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ANALYSES OF PULTRUDED POLYESTER/ E-GLASS COMPOSITES SUBJECTED TO ENVIRONMENTAL DEGRADATION

A Thesis
presented in partial fulfillment of requirements
for the degree of Master of Engineering
in the Department of Engineering

by

Cameron Bosley

August 2017

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ABSTRACT

This paper evaluates the response of experimental mold releases for a pultruded glass fiber reinforced polymer (GFRP). The initial assessment criteria include comparisons between pull forces and surface appearances for each mold release. Composites fabricated using experimental mold releases that exhibited acceptable properties for both were used for further mechanical characterization that included environmental aging for two different exposure lengths (500 hours and 1000 hours) in five different environments: UV, bleach, hydrochloric acid, distilled water at 125°F, and room temperature salt water. Three-point flexural bending and short beam strength tests were undertaken to determine the effects these environments would have on the composite when compared with the strengths of an unaged sample. This principally will determine how well the matrix is able to distribute the load to the stronger fibers after undergoing environmental degradation. This study found that the bleach environment had the most damaging effects for both flexural and short beam strengths. Notable increases in both flexural and short beam strength were seen for UV, elevated temperature distilled water, and salt water exposures which indicates the poor bonding between the fiber and the matrix material and possible secondary curing of the composite. Additional DMA and TGA tests were completed on unaged specimens to determine the glass transition temperature and onset temperature values. The mechanical properties for the experimental and commercially available mold releases did not vary significantly and suggests a closeness in chemical composition.

LIST OF ABBREVIATIONS OR SYMBOLS

GFRP	Glass Fiber Reinforced Polymer
UV	Ultraviolet
T_g	Glass Transition Temperature
FTIR	Fourier Transform Infrared Spectroscopy
T_{onset}	Onset Temperature
HCl	Hydrochloric Acid
phr	Parts Per Hundred Resin
TGA	Thermogravimetric Analysis
DMA	Dynamic Mechanical Analysis
°F	Degree Fahrenheit
°C	Degree Celsius
Hz	Hertz
min	Minute
in	Inch
MPa	Megapascal
ksi	Kilopound per Square Inch
MR	Mold Release

ACKNOWLEDGEMENTS

I would like to express gratitude towards many people that helped in the culmination of this project. First and most importantly to my advisor, Dr. Ellen Lackey, for her knowledge, constant support, and willingness to help with any problem. Without her patience and guidance, this thesis would not have been possible. Similarly, I would like to thank Ryan Hedgepeth and Matt Lowe for their assistance in the completion of this and many other projects. I would also like to thank Dr. Arunachalam Rajendran and Dr. James Chambers for their assistance and guidance in both my academic and personal lives. They have dedicated more time and effort to assisting me than they should ever be asked to do. Lastly, I would like to thank my friends and family for their encouragement and support.

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INTRODUCTION

Composites are a branch of materials defined by the existence of two or more constituent parts in different phases. They are typically known for their strength to weight ratio and for their applications in infrastructure. Concrete and wood are two well-known composites used primarily in these areas. However, polymeric composites (hereafter designated as composites) have seen an increase in usage with the advent of new fibers and manufacturing processes. Carbon fibers are often used in aerospace and automotive industries, aramid fibers are used in military applications, and glass fibers are used in industrial applications. There are numerous methods used to fabricate composites, and pultrusion is one of those most commonly utilized for production of fiber reinforced polymers in infrastructure.

Background

Pultrusion is a continuous manufacturing process used to create constant cross section composites. Continuous fibers are pulled through a heated die after being impregnated with a resin mixture. These can be mixed with woven mats and chopped fibers to add strength in the transverse directions. Typical fibers used are E-glass and carbon fibers, but other fibers such as aramid, basalt, and bio based fibers can be utilized. Common thermoset resins used are unsaturated polyester, vinyl ester, polyurethane, and epoxies as GFRPs. Thermoplastic resins can be used in pultrusion as well but are not as prevalent. Figure 1 is a representation of the pultrusion process.

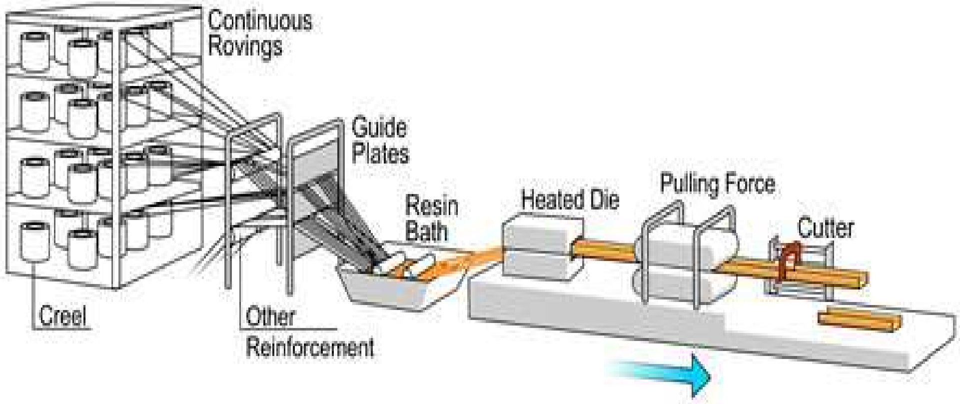


Figure 1 - Pultrusion Schematic [1]

Factors that affect the resin properties include catalysts, fillers, mold releases, and styrene content. Additional crucial process variables include the speed at which the process is operated as well as the die temperatures. These factors, combined with the aforementioned fiber and resins, provide a characteristic pull force for the composite part which determines the ease at which the part can be fabricated. Ideal characteristics would be a high manufacturing speed with exceptional mechanical properties. However, the integrity of the composite is dependent on the bonding between the fibers and the resin. This is especially important for pultrusion as the degree of cure is determined by multiple factors. Establishing the bond between the fibers and resin is paramount to understanding how the composite will behave in complex loading scenarios. This requires testing that specifically targets material properties dependent on the fiber/ matrix interface. If structurally sound, these pultruded parts allow for composites to take the place of traditionally metal materials while offering corrosion resistance and weight reduction. The need for cheap and reliable composite parts can be seen in industries ranging from aerospace and automotive to medicine. Because light weighting and efficiency have increasing importance in nearly every field, a comprehensive understanding of the nature of polymeric composites is required. This is only amplified when the applications may subject the composites to different environments affecting the material properties.

