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Experimental study on chemical resilience of glass reinforced polymer pipes for sewage applications

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ABSTRACT

A comprehensive understanding of composite pipe corrosion behavior is essential due to their exposure to diverse chemical environments. This study examines Glass Reinforced Plastic (GRP) composites, commonly employed in underground construction and industrial applications, with a particular emphasis on the rehabilitation of aging sewage pipelines. The investigation focuses on assessing the chemical durability of a multi-layer GRP material reinforced with polyester resin. The chemical resistance of individual layers and the composite as a whole are evaluated before and after immersion in various chemicals for periods of 30, 60, 90, and 180 days. The chemical environments are designed to replicate in-service conditions, including exposure to Sulfuric acid (H_2SO_4), Sodium hypochlorite (NaClO), Methyl ethyl ketone peroxide (MEKP), distilled water, Sodium chloride (NaCl), Propanone (C_3H_6O), Cobalt Octoate, and Dimethylacetamide (DMA). Mass loss measurements are used to evaluate the effect of chemical exposure. The maximum weight loss of 1.4% was observed after 180 days of exposure to acetone, with a noticeable increase after 60 days, likely due to material swelling. Therefore, it is recommended to limit the use of acetone for surface cleaning after assembling multi-piece pipe systems to reduce any material damage due to chemical exposure. The observations from this study assist in estimating the long-term structural health of composite pipes exposed to harsh chemicals found in sewage water.

Keywords: *Chemical exposure, Composite, Glass fiber, Weight loss, Corrosion*

1. INTRODUCTION

The efficient and safe transportation of wastewater from homes, businesses, and industries to treatment facilities heavily relies on pipelines [1]. These underground networks of pipes have a vital role in ensuring the proper disposal and management of sewage, preventing water sources from being contaminated and safeguarding public health. However, these pipelines can deteriorate over time due to factors like aging, corrosion, ground movements, and increased usage [2]. To address these issues, many researchers have examined the mechanical and chemical characteristics of coupon specimens made from pre-impregnated glass-fiber epoxy resin when exposed to high temperatures and pressures in aqueous environments. These studies aim to enhance the durability and longevity of such materials in real-world applications.

Ellyin *et al*. [1] reported that the moisture diffusion in tubular specimens was slower than that in the coupons. Once penetration occurs, moisture swiftly diffuses along the fibers, quickly infiltrating the resin and causing degradation in the matrix, reinforcement, and the interface between the fiber and matrix [4]. As a result, the flow capacity of the pipelines is restored. The durability of the composite pipes makes it effective in both commercial and industrial applications and is widely being used around the world for underground construction works [5]. Composite pipes have emerged as a promising solution for industrial applications such as underground construction, rehabilitation of sewers, offering several advantages over traditional materials [1, 2]. The installation of composite pipes can be accomplished using trenchless methods, such as slip-lining or pipe bursting, which minimize the need for extensive excavation [6]. The implementation of composite not only minimizes disruption to the surrounding environment and communities but also significantly lowers labor and restoration costs. Additionally, the pipelines made from composite materials exhibit excellent hydraulic properties, promoting a smooth and efficient flow of wastewater [7]. Their interior surfaces are designed to reduce friction, thereby preventing the accumulation of deposits, and minimizing the risk of blockages.

As the demand for sustainable and cost-effective sewer rehabilitation techniques continues to increase, composite pipes are poised to play an increasingly crucial role in ensuring efficient wastewater management while minimizing disruptions to communities and the environment. Fouad *et al.* [8] investigated the solid particle erosion behavior and wear mechanism of commercial epoxy based unidirectional glass fiber reinforced plastics (GFRP) composites. Erosion behavior was assessed in the study at different impingement angles (30°, 60°, and 90°) while

considering variations in erosion time and pressure. The highest erosion loss was observed at a 60° impingement angle. The use of chemical-resistant composite pipes minimizes maintenance and replacement costs associated with corroded or damaged pipes. These pipes require fewer repairs, inspections, and replacements, leading to long-term cost savings for industries and infrastructure projects. Their chemical resistance helps maintain the physical and chemical properties of the transported fluids. Cousin *et al.* [9] investigated the chemical durability of different carbon, basalt, and glass fibers, immersing the fibers in four types of solutions involving acid, saline, alkaline, and deionized-water conditioning methods. They observed fiber mass loss, surface damage, and changes resulting from chemical reactions through weight-loss measurements and scanning electron microscopy. A performance criterion was devised to categorize fiber performance as very good, good, fair, or poor. The findings indicated that carbon fibers displayed superior chemical resistance compared to basalt and glass fibers, as evidenced by weight loss and the presence of chemical reactions.

