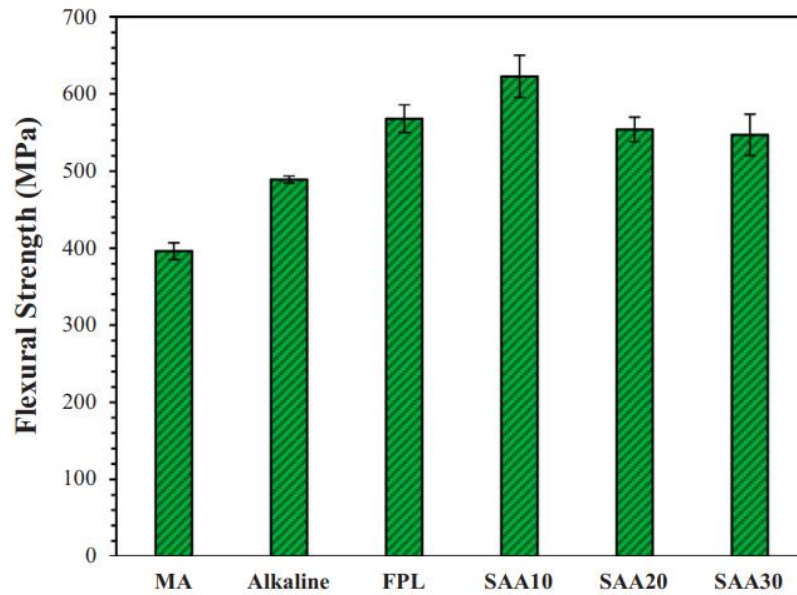


Figure 5. Flexural strength comparison for different surface treatments.

The research on aluminum surface treatment identifies more than 40 mechanical, chemical, and electrochemical methods designed to enhance adhesion at the interface between metal and polymer [18]. Mechanical treatments, like grit blasting, increase macro roughness, but show inconsistency in durability, especially under high humidity and temperature conditions [18]. On the other hand, chemical treatments, such as FPL etching, affect the surface more reliably, but these surfaces remain open to contamination and hydration, which limits long-term durability [19]. Comparative studies have highlighted that electrochemical treatments consistently outperform mechanical and chemical treatments, with phosphoric acid anodizing producing the most durable bonds in the majority of peel tests and crack propagation tests [18]. This is because the key parameters that affect the adhesion are wettability, micro-roughness, oxide layer stability, and contamination level, all of which are covered well with anodizing. Anodizing, an example of electrochemical treatment, produces a stable and porous nano-scale oxide layer which successfully resists moisture displacement at the bondline and also increases the surface area for mechanical interlocking [18]. These findings highlight the importance of developing alternative anodizing methods with similar oxide morphologies without the

environmental and safety concerns that are highly associated with the conventional acidic solutions.

In the recent study conducted by Bakhbergen et al. (2025) [19], shear strength of KCl anodized polyurethane bonded metal joints. The authors compared the result with traditional sulphuric and phosphoric anodizing methods. It was found that KCl anodized lap shear joints improved the shear strength by 66.7% and 75% in comparison to SAA and PAA.

Nanoparticle reinforcement has become one of the most efficient methods of improving the structural integrity of composites. Many studies have been conducted on this matter. A comprehensive quasi-static evaluation of multi-walled carbon nanotubes (MWCNTs) reinforced hybrid FMLs was conducted. The tensile strength, stiffness, and toughness were improved the most at 0.5wt% MWCNT by 27.2%, 33.7% and 22.1% respectively [17]. Meanwhile, Bigdilou et al. [21] have examined the effect of CNTs on the impact properties of Kevlar-ultrahigh molecular weight polyethylene fibers hybrid composites; samples with 0.1wt% CNT added have absorbed 6.5 times more energy than neat ones. Prasad et al [22] have assessed the impact of nano alumina reinforcement on the fatigue life of FML by conducting tension-tension low-cycle tests at different stress levels. The results have demonstrated that the fatigue life of FML with 0.1wt% nano alumina increased by 46%, 45%, and 94% at a stress level of 50%, 60%, and 70% of ultimate tensile strength (UTS), respectively.

3 Materials and Methods

3.1 Design of experiments

After a careful literature review, the list of experiments that would validate our research outcomes were established. The experimental plan consists of two stages; first stage is fabrication and testing of lap shear and T-peel joints while the second one is about manufacturing of FMLs. The purpose of the initial stage is to test different conditions of KCl anodizing and implementing the condition with the highest adhesion to FML manufacturing process.

The methodology of the first stage includes the fabrication of lap shear joints with epoxy followed by single-lap shear and peeling tests. The metal sheets were characterized by surface morphology and topography. The comprehensive design of experiments confirms that all the process parameters and their effects on the results are systematically quantified. For the lap shear and peel test experiments, two variables were selected on three levels; molar concentration of KCL solution (0.05M, 0.1M, 0.2M) and time duration of anodizing (5 min, 10 min, 20 min). The voltage value was held constant at 12 V during all the experiments. The second phase of the experiments is centered around the fabrication and mechanical testing of FMLs. Only the best condition from the first phase is chosen. Structural integrity of laminated structures were evaluated by tensile and flexural tests. After each stage, the fracture analysis was performed on tested samples to understand the underlying material mechanics. In addition, the flexural test was repeated for both HCl etched and KCl anodized FMLs after the durability test.

3.2 Materials and equipment

3.1.1 Materials

1. Aluminium - Al2024 T3 with a thickness of 0.5 mm;
2. Epoxy resin KER 215;
3. Jeffamine D230 hardener;
4. Potassium chloride (KCl) (ACS reagent, 99.0–100.5 %);
5. Nitric acid (Sigma-Aldrich, ACS reagent, 95.0–98.0 %);
6. Woven carbon fiber fabric;
7. Sodium Hydroxide (ACS reagent, 98 %).



Figure 6. Al2024-T3 aluminum sheets cut for anodizing.

3.1.2 Equipment

1. Electronic Weights;
2. Mixer;
3. Personal Computer;
4. Guillotine;

5. Vacuum Assisted Resin Transfer Moulding Machine Composite Integration;



Figure 7. Vacuum assisted resin transfer machine.

6. DC Power Supply;



Figure 8. DC Power supply.

7. Universal testing machine (HST WDW, 100 kN);



Figure 9. Universal testing machine (HST WDW, 100 kN).

8. Waterjet (Wazer);



Figure 10. Waterjet (Wazer).

9. Salt Spray Chamber (S-750, Xi'an LIB Environmental Simulation Industry);



Figure 11. Salt spray test chamber.

10. Contact Profilometer (Dektak XT, Bruker);



Figure 12. Contact profilometer (Dektak XT, Bruker).

11. Scanning Electron Microscope (SEM) - Crossbeam 540 (Carl Zeiss);

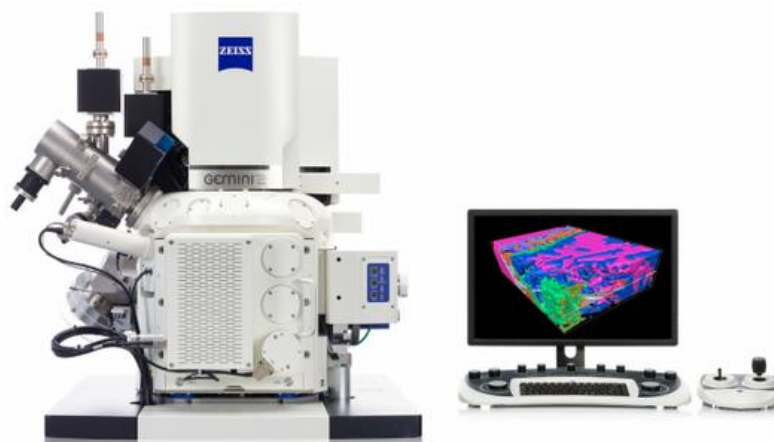


Figure 13. Scanning electron microscope (Carl Zeiss).

12. Attenuated Total Reflectance Fourier Transform Infrared (ATR-FTIR) Spectrometer - Alpha II (Bruker).



Figure 14. Fourier transform infrared spectrometer (Alpha II, Bruker).

3.3 Surface treatment and composite preparation

The full schematics of the anodizing process for test specimens can be seen in Figure 16. Initially, aluminium sheets were cleaned with ethanol to remove grease, dirt and other contaminants (Figure 16 (a)). Then, they were soaked in 5 wt% NaOH solution for 2 minutes to remove residual organics deposits. For the anodizing process, KCl solution was prepared at three different molar concentrations: 0.05 M, 0.1 M, 0.2 M; and time was set at different frames: 5 minutes, 10 minutes and 20 minutes; with constant voltage of 12 V. The solution was continuously stirred during the process to avoid precipitate formation. Two metal sheets were soaked into the solution acting as an anode and cathode.

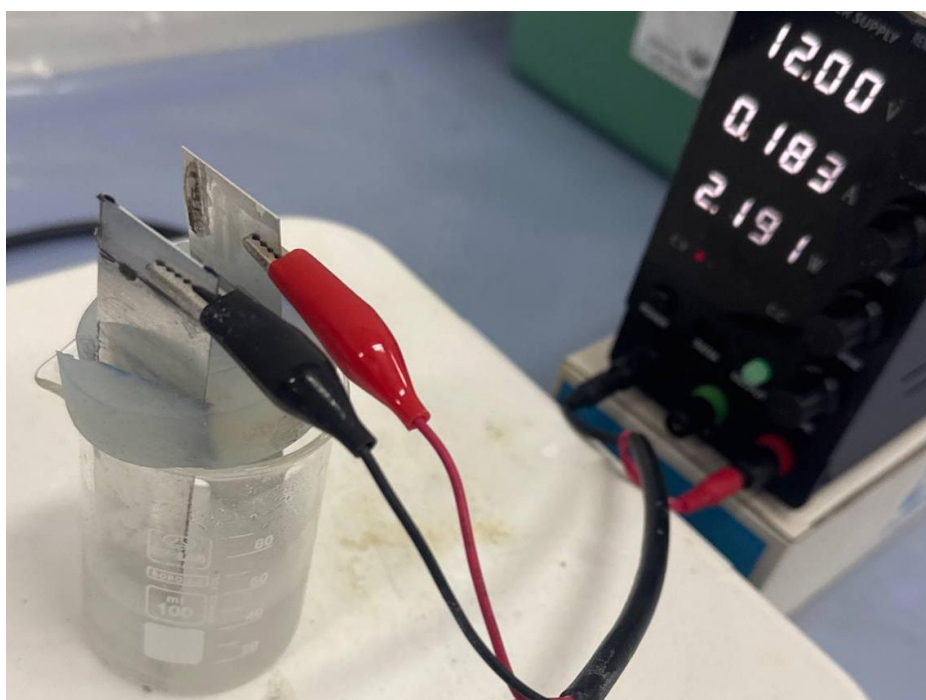


Figure 15. Photograph of the anodizing setup. Two Al2024-T3 strips connected as anode and cathode in KCl solution at 12 V DC.

The distance between them was no less than 30 mm at all times. After the anodizing, the metal sheet was immediately wiped with ethanol, followed by ultrasonication for 30 min in distilled water to stop all the remaining chemical

reactions. Finally, the sheets were dried at 60 degrees Celsius for 3 hours in a vacuum oven.

The size of the aluminum sheets and testing conditions were determined in accordance with ASTM D1002 and ASTM D3330 standards (Figure 16 (e)). Specifically, sample size was 120 mm × 25 mm. The epoxy was mixed with hardener with a ratio of 3.6:1 until uniform distribution. Then, mixed epoxy was applied to a 12.5 mm × 25 mm tip region of both metals. Joints were gently clamped together and left to cure for 3 days. The detailed illustration where epoxy applied for each type of tests is shown in Figure 16 (d).