Project Overview

The first part of this paper will focus on the comparison between multiple proposed release agents with a standard, commercially available release agent. The factors that will be analyzed will be the average pull forces and surface appearances. These will be the two most important factors when determining if a given release agent should be considered as a possible substitute in place of its commercially available competitor and will be applied to each pultruded piece in order to determine the quality. This information is beneficial for companies that wish to drive down the cost of manufacturing while maintaining an appealing product.

The second part of the project will include mechanical testing results and comparisons of the effects environmental aging has on the matrix interaction with the fiber. The results will include multiple sources of environmental aging at two different time periods. The likely causes behind changes in strength are given and estimations are made based on data trends. Additional thermal and mechanical tests are added to provide supplementary information regarding the composite.

The beneficiaries of this work include companies or institutions with wishes for further research in the area of formulation of unsaturated polyester resins for pultrusion. The information contained in the report is also beneficial for industries ranging from automotive to home appliances as many manufacturing industries utilize polyester resins and glass fibers for their lightweight attributes and ease of construction. Additionally, this information will uncover some of the flaws that will be seen in environmental aging testing for pultruded polyester composites. Conclusions and recommendations are given for further research into this area.

THEORY

This section provides a background to the research. A terse introduction to the formation of the material is given while highlighting the significance it plays in the market. A literature review is also provided to show some of the previous work completed in this field. Additionally, calculations used for further material characterization are presented.

Overview

Unsaturated polyester resins are formed from the combination of dibasic organic acids and polyhydric alcohols [2]. Different ranges of polyesters are created by adjusting the amount of dibasic acid and polyhydric alcohol contents. Typical alcohols used are ethylene glycol or other glycols while the acid is generally phthalic acid and maleic acid. An example of polyester polymerization can be seen in Figure 2.

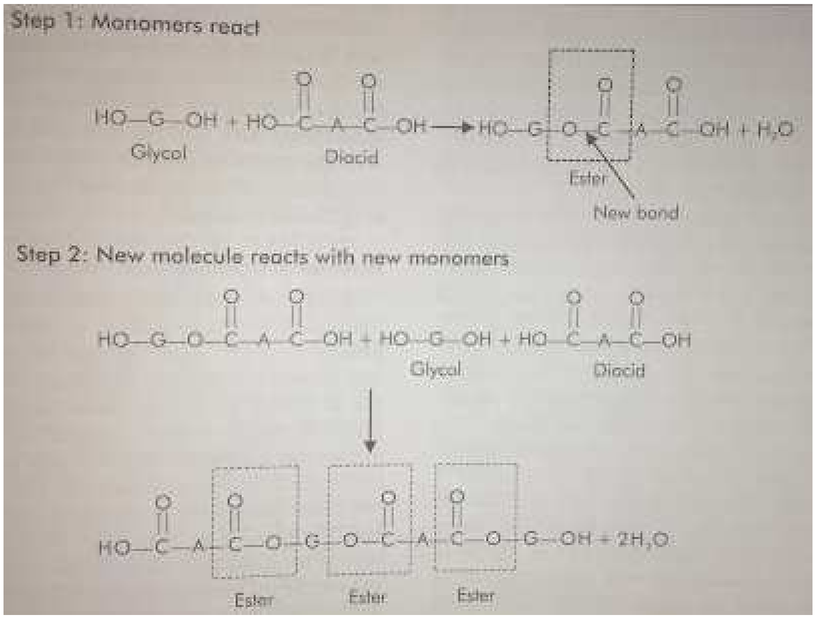


Figure 2 – Polyester Formation [2]

The hydrogen and oxygen atoms on the glycol join with the hydroxide and carbon on the diacid to form water and an ester group. This is repeated to create a chain of esters that results in the creation of the polyester. A significant advantage this type of resin system has over others is that the carbon double bonds allow for more opportunities to crosslink as opposed to epoxies or vinyl esters where the crosslinking occurs on the ends of the chains [2]. To decrease the viscosity, styrene is added along with catalysts that convert the initially liquid resin to a cured solid through crosslinking. Polyester resins are often considered inexpensive amongst thermoset resins but generally have lower mechanical performances. Figure 3 shows generic estimations for mechanical performances compared to the cost for some categories of thermoset resins. Similarly, Figure 4 highlights the price per unit weight and price per unit strength for different materials that are common in construction. When the resins are combined with fibers, the GFRP materials offer many opportunities due to the high strength to weight ratio. However, heat and moisture along with UV or chemical exposure can attack the bonds within the matrix, rendering the composite noticeably weaker and ineffective. Polyesters and vinyl esters are generally used for their resistance to most types of corrosion.