Liu *et al.* [10] conducted a study on the chemical composition, morphology, and properties of basalt fiber, investigated interface issues, and explored modification techniques for composites. They ultimately presented the potential applications of basalt fiber in the electrical materials sector, aiming to offer valuable insights for the future application and promotion of Basalt Fiber Reinforced Polymer (BFRP). Kim *et al.* [11] investigated the degradation of GFRP composites using an accelerated aging method and assessed its impact on the tensile properties. They exposed two types of E-glass/vinyl ester rods to moisture, chloride, alkali, and freeze-thaw cycling conditions for a duration of up to 132 days. Additionally, to examine the micro-level degradation of the GFRP composite, they fabricated and tested strand-type E-glass/vinyl ester rod specimens. The test results revealed a notable reduction in the tensile properties of the GFRP rods following the conditioning process, attributable to the degradation of the GFRP. Ahmad Sawpan *et al.* [12] investigated the long-term durability of GFRP bars by exposing them to an alkaline solution and natural weathering for a decade. Their findings revealed that water absorption plasticizes the polymer matrix, resulting in a significant reduction in flexural modulus, strength, and transverse shear strength. Among these, flexural strength was the most affected, with a reduction exceeding 20%, attributed to the weakened bond between fibers and the matrix. Similarly, Radzif *et al.* [13] conducted chemical endurance tests on thermoplastic particulate composites reinforced with milled carbon fiber powder. They observed that chemical immersion caused surface macrocracks and erosion. The degradation effects were exacerbated when the samples were subjected to a combined hygrothermal environment.

While some research has explored the short-term impacts of chemical attacks on composites, there has been limited investigation into how extended exposure to real-world conditions might influence the long-term properties of these materials. In this study, real-world conditions are opted and are mimicked for examining the chemical resistance of the individual layers that constitute the sandwich GFRP composite. Weight loss and tensile test samples were manufactured and subjected to rigorous exposure environments specified by different testing regions throughout the development and testing phases of the products, enabling the assessment of their performance and durability. California Greenbook [6] for sewage rehabilitation and Australian standard AS 3572.2 [14] have created a chemical resistance guide to assist engineers in selecting the different chemical environments in which the weight loss and tensile testing samples should be exposed to. These specimens are exposed for varying lengths of time at a constant temperature of 25° (+/−2°).

2. MATERIALS AND METHODS

2.1. Materials

This study focuses on the examination of multi-segmented GFRP pipes that have a multi-layered sandwich structure. The manufacturing process involved a manual layup method [15]. The wall of the sandwich GRP pipe comprises different layers, as illustrated in Figure 1. The description and purpose of each layer is as shown in Table 1. All layers, including the surface layer, are made up of glass fiber mats that have been impregnated with Isophthalic resin Crystic 491E. The core of the pipes is composed of blended sand, which has been impregnated with Dicyclopentadiene (DCPD) resin Crystic 2-451. Both isopthalic and DCPD resins were procured from Scott Bader, UK. The reinforcing glass fibers were sourced from different manufacturers. The surface layers (C-glass) were procured from Owens Corning in the USA. The UDM layers (E-glass) were supplied by SKAPS Industries in the USA. The CSM layers (E-glass) were obtained from Sichuan Weibo New Material Group Co., Ltd. in China.