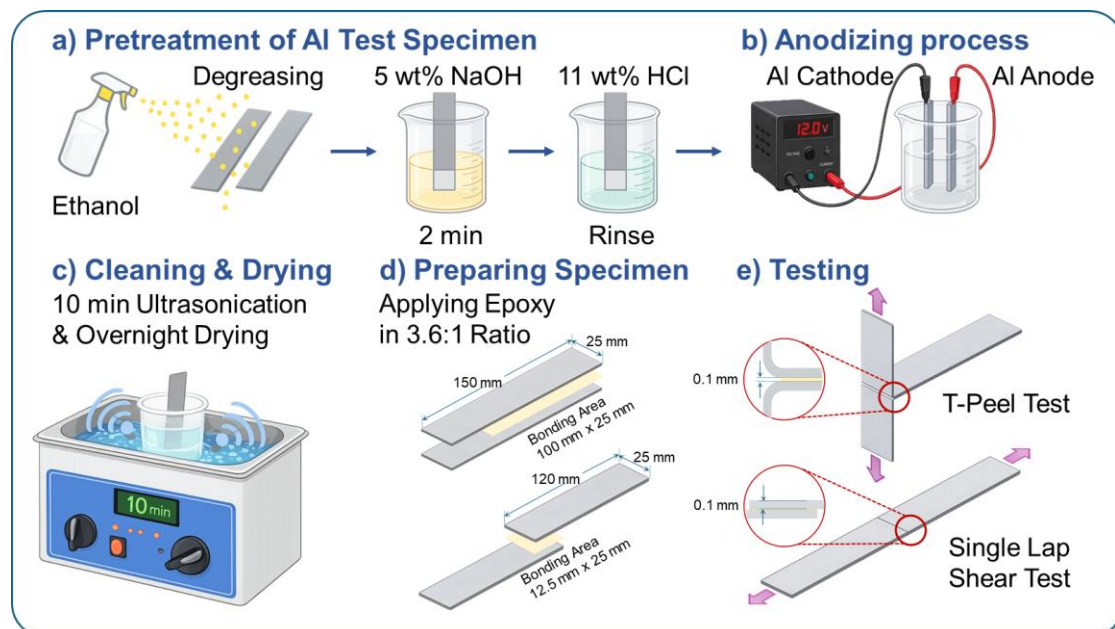


Figure 16. Full schematic of experimental conditions

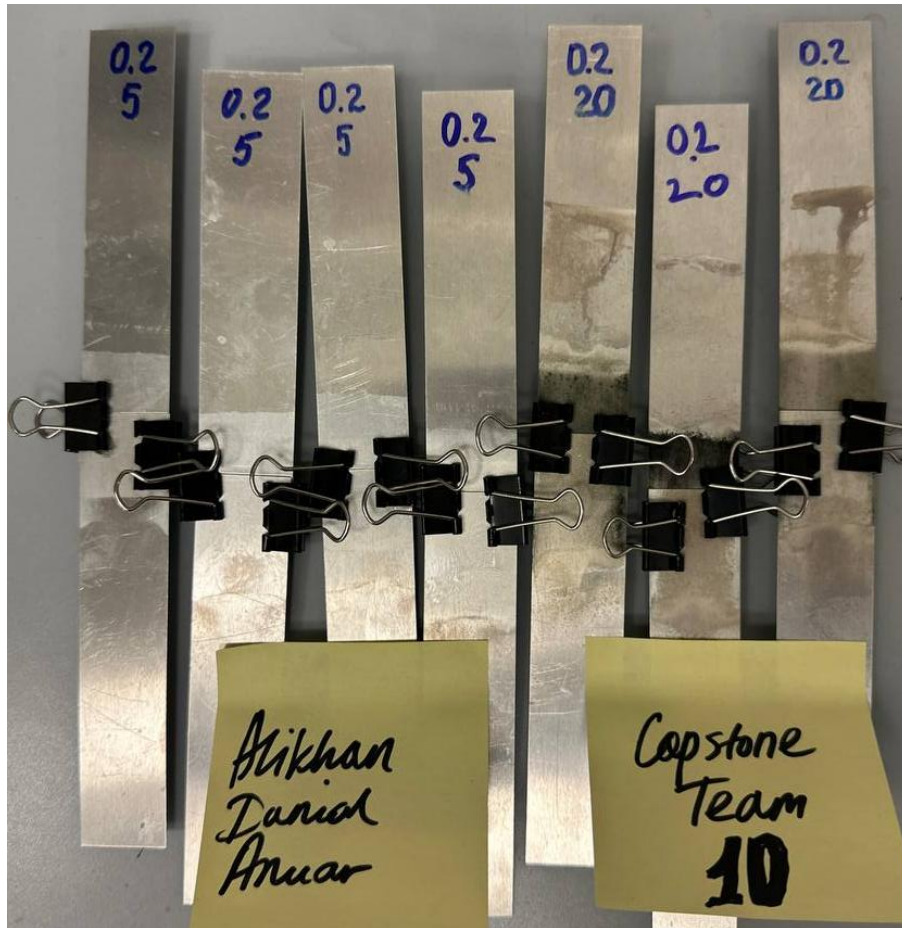


Figure 17. Lap shear joint specimens clamped with clips during the curing stage.

The summary of the FML manufacturing process is shown in Figure 19. Firstly, the aluminum sheets were degreased with ethanol to remove dirt and oil. In order to remove the oxide layer, the sheets were soaked in 5 wt% NaOH solution for 1 minute, followed by rinsing with DI water. Subsequently, they were anodized in 0.1 M KCl solution at 12 V for 20 minutes. Anodized samples were dried in the oven for 3 hours at 60 degrees Celcius.

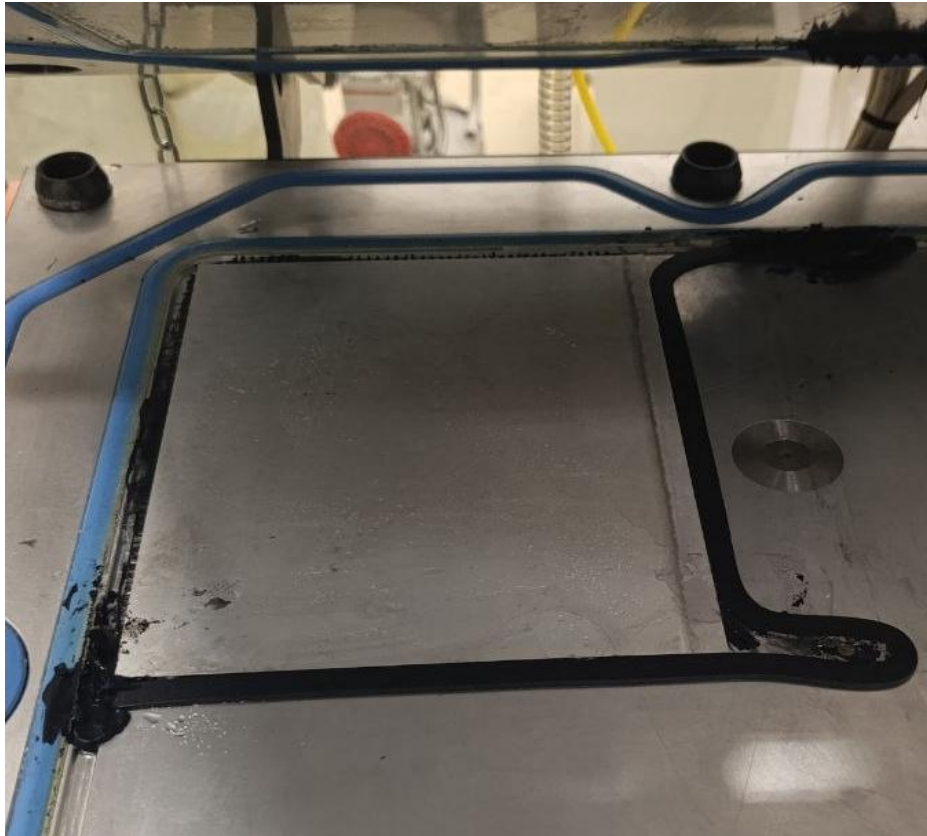


Figure 18. VARTM mold and resin infusion tubing setup.

Surface treated metal sheets and 4 carbon fabric layers were placed in the VARTM machine. The epoxy and hardener were mixed in a 3.6:1 ratio. Then, mixed epoxy was degassed to remove the air bubbles. Before infiltration, the mold was pre-heated up to 80 degrees Celcius. After the epoxy was fully infiltrated, the samples were left to cure at 80 degrees Celsius for 12 hours.

For the manufacturing of FML with conventional acidic treatment, after NaOH cleaning, the metal sheets were etched in 11 wt% HCl solution for 4 min. The subsequent procedures are the same as previous.

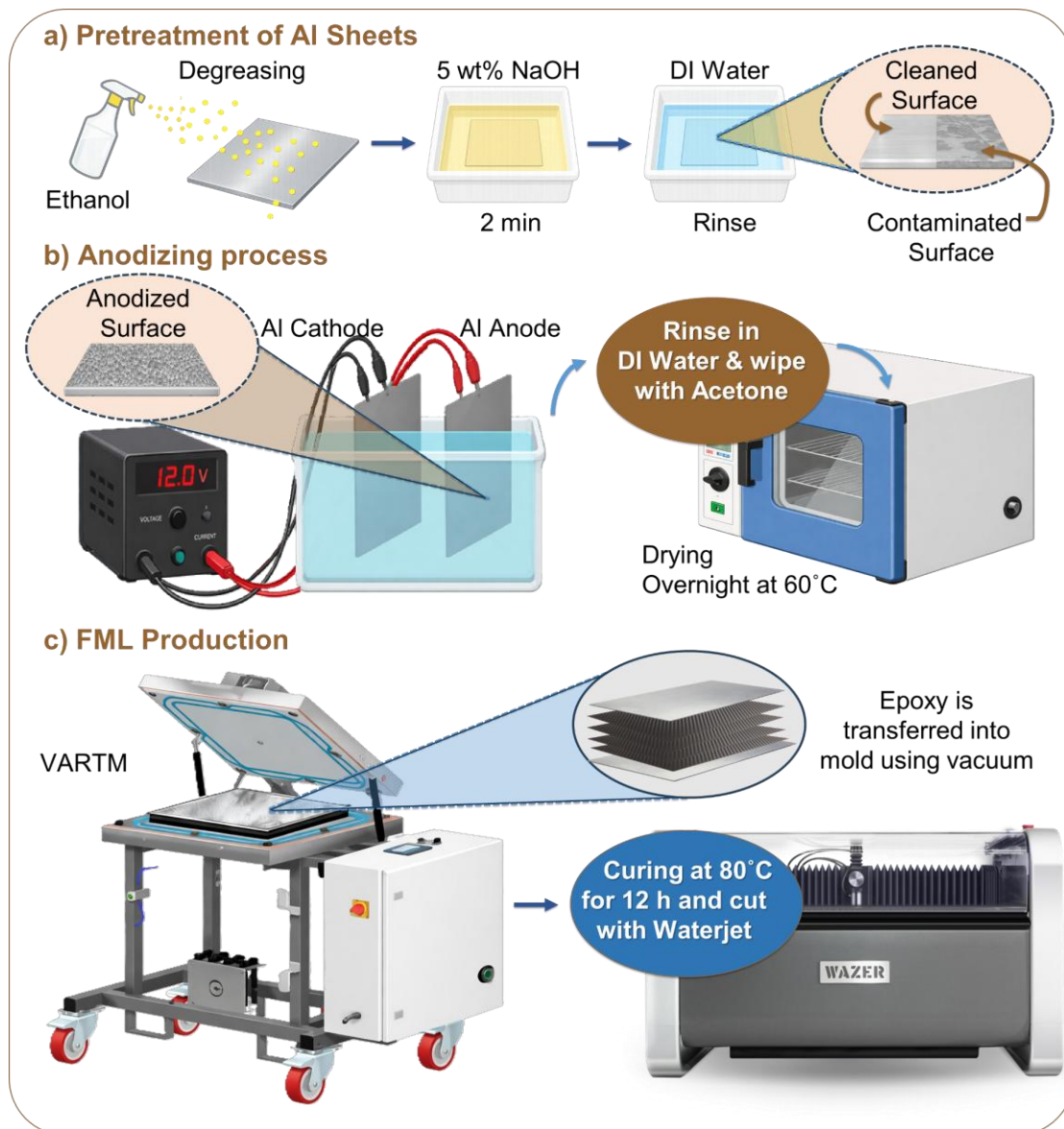


Figure 19. Process of FML fabrication.