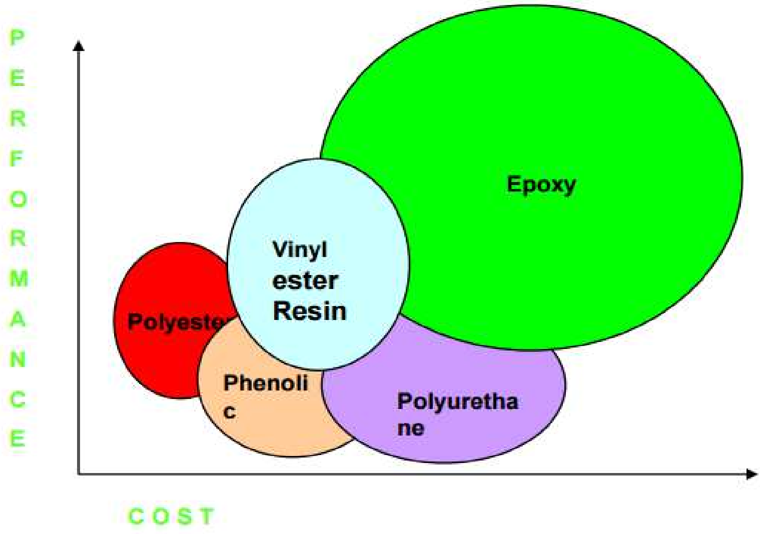


Figure 3 - Resin Mechanical Performance vs. Cost [3]

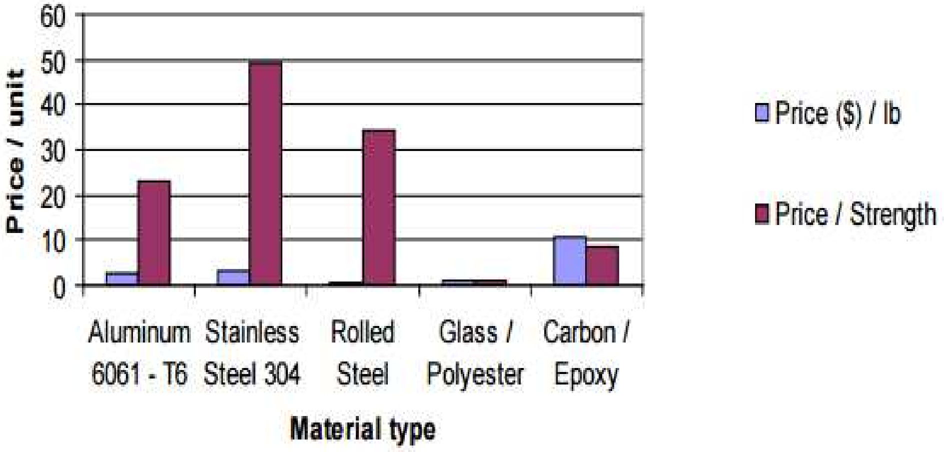


Figure 4 - Material Price Comparison [3]

Glass fibers are often used in composite design for their acceptable and predictable mechanical properties but perhaps most importantly – cost. Other fibers such as aramid and carbon fiber have higher specific strengths and specific modulus values [4], but they lack the affordability that glass fibers provide. The most common types of glass fibers include electric glass, strength glass, and corrosion resistant glass which are designated as E-glass, S glass, and C glass, respectively. The glass fiber used as reinforcement for this project was Owens Corning 366-AD-113. This is a type of E-glass that is specifically manufactured for pultrusion and filament winding with epoxy, vinyl ester, and polyester resin systems but can be also used for polyurethane and acrylic resins [5]. There is a silane based sizing on the fibers to ensure a more complete wet out. The E-glass used has a fiber tensile strength of 3815 MPa (553 ksi) and occupied roughly 67% of composite volume [5]. The void content of pultrusion is typically very low. This coupled with the use of polyesters (which tend to have higher shrinkage than epoxies) allows for a compact part with fewer opportunities for the resin to stick to the die; however, mold releases are typically added to pultrusion resin formulation including polyester resins, to help prevent the part from adhering to the die during the manufacturing process.

Literature Review

For long term usage, new materials must undergo significant testing before they can be used in structural or mechanical applications. There is a need to understand how the material will react when subjected to different environments. Because the properties of composites are dependent on multiple factors, additional data is required to determine the effects of the constituent properties.

With the exception of some short term organic solvents and post cure conditions, the general trend shown in literature is that environments have a negative effect on the mechanical properties of polyester resins [6-14]. These are generally dependent upon the values for T_g, as it correlates to increasing or decreasing plasticization and cross-linking. This was highlighted by Correia et al. in an experiment that indicated increased exposure in demineralized water and salt water caused greater decreases in the glass transition temperature until a point of 12 months where the T_g began to increase [13]. This experiment also showed an increase of glass transition temperature for QUV settings, indicating the possibility of secondary cure from UV light. Other studies were undertaken by the same group on pultruded isopthalic polyester resins where the composites were subjected to room temperature water, elevated temperature water, and QUV exposure. FTIR and mechanical analyses and were carried out intermittently during 264 days of exposure and revealed that room temperature water and elevated temperature water yield similar degradation of the composite while the QUV exposure did not significantly change the flexural strength. Similarly, the flexural modulus remained constant for each of the exposures across the testing period. The FTIR tests indicated no significant change was seen for the aforementioned exposures. However, some carboxyl groups were dissolved in the water indicating that the styrene content was decreased. The latter case was also true for the QUV exposure. The study concluded that the QUV degradation effects were confined to the outermost layer of material [15].

Another study of environmental degradation on pultruded polyester/ E-glass was done by Broughton. This examination unintentionally confirmed some of the findings of the previously mentioned studies by verifying that QUV exposure does not significantly affect the flexural modulus at six months of exposure while the flexural strength declined up to 16%[12].

Further studies were completed by Daly et. al on pultruded unsaturated polyester with 51.5% fiber volume weight. Their experiments included 120 days of immersion in hot (65°C) distilled water and sea water. As previously found, the moduli were not significantly affected by the exposure length. Flexural tests determined that delamination due to interlaminar shear stresses were the causes of failure after exposure to both environments. This led a 16% drop in flexural strength but an increase by 12% in deflection. The average short beam strength decreased 35% after exposure for both the salt water and elevated temperature distilled water while increasing the deflection by 50% [14].