The multi-segmented pipes are joined together using a tongue-in-groove junction (TGJ). To assemble the pipe, the top portion known as the crown is brought down and connected to the bottom section called the invert. Prior to inserting the crown into the invert, a 65% Adhesive-Resin-Fiber (ARF) mix [16–17] is applied to the tongue and groove area. The two segments are promptly joined together right after applying the adhesive, and they are then left to dry for a minimum of three hours. In the current study, individual layers of Isopthalic resin, surface layer, CSM and UDM

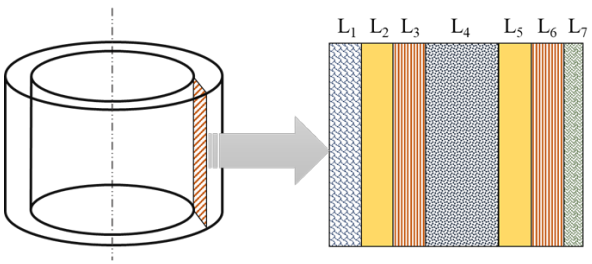

Figure 1. Constitution of the wall of the panel used to construct sewage pipe

Table 1. Constituent layers of the pipe wall [16]

Layer No.	Purpose	Reinforcement
L1: Surface layer	Assures a smooth surface resistant to wastewater and rainwater	Surface tissue in type C-glass
L2: Barrier layer	Contributes to both the structural aspect and the chemical resistance of the pipes	Chopped strand mat (CSM)
L3: Interior layer	Contributes to both the structural aspect and the chemical resistance of the pipes	A layer of CSM and unidirectional mats (UDMs) or bi-directional mats (BDMs)
L4: Core	The core layer contributes to the structural aspect of the pipe, mainly the pipe stiffness	Blended sand and calcium carbonate
L5: Intermediate layer	Contributes to the structural aspect of the pipe	A layer of CSM and unidirectional mats (UDMs) or bi-directional mats (BDMs)
L6: External Structural layer	Contributes to both the structural aspect and the chemical resistance of the pipes	Chopped strand mat (CSM)
L7: External layer	This layer contributes to the Shear bond between the exterior of the pipe and the annular grout	Silica sand

layers, blended sand and the ARF 65% adhesive samples were prepared using hand layup and compression molds. Furthermore, composite specimens with the specified layering sequence were fabricated using the same procedures.

2.2. Methodology

2.2.1. Visual Inspection

Each weight loss coupon sample is assigned to a unique serial number. Detailed information about the sample's material composition and the specific chemical exposure it underwent is recorded for reference. Visible signs of degradation, corrosion, discoloration, blistering, cracking, swelling, or any other physical changes on the surface of the samples are carefully observed. To assess the extent of damage caused by the chemical exposure, a comparison is made between the current appearance of each weight loss coupon sample and its original condition.

2.2.2. Gravimetric Experiments for Chemical Resistance

Weight loss samples were manufactured and subjected to testing under several strict exposure environments, simulating conditions utilized in different regions throughout the development and testing of products. These exposure environments were applied in this investigation, as illustrated in Figure 2. The California Greenbook for sewage rehabilitation and the Australian Standard AS 3572.2 offer a chemical resistance guide to support engineers in selecting suitable chemical environments for testing. This guide outlines recommended chemicals to which weight loss samples should be exposed, ensuring that materials used in sewage rehabilitation are evaluated under realistic and relevant conditions. The goal is to assess their durability and corrosion resistance across various environmental scenarios. The chemicals used in the test are listed in Table 2 and can be categorized into two groups. The first group represents the in–service conditions, simulating the chemical environment typically found in sewage water. The second group consists of chemicals used during the assembly process of a multi-piece pipe, which may remain as residue and enter the sewage environment after the assembly is completed. This categorization helps in evaluating the composite material's resistance both to operational conditions and potential chemical exposure during the installation process. Machined ASTM D543 [18] 25 mm × 75 mm individual layer test coupons and composite test coupons were conditioned in a mechanical convection oven for seven days at a constant weight and temperature of 43° ± 2° C prior to cooling for three hours in a desiccator. From a manufacturability standpoint, the thickness of individual layers varied. CSM, USM, and surface layers were less than 1 mm thick, while ARF, Crystic 491E, and blended sand samples had a thickness of approximately 4 mm. Composite samples, incorporating all the individual layers as detailed in Table 1, had an overall thickness of 25.4 mm. The weight of the individual layer test coupon and composite test coupons were recorded prior to immersion. The sample conditioning was performed both before and after the AS 3572.2 prescribed period of 30, 60, 90, 180 days. After submersion in the solutions for the specified time, the coupons were removed and tested in accordance with Greenbook Standard Specifications to determine physical properties and weight change values.