3.4 Mechanical testing of composites

3.4.1 Tensile properties

The tensile test was performed using a UTM 100 kN machine. The samples were cut according to ASTM D3039 standard.



Figure 20. FML panel during water jet cutting.

The sample size was 160 mm by 25 mm. The strain rate is 1.5 mm/min. The tensile strength was calculated using the following equation:

$$\sigma_t = \frac{F}{bh}$$

Where:

σ_t = Tensile Strength (MPa)

F = Load (N)

b = Width of the sample (mm)

h = Height of the sample (mm)

3.4.2 Flexural properties

The flexural properties of the specimens were tested in accordance with ASTM D790. The samples were 160 mm long and 25 mm wide. The distance between supports is 11 cm. The strain rate is 13 mm/min. The flexural strength was calculated using the formula below:

$$\sigma_f = \frac{3FL}{2bh^2}$$

Where:

σ_f = Flexural Strength (MPa)

F = Load (N)

L = Length of the sample (mm)

b = Width of the sample (mm)

h = Height of the sample (mm)

3.4.3 Durability testing

The fabricated laminates were placed inside of the Salt spray chamber S750 to imitate the real-life environmental conditions. The testing was conducted according to ASTM B117 standard. The image of the salt spray chamber can be seen in Fig. 21. The samples stayed in the chamber for 7 days. The temperature inside of the chamber was 35 degrees C while the salt NaCl concentration was 5wt%. After 7 days, the laminates were rinsed in DI water to remove an excess NaCl solution and dried for 24 hours. The flexural test was conducted to evaluate the effect of environmental exposure on metal-polymer adhesion.

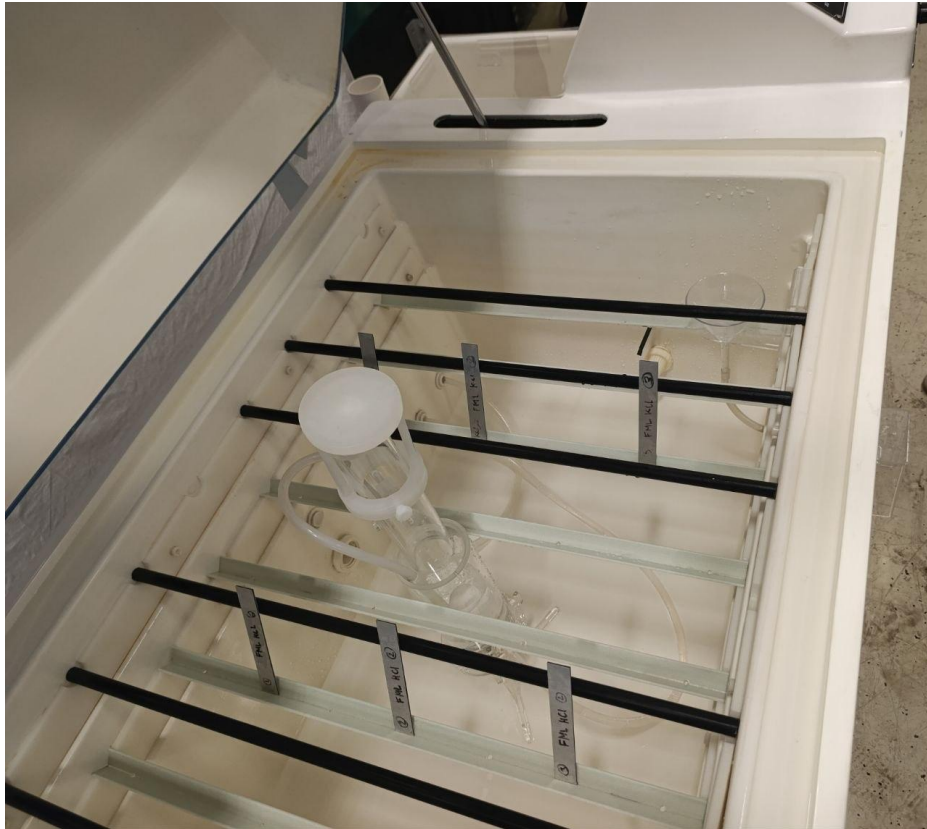


Figure 21. Placement of FMLs inside of the salt spray chamber.

4 Results and Discussions

4.1 Surface morphology

The surface morphology of all nine anodized samples and the anodized reference was examined using scanning electron microscopy (SEM) at 5,000 $\times$ magnification. Across all conditions, progressive changes in surface texture were observed as both KCl concentration and anodization duration increased.

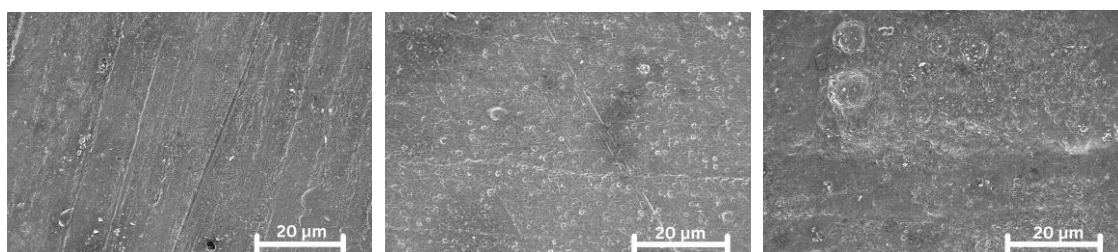


Figure 22. SEM images at 5K magnification for samples anodized with 0.05 M for 5 (left), 10 (center), and 20 (right) minutes.

At the lowest concentration of 0.05 M, the 5 minute anodized sample exhibited a predominantly smooth surface on which linear scratches are attributable to pre-anodization mechanical handling or post-manufacturing friction. These lines remained clearly visible, indicating that oxide layer formation was minimal at this early stage. The 10 minute anodized sample retained a generally smooth character, though shallow pitting features became discernible and the scratch lines remained visible, suggesting the onset of KCl surface modification. By 20 minutes, the scratch lines were no longer evident; however, a non-uniform distribution of pitting was observed, with clusters appearing in spatially random patterns, indicative of localized anodic dissolution at this concentration.

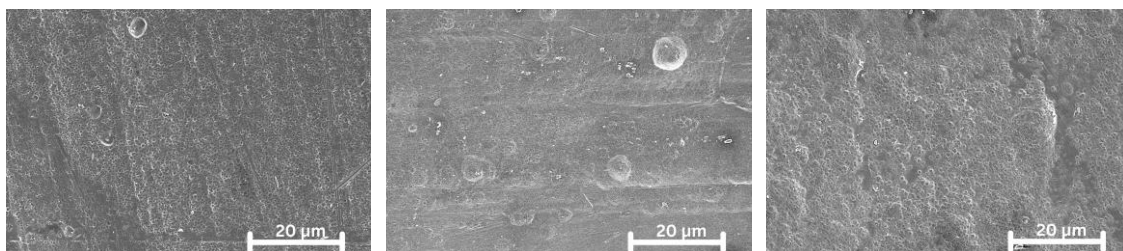


Figure 23. SEM images at 5K magnification for samples anodized with 0.10 M for 5 (left), 10 (center), and 20 (right) minutes.

At 0.10 M, the 5 minute anodized sample displayed a more organized surface texture, characterized by shallow pits of relatively uniform size distributed in a near-regular pattern. Occasional anomalous surface features consisting of isolated bright dots and featureless planar regions were observed, though at low frequency. Residual scratch line topography remained detectable upon close inspection. The 10 minute anodized sample showed a similar pit distribution, albeit with reduced pit depth contrast, suggesting continued surface penetration. The frequency of anomalous dots increased relative to the 5 minutes condition, while the featureless regions disappeared. At 20 minutes anodized sample, a terrain-like texture was observed, with visible surface undulations forming a continuous network of hills and valley-seated pits, suggesting the development of a more structured oxide morphology under prolonged exposure at this concentration.

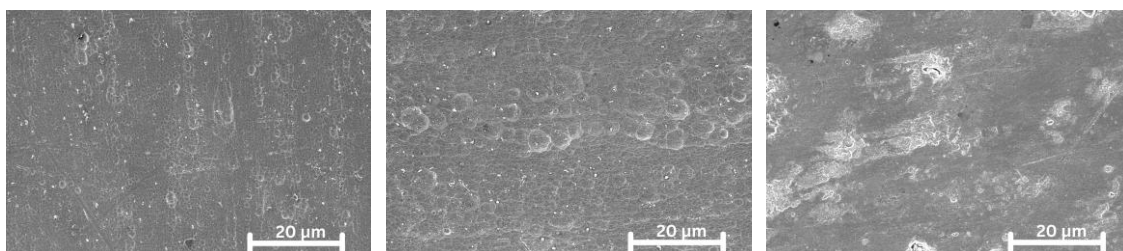


Figure 24. SEM images at 5K magnification for samples anodized with 0.20 M for 5 (left), 10 (center), and 20 (right) minutes.

At the highest concentration of 0.20 M, the 5 minute sample presents faint residual scratch features, less pronounced than those observed in the

corresponding 0.10 M condition. Pitting of mixed size ranging from fine to moderate was distributed in a spatially random manner. The 10 minutes anodized sample exhibited a distinctly different morphology: pits were observed to be seated within larger crater-like depressions, with estimated diameters approximately three times that of the medium-scale pits observed at shorter, 5 minutes duration. These craters exhibited well-defined boundaries and were distributed in a moderately consistent pattern, though scratch features were now largely obscured. The 20 minute sample appeared comparatively smoother than its shorter duration counterparts within this 0.20 M concentration group. The presence of dark spots and crack-like features were noted, which are tentatively attributed to localized over-oxidation or the formation of a thicker, more defect prone oxide layer resulting from prolonged exposure at elevated KCl concentration.

4.2 Surface roughness

Surface roughness was quantified using a contact profilometer (Dektak XT, Bruker) employing a 2 μm radius stylus at a 1 mg stylus force over an 800 μm scan length at 0.133 μm resolution. To account for inherent surface unevenness of the aluminum substrate, four measurement zones were sampled per specimen and the minimum recorded roughness value was retained for analysis. This conservative approach was adopted to isolate the effect of anodization parameters by minimizing the contribution of pre-existing substrate macro-level irregularities. The unanodized reference sample yielded a baseline roughness of 93.69 nm.

As summarized in Figure 25, all anodized specimens exhibited increased surface roughness relative to the reference, confirming that anodization consistently modified the aluminum surface at the micro-scale across all tested conditions.