Studies on the glass transition temperature and TGA onset temperature of polyester/ E-glass composites have also been performed [16-17]. These indicate a range of values for T_g and T_{onset}. The first study showed a T_g in the ranges of 117-142°C as well as a T_{onset} of 304°C while the second displayed its glass transition temperature at 162°C and onset temperature at 310°C. The difference in T_g values indicates the likely variance in resin systems used in each of the composites.

Calculations

In order to obtain data that can accurately represent the effect of environmental degradation, tests that are matrix dependent or fiber/ matrix interface dependent are expected to show more effects than those that are fiber dependent (i.e. tensile strength). This is due to the rule of mixtures which can be seen below in Equation 1

$$s_L^{(+)} = s_{f1}^{(+)} v_f + s_{mf1}^{(+)} v_m \qquad \text{Eq. 1}$$

where $s_L^{(+)}$ is the longitudinal tensile strength of the composite,

$s_{f1}^{(+)}$ is the longitudinal tensile strength of the fiber in the fiber's 1 direction,

v_f is the volume fraction for fibers,

$s_{mf1}^{(+)}$ is the longitudinal tensile strength of the matrix once it reaches the ultimate strain of the fiber and,

v_m is the volume fraction of matrix material.

This shows that the fibers will dominate this test, and thus small changes in the longitudinal tensile strength may not be indicative of matrix degradation. It should be noted that this equation is valid for the proposed experiment. If there were additional added randomly oriented fibers (as in the case of chopped strand mats), the estimation of tensile strength retention would not be valid [13].

The flexural strength as well as the short beam strength can be calculated using ASTM D2344 and ASTM D790 respectively. The tests were selected so that both a fiber dominated property (flexural strength) and a fiber/ resin interface (short beam strength) would be examined for this study. A representation of the test fixtures can be seen in Figures 5 and 6. The specimens were loaded at the mid-span of the upper surface. Both the three-point bending tests and the short beam test result in a mixed mode of loading, with bending stress dominating in the three-point bending test due to the longer support span, and shear stress dominating in the short-beam test due to the shorter support span. An ideal failure for the flexural test would be from tension on the bottom surface and compression on the top. For a rectangular cross section, the maximum stress seen in the lower surface fibers can be calculated using Equation 2

$$\sigma_{11} = \frac{3PL}{2bh^2} \qquad \boxed{\text{Eq. 2}}$$

where P is the maximum load,

L is the length of the span,

b is the length of the base,

and h is the height [18].

A calculation for the maximum shear stress at the neutral plane can also be calculated using beam theory when the sample fails due to interlaminar shear. Equation 3 was used to characterize this

$$\tau_{31} = \frac{3P}{4bh} \qquad \boxed{\text{Eq. 3}}$$

where the nomenclature remains the same as the flexural stress calculation [19].

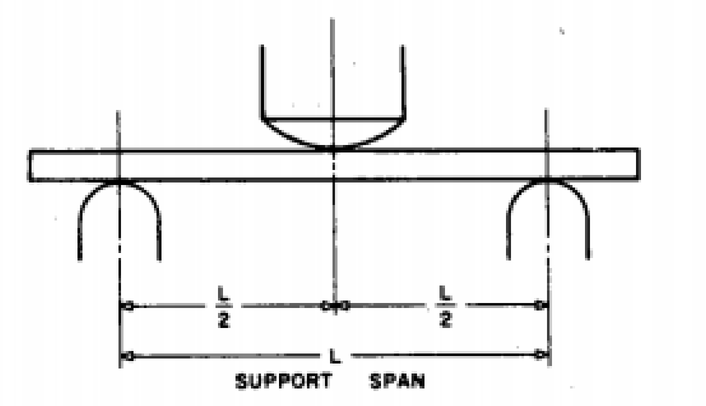


Figure 5 - Three Point Bending Test

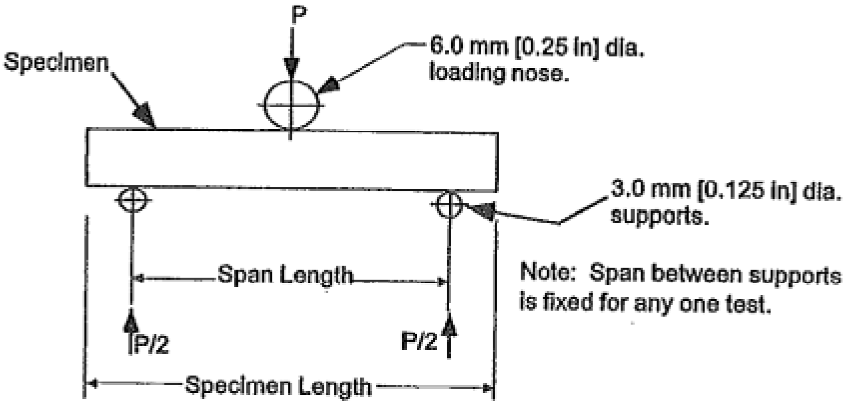


Figure 6 – Short Beam Shear Test

The failure modes associated with flexural tests on GFRPs can be seen in Figure 7. Using this, one is able to estimate the bonding of the matrix to the fibers. The optimal failure for the flexural test is in the outer fibers and is a consequence of the bending moment. This is represented by the simple tension in Figure 7 (a) and is typically the form of failure for many GFRPs. However, if the fibers do not bond well to the matrix and are therefore not supporting the majority of the load, failure modes such as (b) and (f) can be seen. Because the fibers do not bond well with the matrix, the load is not being evenly distributed to the lower fibers resulting in rupture towards the edge or on the inside of the sample. Often due to Poisson strains, these failures are not ideal for modeling or comparative analyses [4].