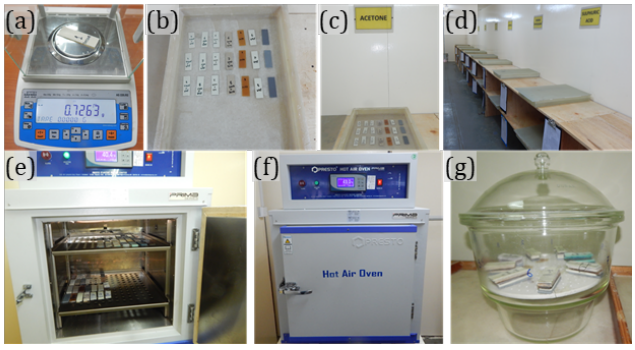

Figure 2. Pictures from the gravimetric tests. (a) Sample weight measurements. (b–c) Different specimens in Acetone solution. (d) Trays for different chemical environments. (e–f) Conditioning of samples in the heating oven. (g) Desiccation of samples

Table 2. Categorization of the chemicals used in the test

Sewage Ingredients	Assembly Process Residues
25% Sulfuric Acid (H_2SO_4)	2% Methyl Ethyl Ketone Peroxide (MEKP)
5% Sodium Hypochlorite (NaClO)	6% Cobalt Octoate
100% Distilled Water	6% Dimethylacetamide (DMA)
50% Sodium Chloride (NaCl)	50% Propanone (C_3H_6O)

3. RESULTS AND DISCUSSION

3.1. Effect of Chemical Exposure on Individual Layers

The weight loss data as a function of time for different layers immersed in the chemicals are presented in Figs. 3–7. Based on the data obtained, we can distinguish the impact of different chemicals on the materials into three categories: weight increase, weight decrease, and weight constancy. It is observed that all the layers except for the blended sand layer gain weight when they are exposed to MEKP, Cobalt Octoate and DMA. MEKP, functioning as a curing agent, can permeate the resin matrix, resulting in weight augmentation. Similarly, Cobalt Octoate serves as a catalyst during resin curing, facilitating further polymerization or cross-linking, consequently enhancing weight. The DMA helps the catalyst to start the chemical reaction and cure the resin faster. Therefore, absorption of MEKP and the catalytic role of Cobalt Octoate and accelerating role of DMA are contributing factors to the observed weight gain in all the layers. The likely reason is that, at the individual layer level, MEKP (as an initiator), DMA (as an accelerator), and Cobalt Octoate (as a catalyst) promote more effective cross-linking within the resin matrix. This enhanced cross-linking increases the stiffness of the layers and induces shrinkage, which leads to the folding of the samples, as illustrated in Figure 5.

When subjected to sulfuric acid and sodium hypochlorite, isophthalic resin (see Figure 3) samples experience weight reduction. Sulfuric acid initiates hydrolysis within the resin matrix, leading to the breakdown of ester bonds and fragmentation of polymer chains, resulting in weight loss. Similarly, sodium hypochlorite, functioning as an oxidizing agent, induces oxidative reactions that degrade the resin's molecular structure, causing weight decrease. The mechanism of the corrosion process is an ion exchange reaction in which metal cations associated with the glass fiber such as Ca^{2+} and Al^{3+} are replaced by acidic proton H^+ from the acid medium [19]. The chemical reaction can be summarized as Equation (1):

$$\overline{C^{n+}} + nH^+ \Leftrightarrow n\overline{H^+} + C^{n+} \quad (1)$$

Here $C^+$is either Ca^{2+} or Al^{3+} and the bar denotes the association of ions with glass phase. The size of newly associated protons with the glass phase is much smaller compared to the cations C^+ , therefore, there may be gaps created on the surface in the form of micro-cracks. The ion exchange process can be described by a second-order kinetic equation, where the rate of ion leaching is proportional to the square of the leached ion concentration. By fitting the weight loss data to the integrated equation [20], a weight loss percentage at the equilibrium (long term exposure) can be determined. The integrated rate equation is given as Equation (2):