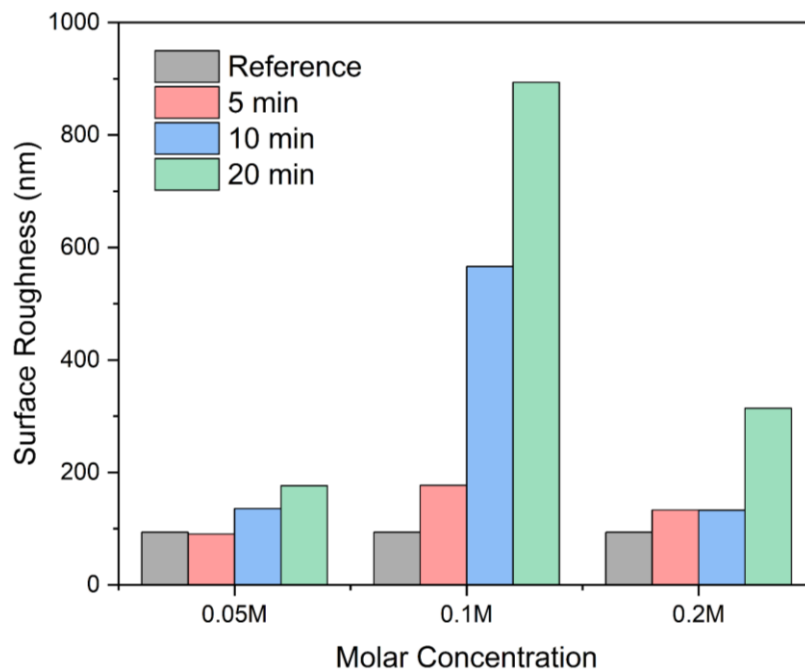


Figure 25. Surface roughness of all 9 conditions.

Within the 0.05 M concentration group, roughness increased monotonically with anodization duration, from 90.47 nm at 5 minutes, comparable to the reference, to 135.84 nm at 10 minutes and 176.41 nm at 20 minutes. The near-reference value at 5 minutes is consistent with the SEM observation of a barely discernible oxide layer at this condition, suggesting that 5 minutes at 0.05 M is insufficient to produce meaningful surface modification.

At 0.10 M, a monotonic trend was observed. Roughness increased substantially to 177.05 nm at 5 minutes, increased to 566.26 nm at 10 minutes, and to 893.78 nm at 20 minutes, the highest value recorded across all conditions. This result suggests that extended anodization at intermediate KCl concentration promotes significant surface disruption, consistent with the terrain-like morphology observed in the corresponding SEM image.

At 0.20 M, roughness stayed almost the same at 5 minutes and 10 minutes; 133.32 nm and 132.81 nm respectively. At 20 minutes, the roughness increased significantly to 314.32 nm. A clear trend was observed; as anodizing time rose, the surface roughness also increased. The similar behaviour was observed at all three concentrations. 20-minute sample and its associated dark-spot and crack-like features, potentially indicative of a compact but defect-bearing oxide film.

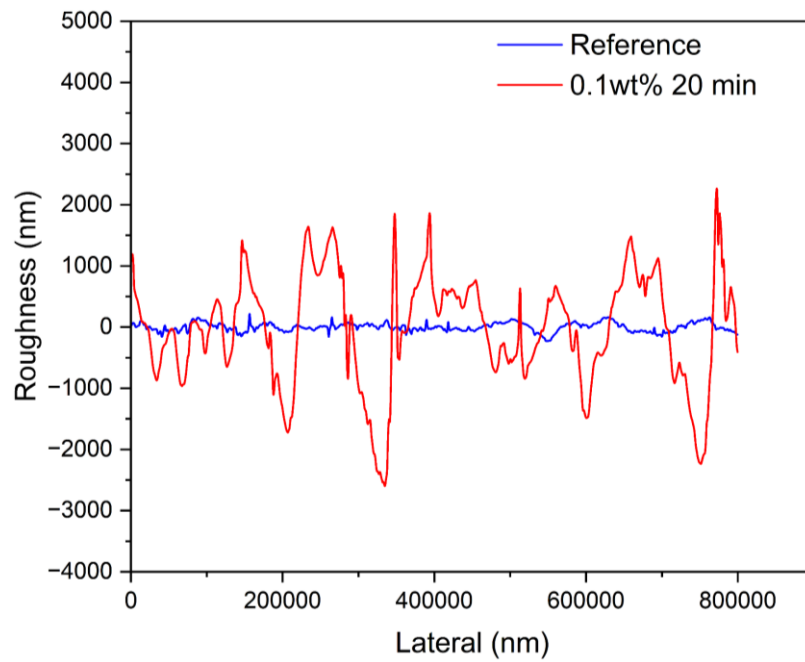


Figure 26. Surface roughness of reference sample and most rough sample.

In summary, the results demonstrate that there is considerable significance of both concentrations of KCl and the time of anodization in affecting surface roughness, with effects from the interaction of both factors being non-linear. Of all the samples studied, those treated with 0.10 M for 20 minutes showed the greatest roughness improvement compared to the baseline by about 9.5 times, and may thus be a good choice for testing FML applications.

4.3 Single lap shear strength test

The average of results of lap shear strength for all nine KCl anodizing conditions with comparison with the untreated reference are shown in Figure 27. Untreated reference showed an average shear strength of 1.95 MPa, whilst all KCl-anodized conditions significantly outperformed the reference, proving that electrochemical surface treatment significantly enhances adhesion between metal and polymer.

Among all tests, the highest lap shear of 9.63 MPa was achieved with 0.1M KCl and 20 min of anodizing. It is visible that the effect of anodizing duration differed across concentrations. At 0.1M, shear strength increased consistently with time: 8.6 MPa at 5 min, 9.23 MPa at 10 min, and 9.63 MPa at 20 min. At 0.05M, the best result happened at 10 min, while at 0.2M, 20 min of anodizing yielded the highest shear strength of 9.23 MPa. At high concentration of KCl, 5 min and 10 min both yielded the comparable results, which can be explained by the excessive roughness's introduction of stress concentrators that reduces adhesion strength locally, as discussed by Bakhbergen et al [19].

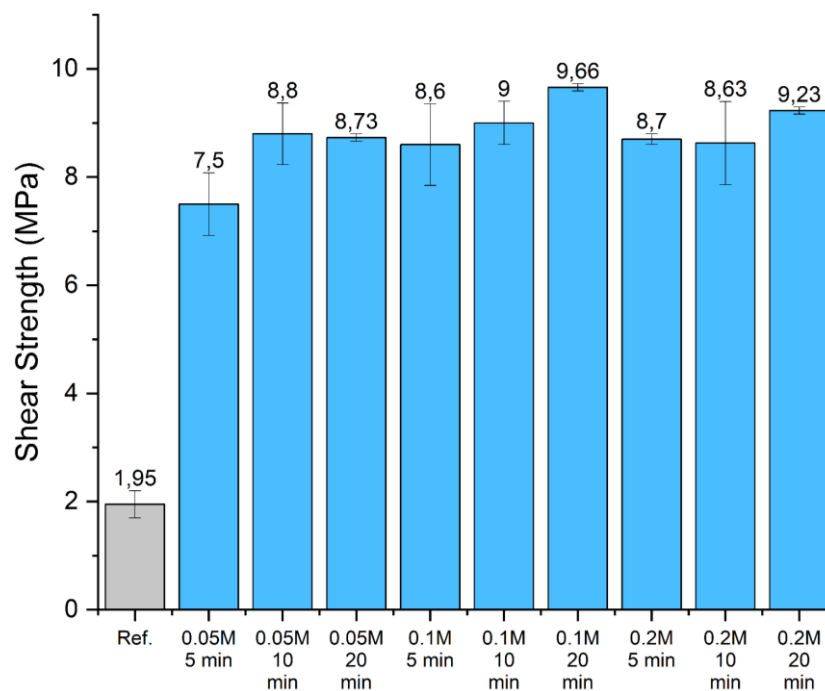


Figure 27. Results of the single lap shear strength test.

4.4 Peeling test

The peel test evaluates the resistance to mode I-dominated interfacial crack propagation and gives a characterization of the different loading mode. For this test, the average peeling force was normalized by an average sample width of 25 mm to get peel strength in N/m. Results for all conditions, including the reference, are given in Figure 28.

The reference sample showed a peel strength of 240.2 N/m, when all anodized conditions produced higher results. Across all conditions, anodizing duration showed a consistent positive effect on the peel strength. This trend suggests that longer anodizing develops a deeper porous oxide layer that resists better to peel-dominated crack propagation, consistent with the findings reported in Bakhbergen et al. [19]. The highest peel strength was seen at 0.2M at 20 min, but at the concentration 0.05M the average strength across three durations was higher. Notably, 0.1M, which performed the best in the lap shear test, produced the lowest results in average among three durations.

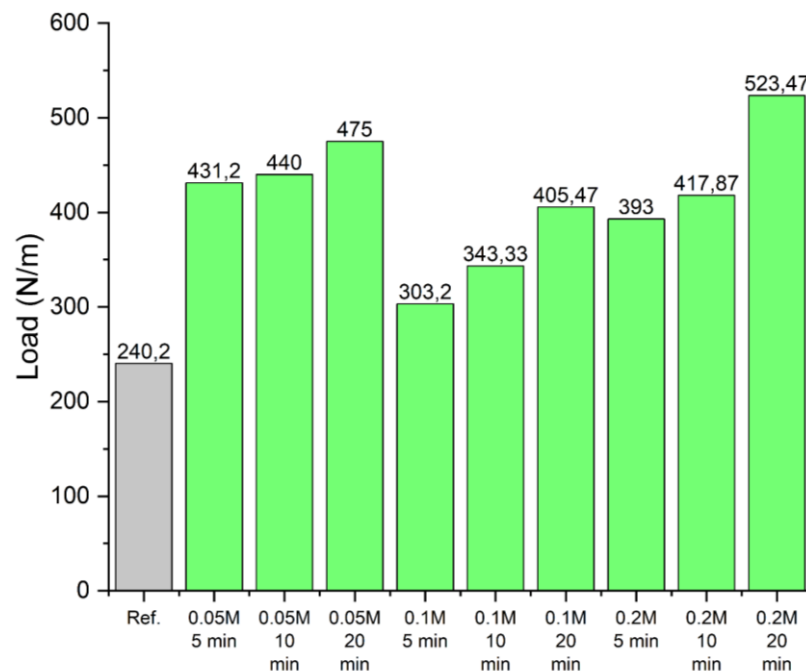


Figure 28. The results of the peeling test.

4.5 Interface analysis

The SEM images of the metal-polymer interface were taken after the lap shear test. The fractured samples for three conditions were analyzed; untreated sample, 0.05M at 5 min and 0.1M at 20 min. Above mentioned conditions have demonstrated different results in shear tests. The reference sample without any surface treatment expectedly have shown the lowest shear strength. From Figure 29 (a), it can be seen that the surface of the aluminum is almost flat leading to poor mechanical interlocking effect. The resin cannot penetrate deeply into the metal. Therefore, low surface roughness directly caused the weak interfacial adhesion. The similar trend can be observed in other two conditions with higher shear strength. Clearly, the metal surface is more rough and wavy. The interlocking effect between metal and polymer resin is facilitated by depth of the micro-pours. The interface analysis between metal and polymer shows that surface roughness is directly proportional to the adhesion strength.