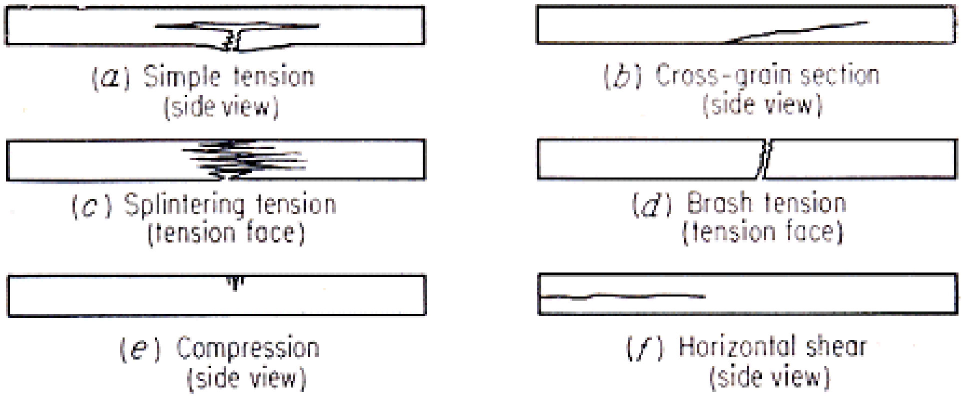


Figure 7 - Flexural Failure Modes [20]

The typical failure modes for short beam tests are given in Figure 8. Because the goal is to characterize the material with respect to its interlaminar shear, the first two failure modes are the most acceptable. If the span is too large or the specimen has been fabricated incorrectly, failure may be seen in a flexural manner.

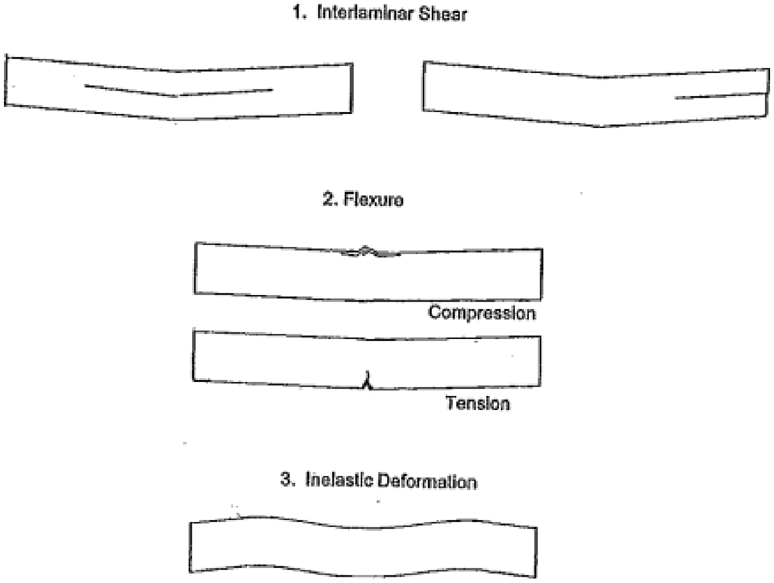


Figure 8 - Short Beam Failure Modes [19]

ENVIRONMENTAL AGING

This section highlights the environments and their probable aging effects on the samples. The environments were chosen based on multiple factors: previous research of exposure types, ease of testing, real world application, and significant acids/ bases. With the limited material, these experiments aimed at creating scenarios the composites would often undergo and situations that would be more detrimental to retaining fiber/ matrix based mechanical properties.

QUV

Photo-oxidation, commonly known as weathering, is the change in chemical or physical properties by means of absorbed radiation. Photo-degradation results from exposing the composite to UV light <350nm and is typically done by the sun. The reaction of UV creates free radicals by dissociation of the CH bonds which react with oxygen to create hydroperoxides [21-23]. The creation of hydroperoxides can create further degenerative products such as ketones and aldehydes. Free radicals will continue to be created even after the exposure to UV has stopped by means of propagation reactions. This means the process will continue until the creation of free radicals is no longer possible. Depending on the length of the polymer chain, the free radicals are rarely the cause of the decline for mechanical properties. The reduction stems from beta scission reactions that split the carbon-carbon bonds [21-23]. Creation of these free radicals is dependent on oxygen prevalence which indicates that the surface will be the primary affected region. Discoloration is also common from this reaction. In some cases, the UV can act as a secondary curing stage for the composite to achieve a greater crosslinking density. This is generally seen when exposure is short and temperatures are increased, but the mechanical properties will decline with increased exposure. In order to have a repeatable method to use to characterize photo degradation due to UV exposure, ASTM G154 “Standard Practice for Operating Flourescent Ultraviolet Lamp Apparatus for Exposure of Nonmetallic Materials” is used.

Shown is the setup for the QUV accelerated weathering environment using ASTM G154-12a to characterize this exposure [24]. For eight hours, the temperature was raised to 60°C and exposed to an irradiance level of 0.89 W/m^2/nm. When this was completed, a four-hour period of 50°C with no irradiance was observed. Both of these periods contained an exposure to condensation. This exposure condition is also specified in the Pre-Standard for Load and Resistance Factor Design of pultruded composites [25].

Figure 9 - QUV Exposure Machine

Bleach

For this exposure, a highly basic/ alkaline solution was required. To achieve this, a brand of common household bleach with 6% sodium hypochlorite and a pH of approximately 12.6 was used [26]. The solution attacks electron sources when a complete cure is not achieved by creating hypochlorous acid and hypochlorite ions [26]. If chlorine ions are present, they may bond with other compounds to create more dangerous compounds. Increases in temperature, fluctuations in pH, and moisture presence will also have an effect on how aggressively the bleach will attack the composite by exposing more free radicals. The bleaching also affects the coloration of the composite and can also expose fibers that have separated from the fiber/ matrix interface.