$$\Delta W = \frac{at}{b+t} \quad (2)$$

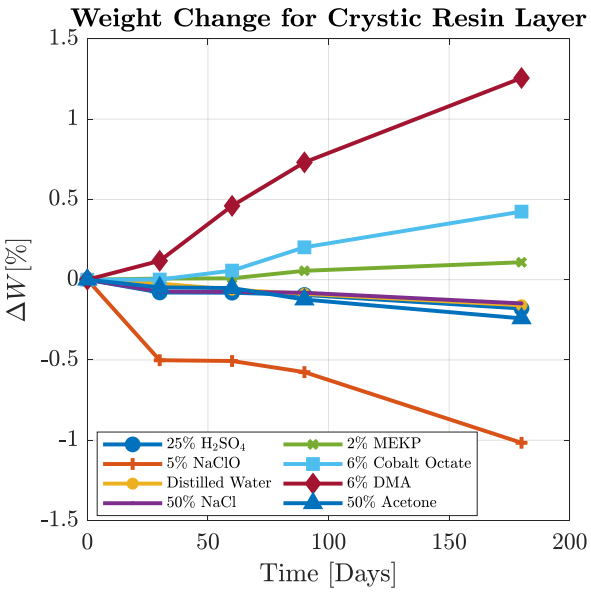

Figure 3. Weight Change (%) for Crystic 491E resin

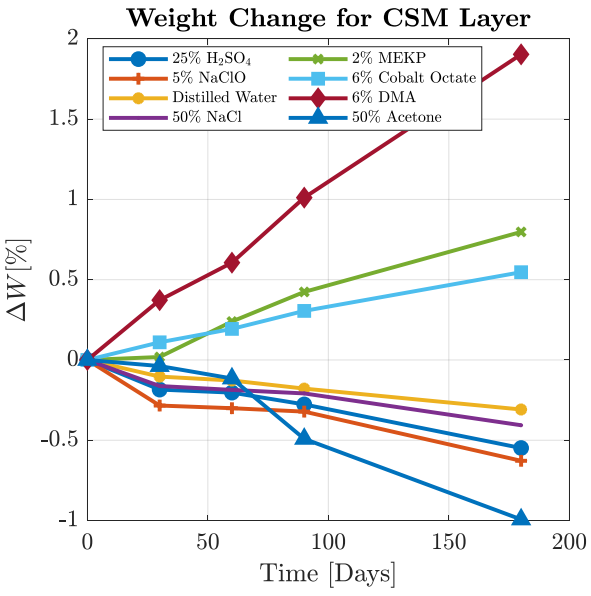

Figure 4. Weight Change (%) for CSM impregnated with Crystic 491E resin

Figure 5. Folding of samples after exposure to manufacturing process residue chemicals except acetone and (b) Acetone

where constant a is the weight loss at the equilibrium ($t = \infty$) and b denotes the reaction rate. Based on the experimental data the blended sand layer shows the highest weight loss (~4%) and the next highest weight loss is predicted in the CSM layers at ~2%.