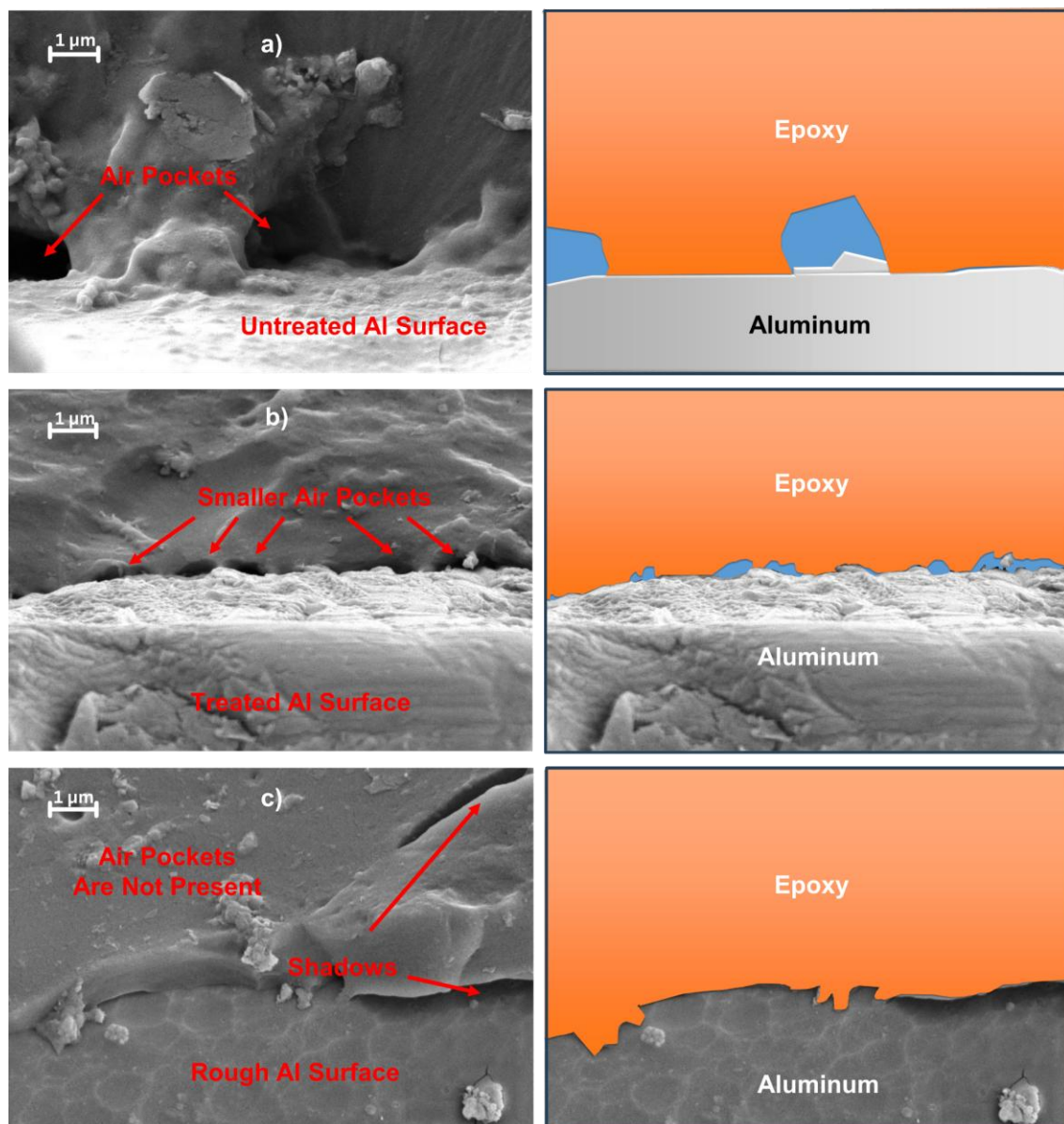


Figure 29. The SEM images of metal-polymer interface in (a) untreated reference sample, (b) 0.05M concentration for 5 min (c) 0.1M concentration for 20 min.

4.6 Tensile test of FMLs

Tensile tests were performed on both KCl-anodized and HCl-treated samples. The stress-strain curves are shown in Figure 30. It can be seen that FML with HCl etching exhibits ultimate tensile strength and Young's modulus of 387 MPa and 56 GPa, respectively. While the tensile strength value meets the literature-reported standard of around 400 MPa [23]. The samples with a KCl-anodized surface showed similar results in both strength and elongation (Figure 31). Therefore, surface treatment did not affect the tensile properties of the FML. A cross-sectional load can explain this phenomenon during the tensile test. The inherent properties of bulk materials limited the material's strength. In our case, the carbon fiber itself.

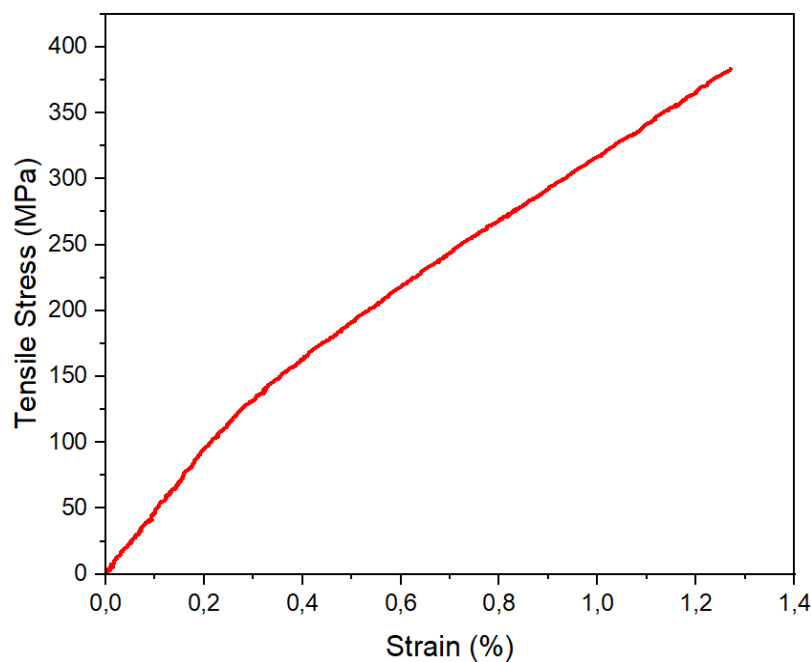


Figure 30. Stress-strain curve of HCl etched FML.

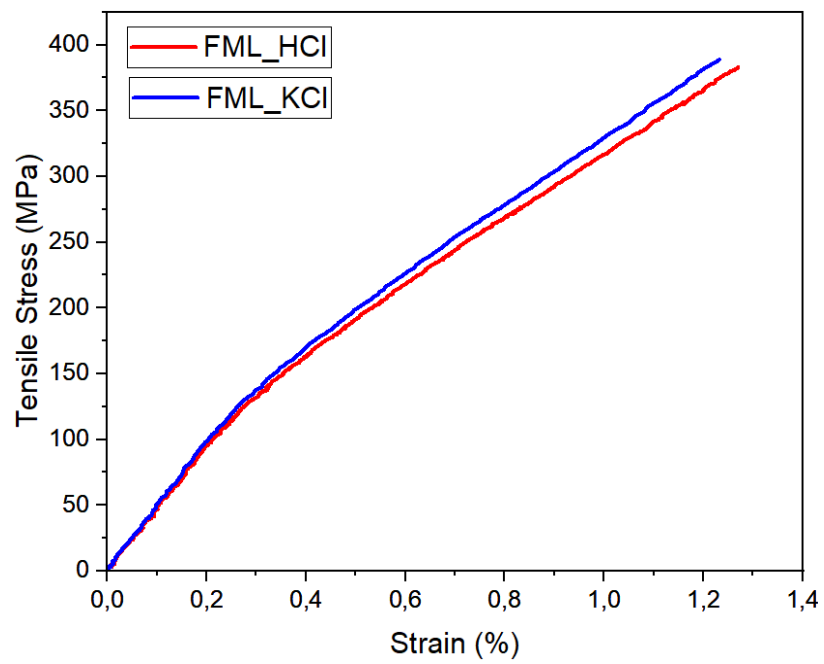


Figure 31. Comparison of tensile strength between HCl-etched and KCl-anodized FML.

4.7 Flexural test

The 3-point bending test was performed for both surface treatments. This test is crucial for examining the structural integrity of laminated materials such as FML. The adhesion between two dissimilar materials remains the weakest part of the structure. In a 3-point bending test, the shear stress is applied on the interface between the layers. Therefore, the results of the flexural test demonstrate whether adhesion is satisfactory or not. The flexural stress-strain curve of FML with HCl-etched metal can be seen in Figure 32. The ultimate flexural strength of 373 MPa is acceptable according to the literature. However, the samples with KCl-anodized metals showed a flexural strength of 595.8 MPa (Figure 33), resulting in a 64% improvement. KCl anodizing improved the adhesion strength between the metal and polymer while making the production process more sustainable.

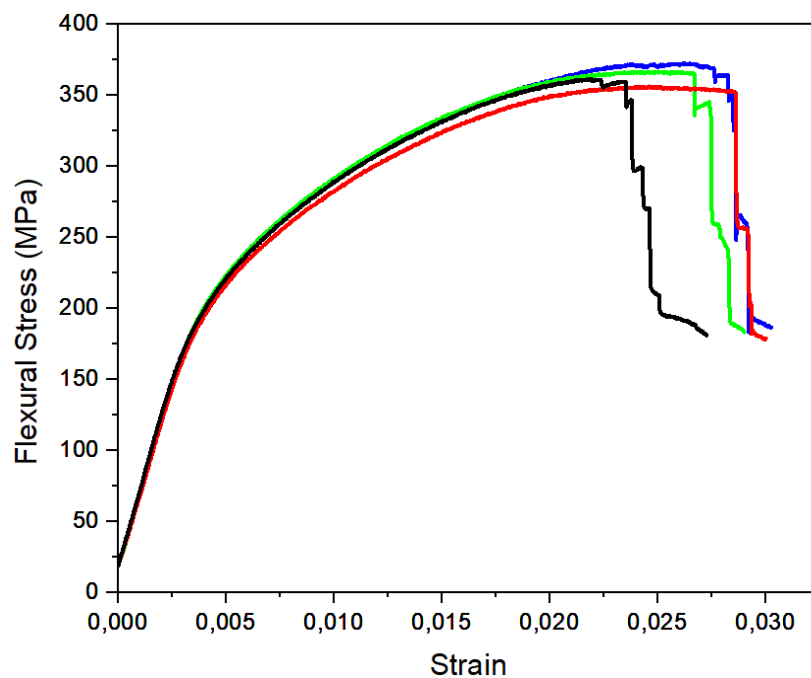


Figure 32. Flexural stress-strain curve of HCl etched FML.

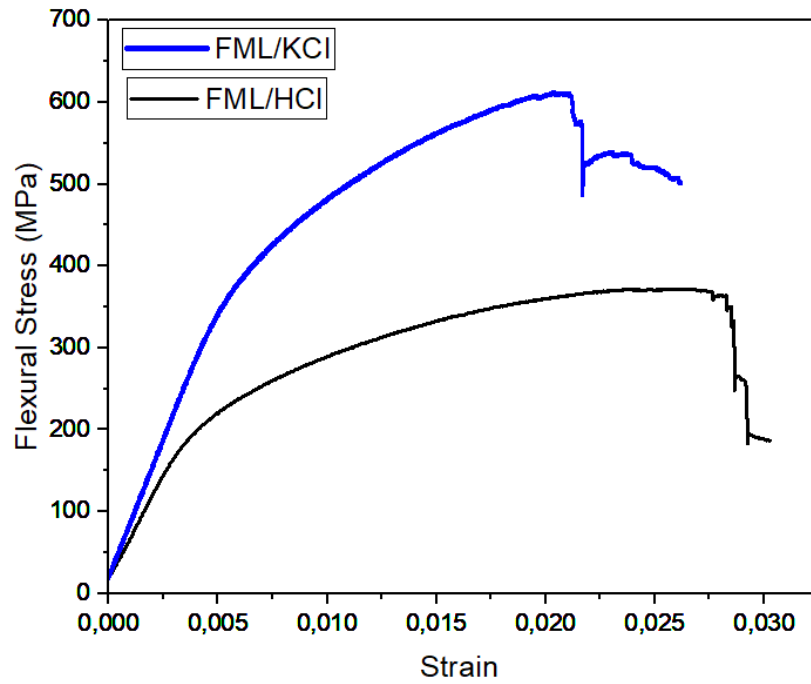


Figure 33. Comparison between HCl-etched and KCl-anodized FML.