Hydrochloric Acid

Although vinyl esters and polyesters are effective at resisting common attack from acids, sulfuric acid and hydrochloric acid are particularly aggressive. Hydrochloric acid is easier to handle and purchase, and was thus selected as an environment. The hydrochloric acid degradation is dominated by osmosis due to the small size of the HCl molecule. This can form water soluble salts which can cause blistering if any moisture is retained within the composite [27]. Shown in Figure 10 is the setup for the HCl aging.

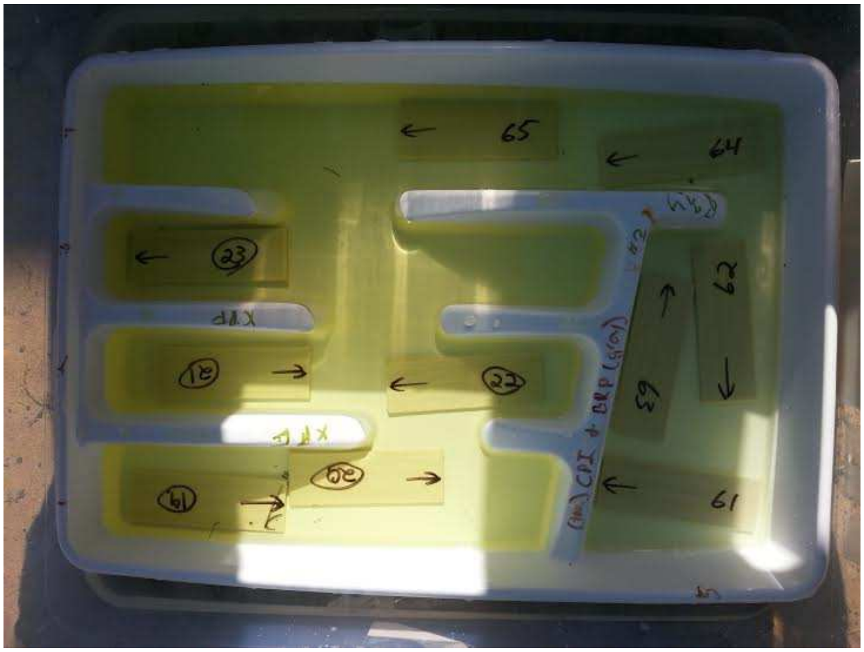


Figure 10 - HCl Exposure

Elevated Temperature Distilled Water

Changes in the moisture content and temperature affect the polymer matrix by giving a softening effect. The combination of these hydrothermal and hygroscopic effects can be modeled using a combination of elementary mechanics and Classical Lamination Theory [4]. This can be seen in equation 4

$$F_m = \frac{P}{P_0} = \left(\frac{T_{gw} - T}{T_{go} - T_0}\right)^n \qquad \text{Eq. 4}$$

where F_m is the degradation factor,

P is the strength after hygrothermal degradation,

P_0 is the original strength,

T is the predicted temperature,

T_{go} is the glass transition temperature of the reference temperature,

T_{gw} is the glass transition temperature for the wet matrix,

T_0 is the temperature at which the initial properties were tested,

and n is a curve fitting parameter dictated by the specific material.

This equation is combined with the previously tension equation to yield equation 5

$$s_L^{(+)} = s_{f1}^{(+)} v_f + F_m s_{mf1}^{(+)} v_m \qquad \text{Eq. 5}$$

which indicates that the matrix will only be affected. There is a degradation to the fibers, but it is considered negligible in most cases. Similar to the QUV exposure, these equations are generally valid if no secondary curing is present. A device that measures salinity was used to verify that the distilled water did not contain significant contaminants. In this exposure scenario, the water was heated to approximately 125°F for the duration of the exposure.

Salt Water

Exposure to salt water is similar to the distilled water but adds the further damaging effects of salt ions. As mentioned in the HCl section, a salt concentration can cause internal stresses that lead to blistering. As shown previously, the salt water and distilled water have similar effects for most exposure lengths. Applications of different loading types (thermal and mechanical) will increase the effect salt water has on the sample. The salt water was created by mixing distilled water with a commercial fish tank solution. The salinity was checked to ensure levels close to 32 parts per trillion were present. This is seen in Figure 11.

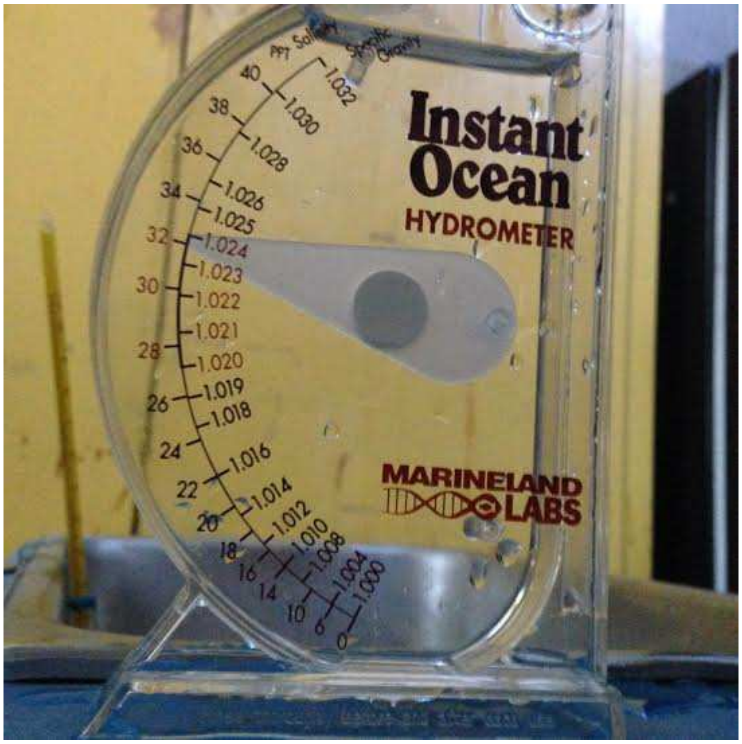


Figure 11 - Salt Water Salinity

METHODS

This section briefly describes the manner in which the composites were fabricated. It details the variables that were held constant during fabrication and introduces the components of the resin system. In addition, this section also details the data that was obtained during the testing.