Additionally, prolonged exposure to propanone, acting as a solvent, dissolved or softened the resin matrix, leading to weight loss as resin constituents are dissolved. Similar weight reduction trends are observed in chopped strand mat (CSM) (see Figure 4) and unidirectional mat (UDM) (see Figure 7) laminated with isophthalic resin when exposed to sulfuric acid and sodium hypochlorite. Although weight change trends are similar in CSM and UDM layers, a few chemicals show noticeable differences in the amount of weight change. For instance, exposure to NaOCl resulted in approximately 1.25% weight loss in UDM layers, whereas the CSM layer experienced a comparatively lower weight loss of only 0.5%. The effect of acetone was more severe on the CSM layer, with a weight loss of 1%, compared to the UDM layer, which showed a weight loss of 0.5% after 180 days of exposure. Additionally, the CSM layer exhibited significantly higher weight gain (2%) compared to the UDM layer (0.4%) when exposed to DMA. These variations in weight loss or gain can be attributed to the architectural differences between the layers. The CSM layer is composed of chopped fibers with lengths less than 1 mm, resulting in open fiber ends being exposed to chemicals on all surfaces of the sample. In contrast, the UDM layers consist of fibers aligned in the longitudinal direction, with open fiber ends exposed to chemical agents only on the two surfaces perpendicular to the longitudinal direction. The weakening effect of these chemicals on the resin coating or fibers causes disintegration and detachment, contributing to weight loss. Prolonged exposure to propanone also results in weight decrease due to solvent action and surface erosion, further exacerbating resin breakdown and weight reduction. For blended sand impregnated with DCPD resin (see Figure 6), weight loss occurs upon exposure to sulfuric acid, sodium hypochlorite, and propanone. Sulfuric acid and sodium hypochlorite prompt resin degradation and dissolution within the sand blend. Propanone, functioning as a solvent, can dissolve or soften the resin within the sand, resulting in weight reduction.

Chemicals such as distilled water and sodium chloride do not induce significant weight changes in the materials. While prolonged exposure to these substances may cause minor physical alterations like surface erosion or roughening, the overall weight of the materials remains relatively stable. This suggests that these materials exhibit resistance to the effects of these chemicals and can endure prolonged exposure without significant degradation or alteration. The ARF 65% specimens (see Figure 8) maintained their physical form and configuration across various chemical environments.

This stability suggests a robust resistance to chemical degradation, with weight fluctuations being insignificant. Furthermore, fungal growth was observed in propanone trays, though no major physical changes occurred, aside from slight folding of samples due to moisture uptake.