To justify the improved adhesion strength, SEM images of Al sheets after both surface treatments were taken; the picture of the HCl etched sample can be seen in Figure 34. After the alkaline cleaning procedure, the oxide layer is removed from the metal. The subsequent soaking in an acidic solution created micro-pores on the metal's surface. When the epoxy resin infiltrates, the polymer penetrates the pores and forms a strong bond with the metal. This process is called mechanical interlocking. It is one of the most important mechanisms that governs the adhesion of FMLs.

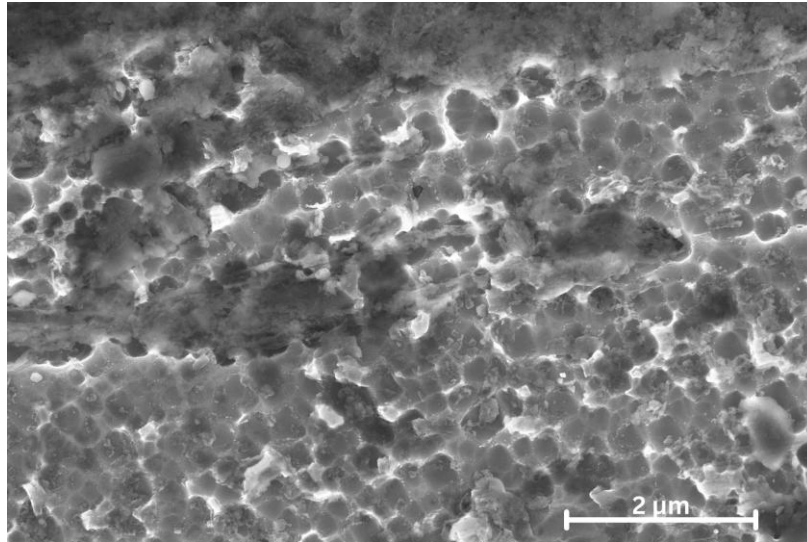


Figure 34. SEM image of HCl-etched Al sheet.

The SEM image of the metal surface after KCl anodizing is shown in Figure 35. The anodizing process also created micro-pores for a resin to interlock with. In addition to micro-scale pores, KCl anodizing creates nano-scale pores. The epoxy has significantly more points of contact with metal, making adhesion even stronger.

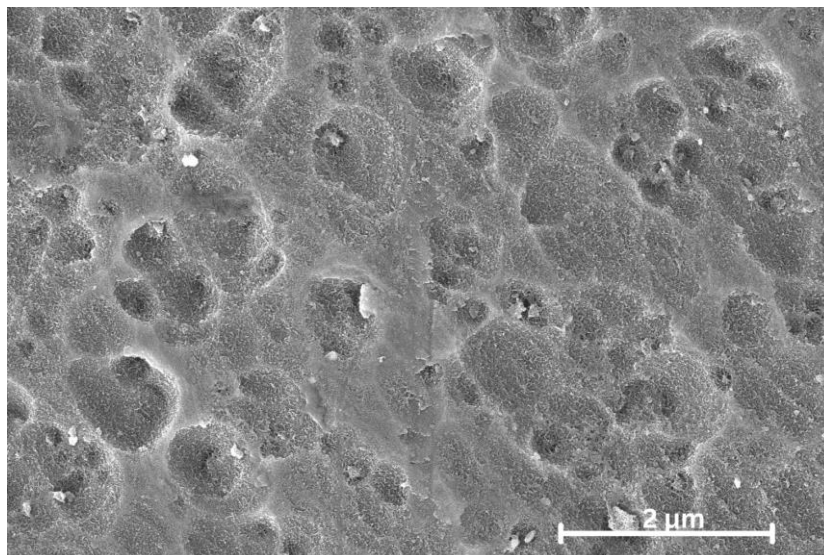


Figure 35. SEM image of KCl anodized Al sheet.

4.7 Durability test

After the FMLs stayed in the salt spray chamber for 7 days, the flexural test was repeated for both HCl etched and KCl anodized laminates. The obtained results were compared with the mechanical properties of laminates before the durability test. The flexural stress-strain curve for HCl etched FML before and after the test can be seen in Fig. 36. It was found that the tensile strength decreased from 363 MPa to 203 MPa while the breaking strain fell roughly to 0.0025. The premature failure in an elastic zone occurred due to severe delamination. Therefore, it can be concluded that exposure to harsh environmental conditions significantly weakened the interfacial area between metal and composite. This behaviour can be attributed to the removal of the native aluminium oxide layer (Al_2O_3) during the acidic treatment. In the absence of a protective oxide layer, the rough aluminum surface became vulnerable to chemical attacks. Negative Cl ions from the NaCl spray solution initiate the pitting corrosion process. Consequently, stress concentrations are formed due to pitting corrosion leading to weakening of the interface [29].

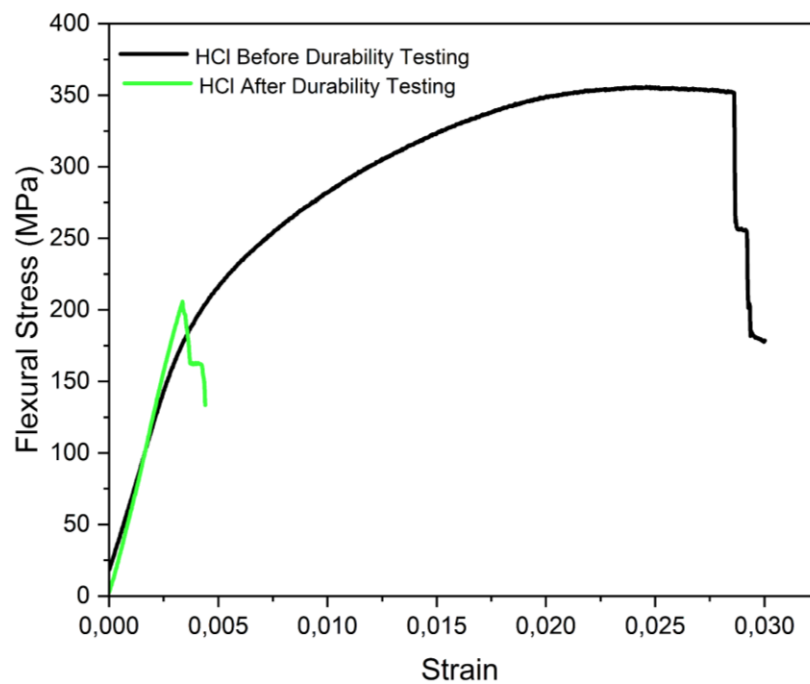


Figure 36. The comparison of HCl etched FMLs' stress-strain curves before and after the durability test.

The comparison of flexural stress-strain curves for KCl anodized laminates are illustrated in Fig. 37. It can be seen that tensile stress increased but less significantly in comparison to HCl etched laminates; from 595.8 MPa to 532 MPa. The mechanical behaviour of the FML was almost exactly the same until the failure at the strain of 0.015. Overall, anodized laminates were noticeably less susceptible to environmental conditions. This phenomena can be explained by the presence of the protective aluminium oxide layer. This thick layer of oxide as a barrier for corrosion attacks from negative Cl ions. Since HCl removed this chemically stable layer, acidically treated laminates demonstrated significant vulnerability to corrosion [30].

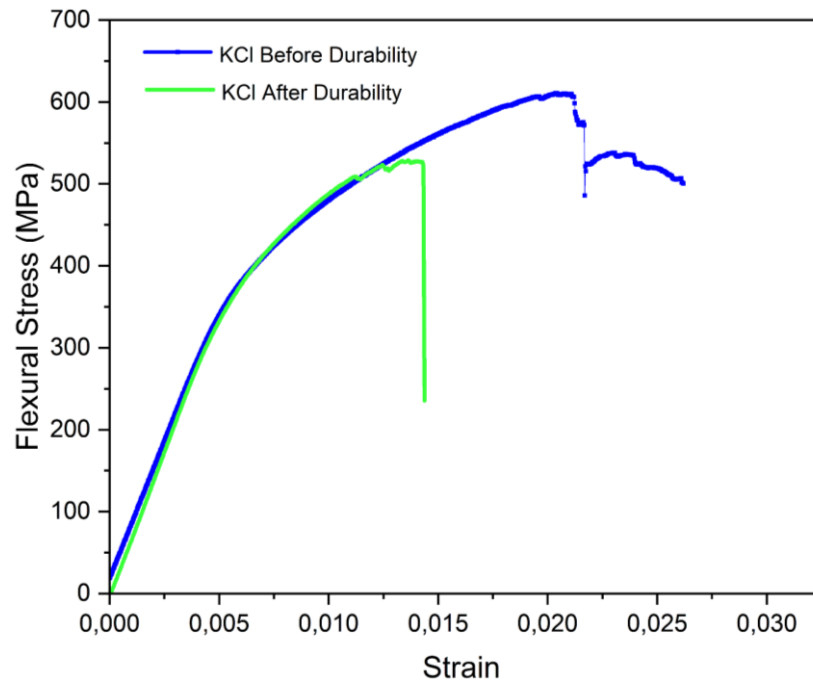


Figure 37. The comparison of KCl anodized FMLs' stress-strain curves before and after the durability test.

4.8 Fracture analysis of FMLs

The fractographic analysis was conducted on samples after a flexural test because the fractured surface enables us to directly assess the interface adhesion quality and identify failure mechanisms. The SEM images of the both HCl etched and KCl anodized laminates after fracture are illustrated in Figures 39 and 41.

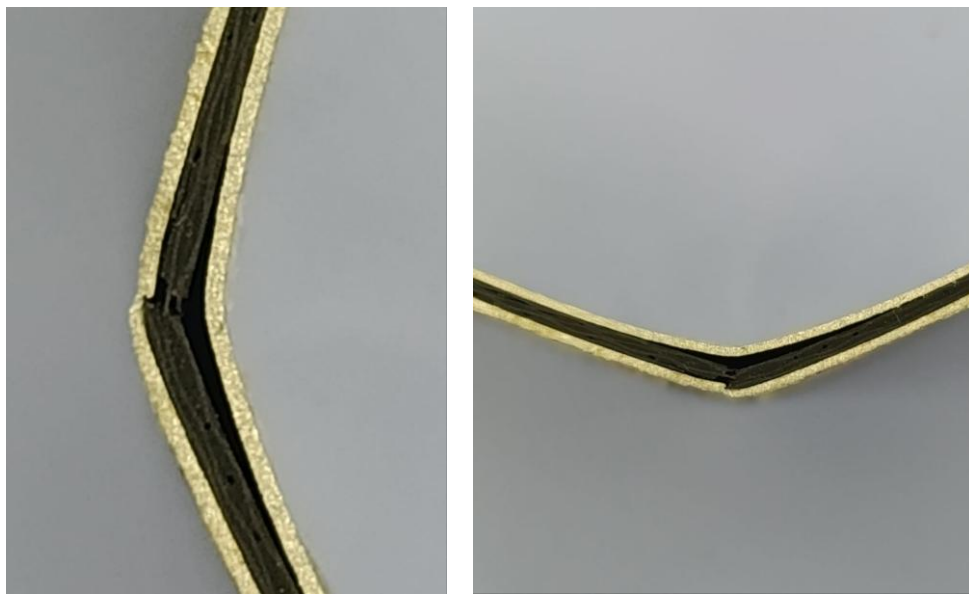


Figure 38. Cross-sectional view of FML specimen after three-point flexural testing.