Procedure

The first part of this project involved identifying the effects the release agents and an added pigment would have on the physical characteristics of the finished composite part. Four different mold release products and three different pigments were examined in these pultrusion manufacturing experiments. The ultimate goal was to identify mold release products which produced a smooth surface with a low pull force required to maintain constant composite construction while being able to manufacture different colored composites. The simplest method to determine the surface characteristics was to examine the edges of the composite post production. The pieces that exhibited a poor surface quality or exposed fibers were rejected from further mechanical testing. In order to directly compare the effects of release agents, all other factors were held constant with the exception of pigment variation. Table 1 shows the parts per hundred resin (phr) ratios for each variable while Table 2 indicates the die temperatures and pull speed. Specific phr values for each run will be indicated in further sections. The materials were mixed thoroughly via an air powered mixer in the same order in which they are listed in Table 1. Once mixed, the resin was placed in the bath and pulled by the PTI Pulstar 804 pultrusion machine at a constant rate of twenty-four inches per minute. When the first cured section reached the second puller, the materials exiting the die and those following were deemed to be an accurate representation of the cured composite and thus useful for pull force averages and mechanical characterization.

Table 1 - Resin Mixture Quantities

Material	PHR
Aropol 50440	100
Monomer, Styrene	9.50
Catalyst, Perkadox 16	0.2
Catalyst, Trigonox 121C75	0.6
Catalyst, TBPB Trigonox C	0.1
Low Profile Additive, Microthene FN510	2
Pigment	0.6
Filler, Calcium Carbonate Fine	16.7
Filler, Aluminum Trihydrate	10
Mold Release	3

Table 2 - Pultrusion Constant Parameters

Die Parameter	Value
Pull Speed (in/min)	24
Die Zone 1 (F)	285
Die Zone 2 (F)	305
Die Zone 3 (F)	295

After fabrication of pultruded composites using the commercial mold release as a control and one of the candidate mold releases, the second part of the project aimed to determine the effects different corrosive environments would have on the matrix dominated material properties for these two composite materials. This allowed for the comparison of the candidate and commercially available mold releases. The environments are discussed in the previous section. These environments were held constant for both five hundred and one thousand hours and tested for their mechanical properties. These were compared against the values taken with no environmental aging. The three-point flexural bending and short beam tests were selected as the best to understand the affected relationship between the environment and the composite. The standards utilized were the ASTM D790 and ASTM D2234 for the flexural strength and short beam strength respectively. The speed of the mechanical testing was set at 0.001 in/min. Further tests to determine glass transition temperature and maximum service temperature were also conducted and compared for the unexposed materials. Thermogravimetric analysis and dynamic mechanical analysis tests were performed in order to determine the effects of the mold releases on the maximum operating temperatures and glass transition temperatures. Experimental results can be easily obtained using DMA and TGA making the materials directly comparable. The importance of the characterization of these composites is due to the viscoelastic properties exhibited on even short temperature changes. This means that a small addition or subtraction of a constituent material can have a significant effect on the performance of the composite. TGA tests yield an accurate representation of the material decomposition as a function of temperature. The specific temperature is dependent on heating rate, which if raised too high, can produce significantly different results than a low heating rate. This experiment utilized TGA Q500 machine by TA Instruments, Inc.

Pultruded Composite Fabrication

The constituent materials for the resin formation were provided by a private company. These were mixed and pultruded at the University of Mississippi using 113 yield E-glass rovings that were pulled through a 36-inch-long rectangular die with dimensions of 2.5” x 1/8”. The fiber volume fraction for the composite was 67%. Sections with a length of four feet were automatically cut by a saw at the end of the pultrusion machine. The last five pieces of the material were taken as the best representation of the composite properties and were thus used for the physical and mechanical characterization. Pull force data was measured during the pultrusion manufacturing experiments using a load cell that measured force applied by the die to the load cell as the pultruded composite was pultruded.

With the samples divided into sections of four feet, these were cut into appropriately sized mechanical test samples using a water jet at the University of Mississippi. The dimensions of the flexural samples were approximately 3” x 1” x 0.125” while the short beam samples were created by a combination of the water jet and diamond saw with dimensions of 0.75” x 0.5” x 0.125”. Once dried, they were labelled and measured for the length, width, height, and pre-exposure mass. These were then separated to their respective exposure environments where they remained for periods of 500 and 1000 hours. However, due to technical issues, there were no flexural samples exposed for 500 hours and no short beam samples exposed to 1000 hours of HCl. When the samples met their required environmental exposure length, the masses were measured again and were tested only for their maximum strength values as the modulus is not majorly dependent on exposure. The aged flexural and short beam strengths were compiled and contrasted with the unaged strengths to determine the consequences of chemical and thermal attack.

FABRICATION RESULTS

This section explains the results of the first part of the experiment obtained during the fabrication of pultruded composites. Differentiations between sets of data are shown more easily in graph form and can be compared directly. Any deviations from expected values are highlighted and explained. The selection process for additional mechanical testing is also explained.