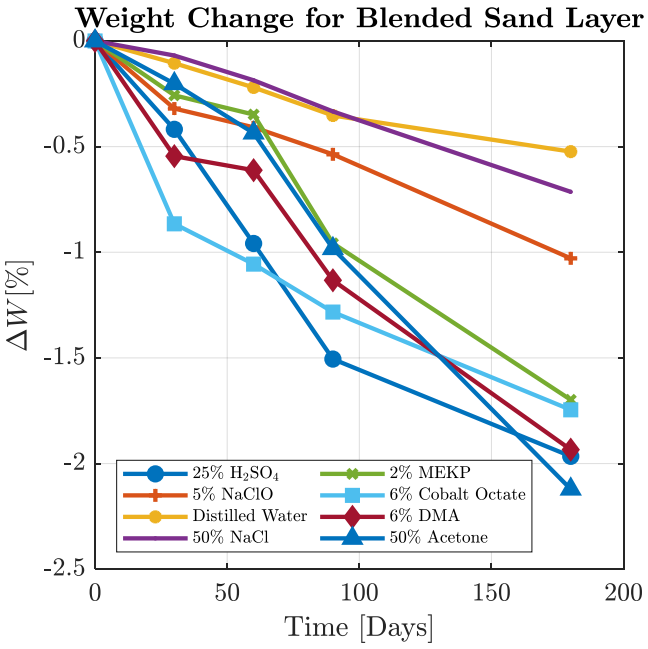

Figure 6. Weight Change (%) data for blended sand impregnated with DCPD resin

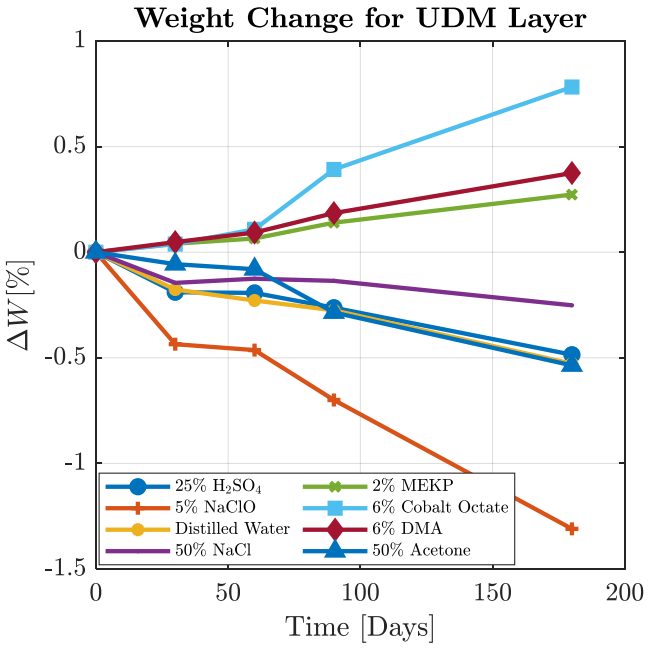

Figure 7. Weight Change (%) data for UDM impregnated with Crystic 491E resin

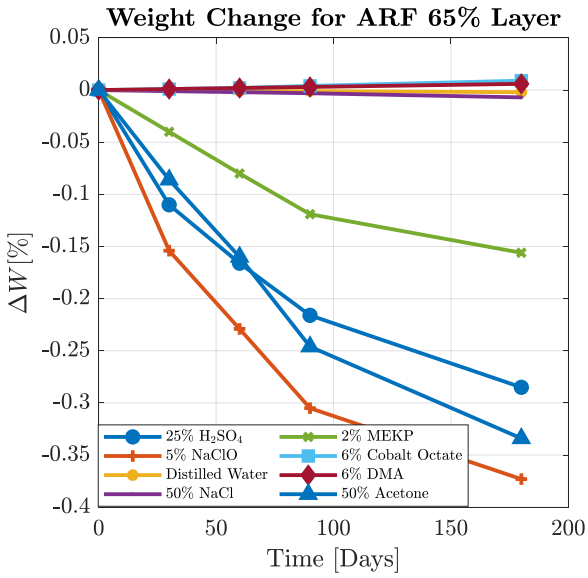

Figure 8. Weight Change (%) data for ARF 65%

3.2. Effect of Chemical Exposure on Composite

The change in weight (%) for the composite sample for all the chemical environments is shown in Figure 9. For all tested chemicals, the reduction in weight exhibits a consistent pattern. The data indicates that exposure to acetone results in significantly greater weight loss compared to the other chemicals. Specifically, at 180 days, the weight loss due to acetone exposure is approximately 1.4%, whereas the weight loss for all other chemicals ranges from 0.04% to 0.16%. Notably, the rate of weight loss during the initial 60 days is relatively lower compared to the subsequent period beyond 60 days. This phenomenon can be attributed to the swelling observed in the samples, which affects the diffusivity of both individual layers and the sandwich composite material.

The weight loss percentage in composite samples is significantly lower compared to that of the individual layers. This reduced weight loss can be attributed to the multi-layered architecture of the composite, which minimizes the exposure of internal layers to the chemical environment. Only the inner surface and outermost layers are directly exposed, while the intermediate layers are only exposed through their thickness, typically less than 1 mm, except for the blended sand core. Consequently, the observed weight change primarily originates from the top and bottom layers. However, when calculating the percentage weight loss, the total mass of the composite is substantially higher than that of individual layers, reducing the overall percentage loss. Given that these layers are resin-rich, exposure to acetone, a potent organic solvent, induces swelling of the resin. This swelling can generate micro-cracks, facilitating the penetration of acetone into deeper layers of the composite. Once inside, the acetone disrupts the resin cross-linking network, leading to a loss of stiffness and compromising the structural integrity. Over time, this degradation can significantly impair the long-term load-bearing capacity of the composite, making it vulnerable to mechanical failure under sustained stress. No significant physical changes such as color alteration, configuration change, macro-cracks, or pore formation were observed in any sample after the 180-day exposure period.

4. CONCLUSION

This study emphasizes the significant influence of chemical exposure on the long-term performance of materials, particularly in environments where sewage chemicals and assembly residues are prevalent.