Usually, the weakest area of FMLs is the interface between two dissimilar materials. The carbon fiber is significantly stiffer than aluminum which leads to a strain mismatch. When the laminate is loaded, the aluminum wants to stretch more but is constrained by the low strain of the carbon layer. This behavior will lead to shear stress concentration at the interface between layers [31]. In Figure 39 (a), it can be seen that the upper interface between metal and epoxy has delaminated while the lower one still remained intact. The interface surface is not smooth but rough. The micro porous surface created by acidic treatment was penetrated by a polymer resin.

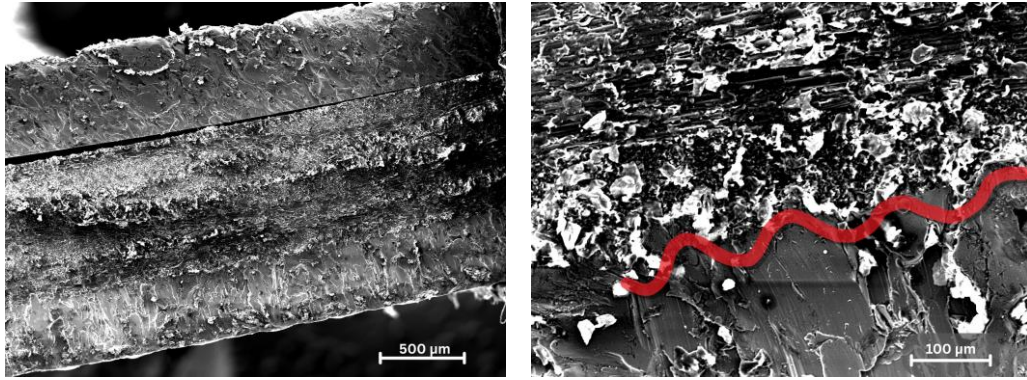


Figure 39. SEM images of the HCl etched FML interface.

The primary mechanism of failure in the laminate was fiber breakage. This behaviour implies that the adhesion between metal and polymer layer is strong. In case of the weak adhesion, the primary failure mechanism is delamination. It is considered to be an undesirable event because it leads to a catastrophic failure. However, when the fracture of fiber occurs before delamination, it means that the weakest part is the flexural strength of the carbon fibers' itself. In the magnified images, clear fiber breakage and fiber pull-out mechanisms can be noticed.

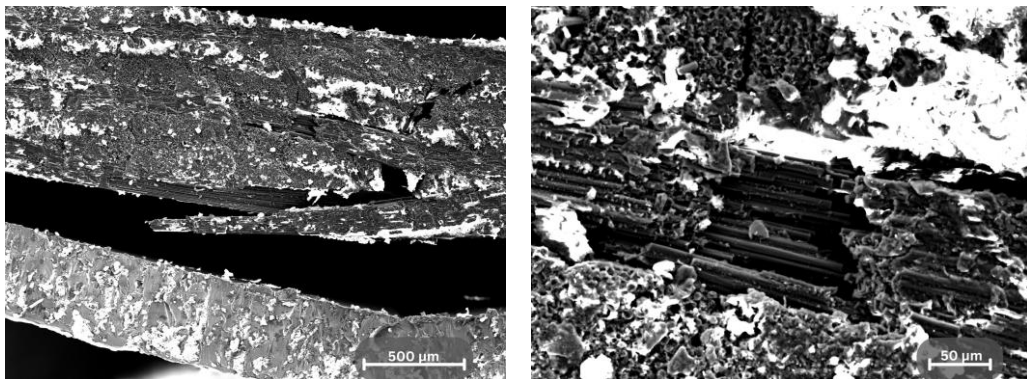


Figure 40. SEM images of fractured HCl etched FML.

For the comparison, the SEM images of KCl anodized laminates are shown in Figure 41. There are two types of failures in adhesively bonded structures; adhesive failure and cohesive failure. The former describes the delamination of the interface between adhesive and adherent. On the other hand, when the fracture occurs within the adhesive or the adherent, this phenomenon is called

cohesive failure. Adhesive failure indicates that the interface is the weakest part of the laminates. In general, cohesive failure is considered to be more desirable because it is governed by the internal strength of the adherent itself [32]. The cohesive failure between carbon layer and epoxy can be clearly seen in KCl anodized specimens. This phenomenon can justify the improvements in the flexural strength in comparison with the HCl treated laminates.

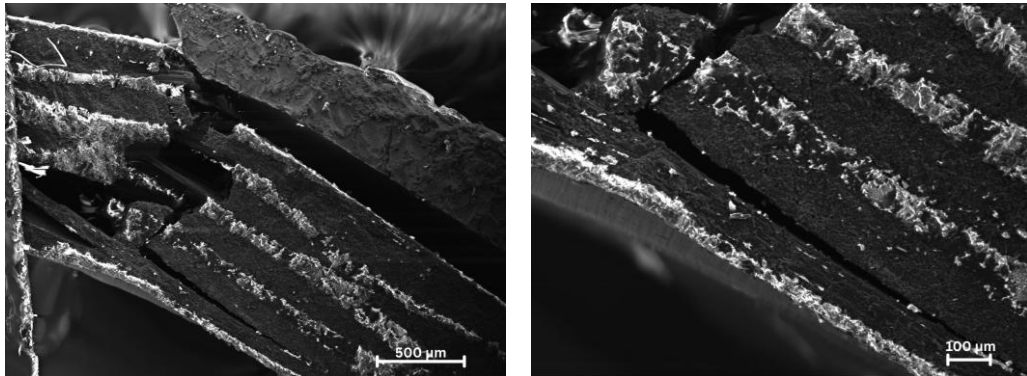


Figure 41. SEM images of fractured KCl anodized FML.

5 Date Seed Nanoparticles

5.1 Background

Dates are produced in large quantities worldwide, contributing to substantial agricultural waste in the form of date seeds. Din et al. [27] gives an example in the case of the UAE, where apparently around 10 million palm trees are growing, giving around 249,000 tons of dates annually. The subject of this research is the seeds of these dates, which constitute 8-15% of the mass of each date. These seeds are not used for consumption and, therefore, are frequently thrown away, which makes them an ideal source of nanocarbon because of their sustainability.

The thermochemical transformation of lignocellulosic biomass has been extensively studied as a means to produce carbon materials for environmental and structural applications. Zouari et al. [28] demonstrate that at 600 °C for 2 hours, *Arundo donax* and olive stones can be carbonized in nitrogen to produce biochar powders exhibiting favorable structural characteristics. The study found that demineralization reduced ash content by 48% in *Arundo donax* and 40% in olive stones, consequently enhancing pore development and enlarging surface area. Additionally, ball milling at 400 rpm resulted in micro-sized carbon particles measuring 13-22 µm for processed olive-stone and 30-42 µm for unprocessed material. Ball-milling enhanced the surface area by 47-124%, varying by treatment conditions, which highlights its effect on producing high-surface-area carbon suitable for polymer reinforcement [28].



Figure 42. Illustration of the complete formation of OACDS. [27]

These studies support our theory, showing that nanocarbon production can be done with simple steps of carbonization and ball milling, and that this produced nanocarbon has the mechanical properties for composite reinforcement.

Research by Din et al. [27] has already used date seeds to produce oxidized carbon. Their method consisted of calcination at 350°C for 5 hours and activation of the product in 98% sulfuric acid. This finding of the exact experiment covering date seeds provides a solid ground for further research, proving the theory that date seeds can be used for nanocarbon production. Taken together, these studies support the feasibility of natural and hybrid fiber FMLs as a more sustainable alternative to conventional synthetic fiber FMLs.

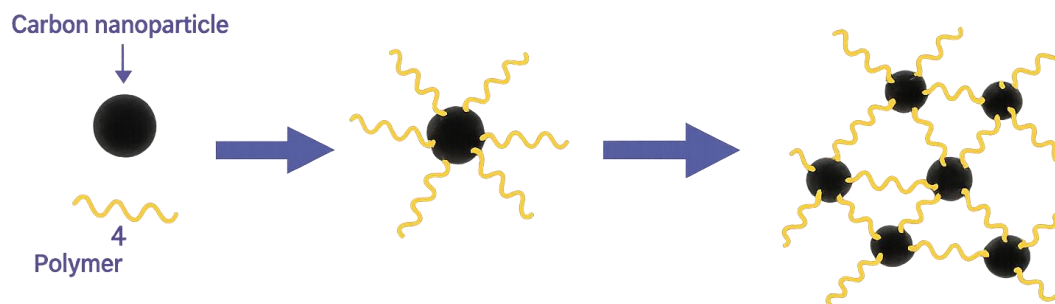


Figure 43. Schematic illustration of the preparation strategy for carbon nanoparticle and polymer colloidal nanocomposite clusters.

In parallel with the surface treatment and nano-reinforcing research, considerable attention was given to replacing synthetic fibers with natural or hybrid fibers in FMLs. This attention was motivated by a research of Ng et al. that demonstrated that FMLs using pineapple leaf and glass fiber hybrids achieved competitive mechanical test results, confirming the viability of agricultural fibers as FML reinforcement [24]. Similarly, Feng et al. showed that fibers of kenaf and pineapple leaf have acceptable mechanical properties in both tensile and flexural loading tests, where hybridization ratio and fiber orientation are proved to be the key variables contributing to the performance [25]. A comparison of Mohammed et al. of FMLs with natural fibers versus synthetic fibers proved that natural fibers can be used reliably in structural applications if adhesion is controlled reliably [26].

5.2 Experimental matrix

Palm dates were initially sourced, and their stones were manually separated. The stones were thoroughly washed and dried to ensure all external residues were removed. Cleaned seeds were carbonized in a muffle furnace using a controlled heating protocol: the temperature was raised at 5 °C/min to 600 °C. Then, the stones were maintained at this temperature for 2 hours under an oxygen-limited environment to promote carbon formation while minimizing ash and volatile content.

After carbonization, the resulting seeds were cooled to ambient temperature and ground in a mortar and pestle to produce a fine powder. Then, the powder was sieved to isolate particles smaller than 100 micrometers. This coarse powder underwent further size reduction via ball milling. The milling process was conducted at 400 rpm for a total of 4 hours, with periodic pauses (7-minute stops every 30 minutes of milling) to prevent overheating and maintain nanoparticle quality. Finally, this powder was designated as a carbonized date-seed nanoparticle for composite fabrication.

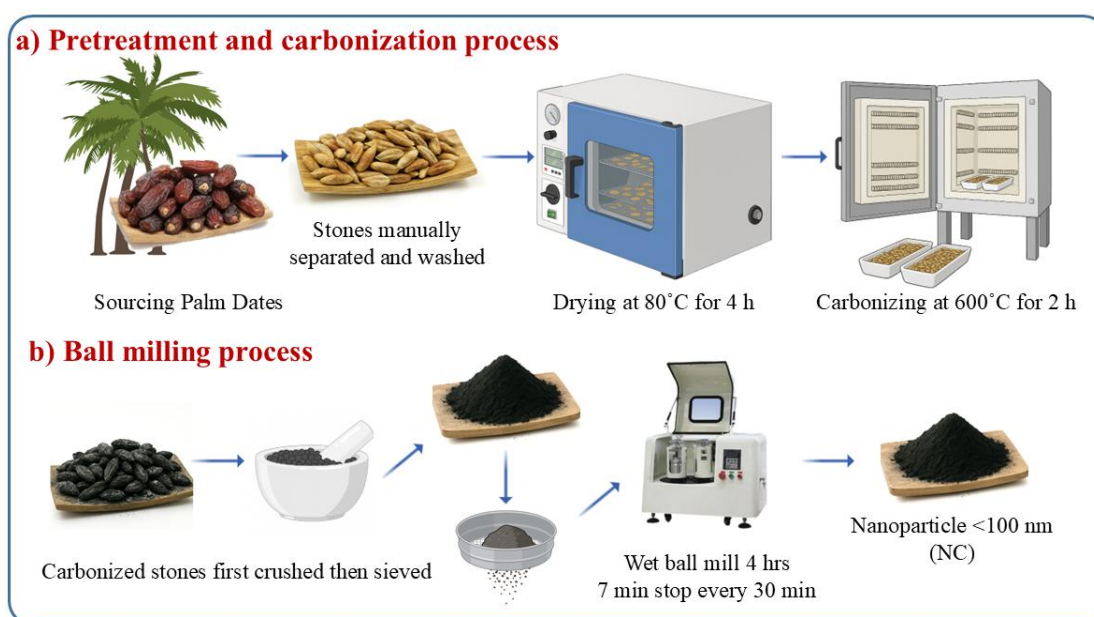


Figure 44. Process of carbon nanoparticle fabrication.

However, this was not enough in terms of reaching the desired yield in nanocarbons and thus we proceeded in conducting a full experimental matrix of nine different conditions: for each ball milling time (30, 60 and 120 minutes) a certain amount of rpm (200, 300 and 500 rpm).



Figure 45. Fabricated fiber-epoxy composite with addition of date seed derived particles prepared for tests.

5.3 Characterization

To evaluate the properties of the fabricated carbonized date-seed nanoparticles (NP), morphological and structural characterization were performed using standardized techniques.

5.3.1 Morphology analysis

Surface Morphological Studies The morphology of the nanoparticles was studied through Scanning Electron Microscopy (SEM). SEM was helpful in studying the surface morphology, shape, and sizes of the particles. Before analyzing the surface morphology, the samples were treated to ultrasonication, to facilitate dispersion of the nanoparticles, before dropping them on copper foil and coating them with gold for SEM imaging purposes. Through SEM imaging, it was observed that a very minute percentage of particles was in an optimum state for reinforced FML due to their size (less than 100nm).

5.3.2 Structural analysis

FTIR Spectroscopy (Fourier Transform Infra Red Spectroscopy) was used as the basis of structure elucidation, hence its inclusion in this study. The FTIR analysis was done with the aim of determining the surface functional groups through absorption peaks, specifically for the C-O, C=O, and aromatic C=C bonds. This was useful in validating the results obtained in our sample compared to those found in the literature.

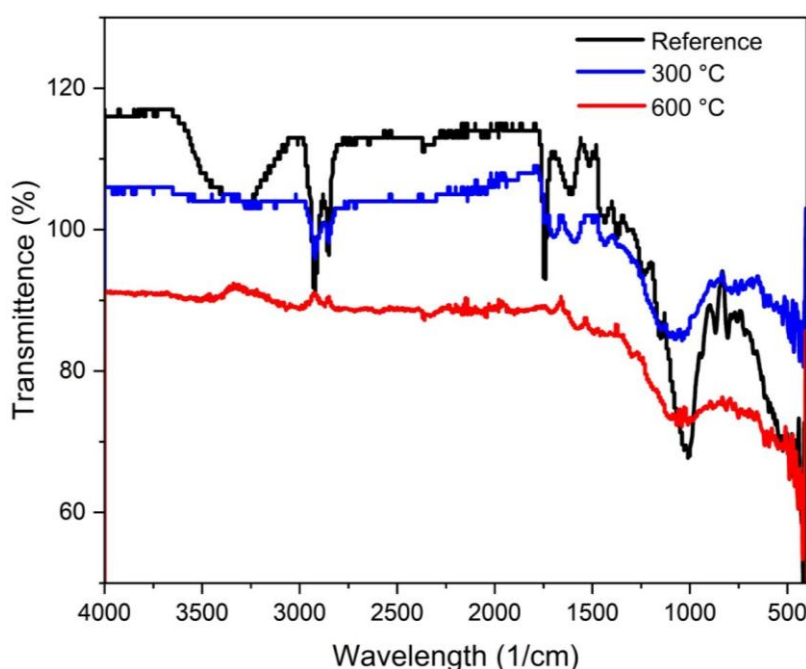


Figure 46. ATR-FTIR of reference and burned samples of date pits at different temperatures.

The functional groups present in the reference sample have obvious absorption bands within the range of 3000 to 3500 cm^{-1} due to the presence of O-H and C-H stretching vibrations, which are the characteristics of cellulose, hemicellulose, and lignin, which constitute the biomass materials. The other obvious absorption bands include those in the 1600 to 1700 cm^{-1} region for C=O stretching vibrations, and those at about 1000-1200 cm^{-1} for C-O and C-O-C ether bonds. As the pyrolysis temperature increases up to 300 °C, the intensity of these functional groups reduces. The spectrum flattens even further when the

temperature is increased up to 600 °C, resulting in almost complete reduction in absorption bands for O-H, C=O, and C-H. This finding is consistent with results obtained from FTIR studies on date seed pyrolysis, where the loss of polar groups like –OH and -C=O groups was confirmed.

5.4 Results

In the process of developing nanoparticles, there have been numerous modifications done to approach the 1-100 nm target range. There were several processes involved, from collecting palm dates until producing samples that will undergo characterization tests. The pretreatment process of palm dates is seen as significant as pyrolysis and ball milling, with improved procedures for cleaning and drying the surface. Despite all experimental setups and approaches that were geared towards getting the desired properties, mainly nano size, varying conditions for the intermediary stages result in different results. Controlling critical parameters such as critical temperature of pyrolysis, burning time, and pretreatment, including stirring with NaOH solution and drying using the oven, affect the powder quality after grinding with a mortar and pestle, regardless of other parameters used in ball milling, including rpm speed, ball milling time, and presence of surfactant. Moving forward, here are some SEM images of powder samples without pretreatment, pyrolyzed at 300 °C (Figure 47) and 600 °C (Figure 48). While the first SEM image shows agglomerates of particles, the second one depicts graphite sheet-like layered bulky pieces, which have a porous surface and small humps. In either case, the sample powder is to be processed further to reach the 1-100 nm range.

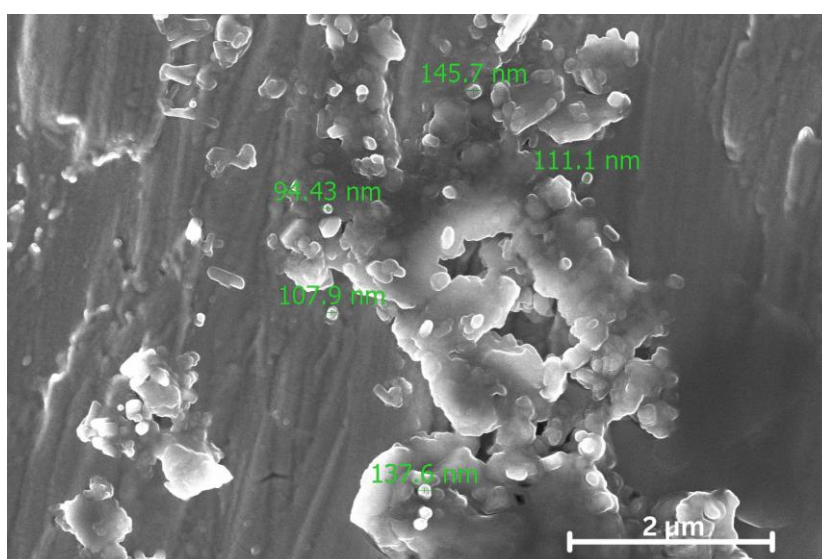


Figure 47. SEM on carbon powder burnt at 300 °C and 4 hr ball milling.

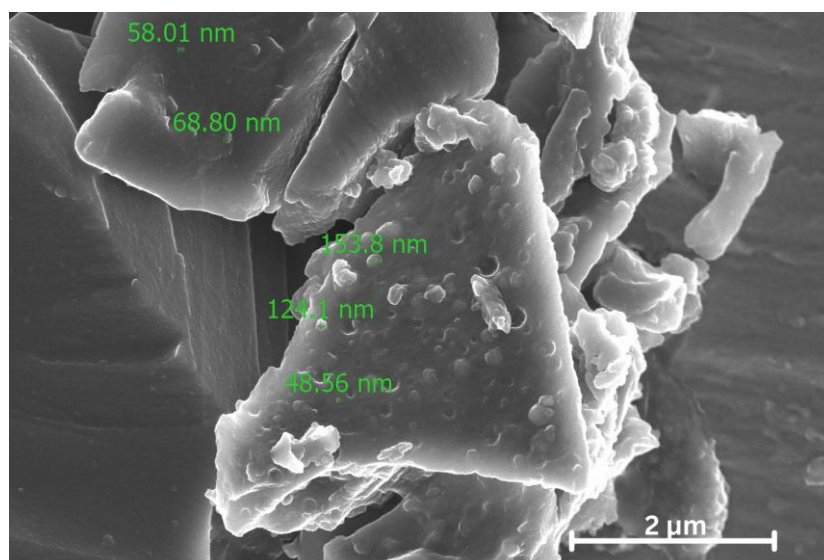


Figure 48. SEM on carbon powder burnt at 600 °C and 4 hr ball milling.

6 Conclusion

The goal of this research was to develop lightweight and high performance FMLs with eco-friendly manufacturing methods. Conventional acidic metal surface treatments present serious both environmental and operational concerns. In this work, a novel green chemical based anodizing process was proposed. In order to find the optimum conditions for an anodizing process, an experimental matrix with varying molar concentration and time was created. The decision on optimal condition was done based on a lap shear and peeling test. These tests allowed us to mimic the behaviour of fiber metal laminates without significant material waste. After finding the parameters for superior adhesion, the current method of surface treatment (HCl-etching) to the green alternative (KCl-anodizing) was compared on FMLs. The differences in the tensile performance were proven to be absent, while the flexural performance of the green alternative proved to be 64% better than the conventional method of surface treatment. This proved KCl-anodizing to be a superior method, not only because of its lesser environmental impact, but also due to its better mechanical performance.

In order to justify the obtained results, metal surface morphology was characterized by SEM imaging and profilometry. It was demonstrated that anodizing creates micro and nano scale pores on the metal surface that facilitate the mechanical interlocking effect. Moreover, the fracture analysis of FML after flexural test showed that strong adhesion in KCl anodized samples fails in a cohesive manner.

Since the FML based structures might operate in environmentally harsh conditions such as high temperature and humidity, the durability test was conducted. After 7 days in the salt spray chamber, HCl etched FML has significantly weakened in comparison to KCl anodized sample. This phenomena can be explained by the aluminium oxide layer that protects the aluminium from corrosion attacks. On the other hand, acidic treatment removes this layer and creates a rough metal surface that is exposed to salt spray attacks.

Overall, the findings of this research offer a strong basis for future research on eco-friendly FML production. Further research should focus on analyzing the impact properties and developing a numerical model. Moreover, there is a potential to fabricate nanoparticles from abundant date seeds.

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