Individual Layers: The individual layers exhibited three types of behavior in response to chemical exposure: weight increase, weight decrease, and weight stability. Weight gain

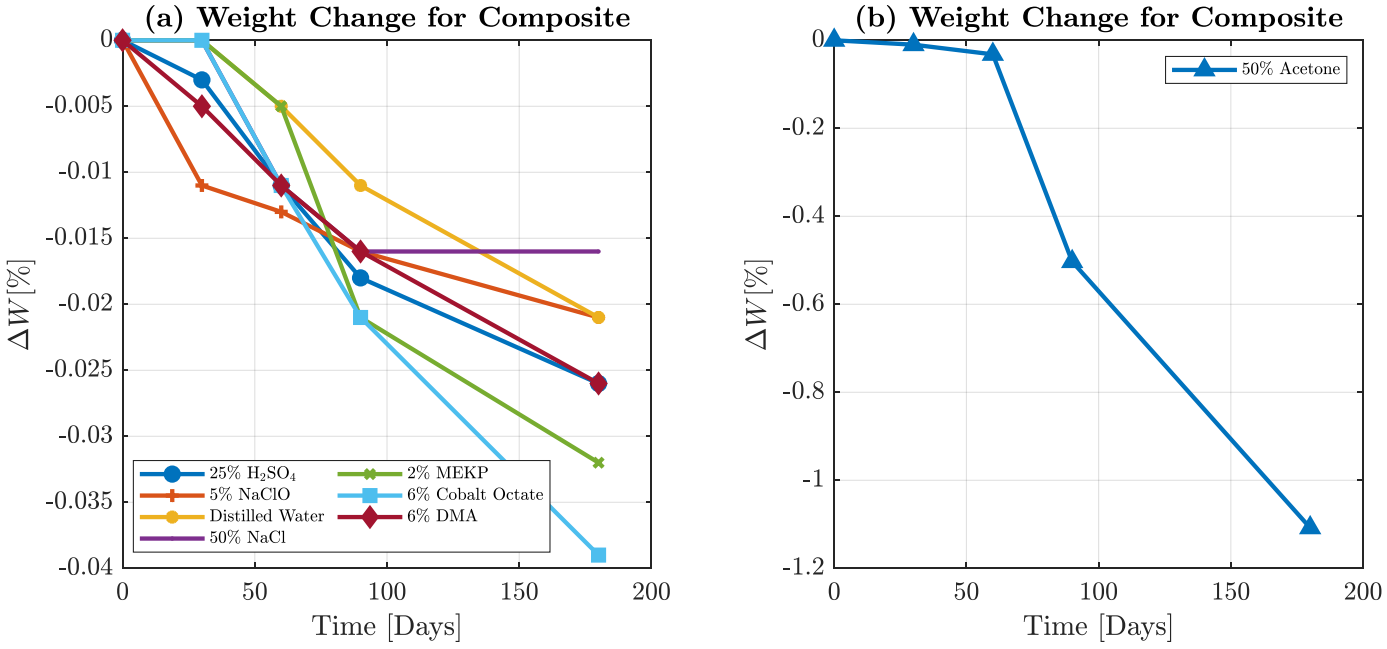

Figure 9. Weight Change (%) data for composite pipe samples after exposure to chemicals.
(a) All chemicals except acetone and (b) Acetone

was predominantly observed when the samples were exposed to assembly process residue chemicals, primarily due to resin absorption and enhanced polymerization. Sulfuric acid, sodium hypochlorite, and propanone led to significant weight loss, especially in isophthalic resin layers, due to hydrolysis, oxidation, and solvent action. Materials exposed to distilled water and sodium chloride maintained stable weight, indicating good resistance to these chemical environments. ARF 65% specimens exhibited minimal weight change across all chemicals, demonstrating the strong chemical resistance of the newly developed adhesive system for rehabilitation work.

Composite: Unlike individual layers, even the assembly process chemicals showed weight loss in the composite samples. The highest weight loss (1.4%) was recorded after 180 days of exposure to acetone, with a notable increase in weight reduction after 60 days due to material swelling. It is recommended therefore to use acetone is moderation while cleaning the surface after the assembly process for a multi-piece pipe system. The residue acetone is detrimental to the rich resin layers. Despite some weight loss, no significant physical degradation (such as macro-cracks or color changes), aside from minor twisting and folding due to hygral stresses, was observed after 180 days, suggesting that the material's structural integrity was largely maintained.

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