Manufacturing Results

The experimental phr values for each run is seen in Tables 3-5. Pull forces from these experiments are separated by both color and release agent and can be seen in Figures 12-18. The mold releases designated at MR B, MR C, and MR F are experimental while the commercially available MR A is used as a comparison.

Table 3 - phr Values for Pultruded Composites with Beige Pigment

		Actual Beige phr Values			
Material	Theoretical	MR A	MR F	MR B	MR C
Aropol 50440	100	100	100	100	100
Monomer, Styrene	9.50	9.51	9.52	9.50	9.50
Catalyst, Perkadox 16	0.2	0.20	0.20	0.20	0.20
Catalyst, Trigonox 121C75	0.6	0.60	0.60	0.60	0.60
Catalyst, TBPB Trigonox C	0.1	0.10	0.10	0.10	0.10
Low Profile Additive, Microthene FN510	2	2.00	2.00	1.99	2.00
Pigment	0.6	0.60	0.60	0.60	0.60
Filler, Calcium Carbonate Fine	16.7	16.54	16.72	16.69	16.69
Filler, Aluminum Trihydrate	10	9.93	10.02	10.08	9.92
Mold Release	3	3.01	3.00	3.00	3.00

Table 4 - phr Values for Pultruded Composites with Yellow Pigment

		Actual Yellow phr Values			
Material	Theoretical	MR A	MR F	MR B	MR C
Aropol 50440	100	100	100	100	100
Monomer, Styrene	9.50	9.51	9.51	9.50	9.50
Catalyst, Perkadox 16	0.2	0.20	0.20	0.20	0.20
Catalyst, Trigonox 121C75	0.6	0.60	0.60	0.60	0.60
Catalyst, TBPB Trigonox C	0.1	0.10	0.10	0.10	0.10
Low Profile Additive, Microthene FN510	2	1.99	2.00	2.00	2.00
Pigment	0.6	3.00	3.00	3.00	3.01
Filler, Calcium Carbonate Fine	16.7	16.73	16.73	16.69	16.73
Filler, Aluminum Trihydrate	10	9.93	9.93	9.92	9.93
Mold Release	3	3.00	3.00	3.00	3.01

Table 5 - phr Values for Pultruded Composites with Blue Pigment

		Actual Blue phr Values		
Material	Theoretical	MR A	MR B	MR C
Aropol 50440	100	100	100	100
Monomer, Styrene	9.50	9.51	9.50	9.51
Catalyst, Perkadox 16	0.2	0.20	0.20	0.20
Catalyst, Trigonox 121C75	0.6	0.60	0.60	0.60
Catalyst, TBPB Trigonox C	0.1	0.10	0.10	0.10
Low Profile Additive, Microthene FN510	2	2.00	2.00	2.00
Pigment	0.6	3.00	3.00	3.00
Filler, Calcium Carbonate Fine	16.7	16.73	16.69	16.54
Filler, Aluminum Trihydrate	10	9.93	9.92	9.93
Mold Release	3	3.00	3.00	3.00

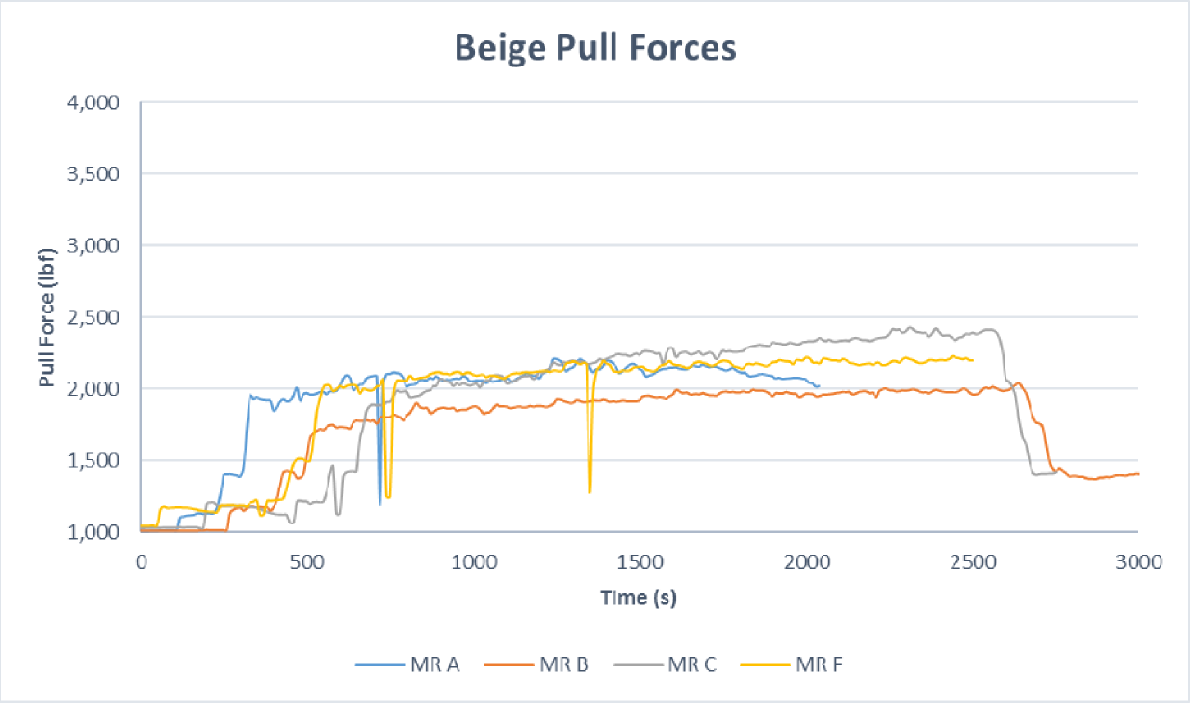


Figure 12 - Beige Pull Forces

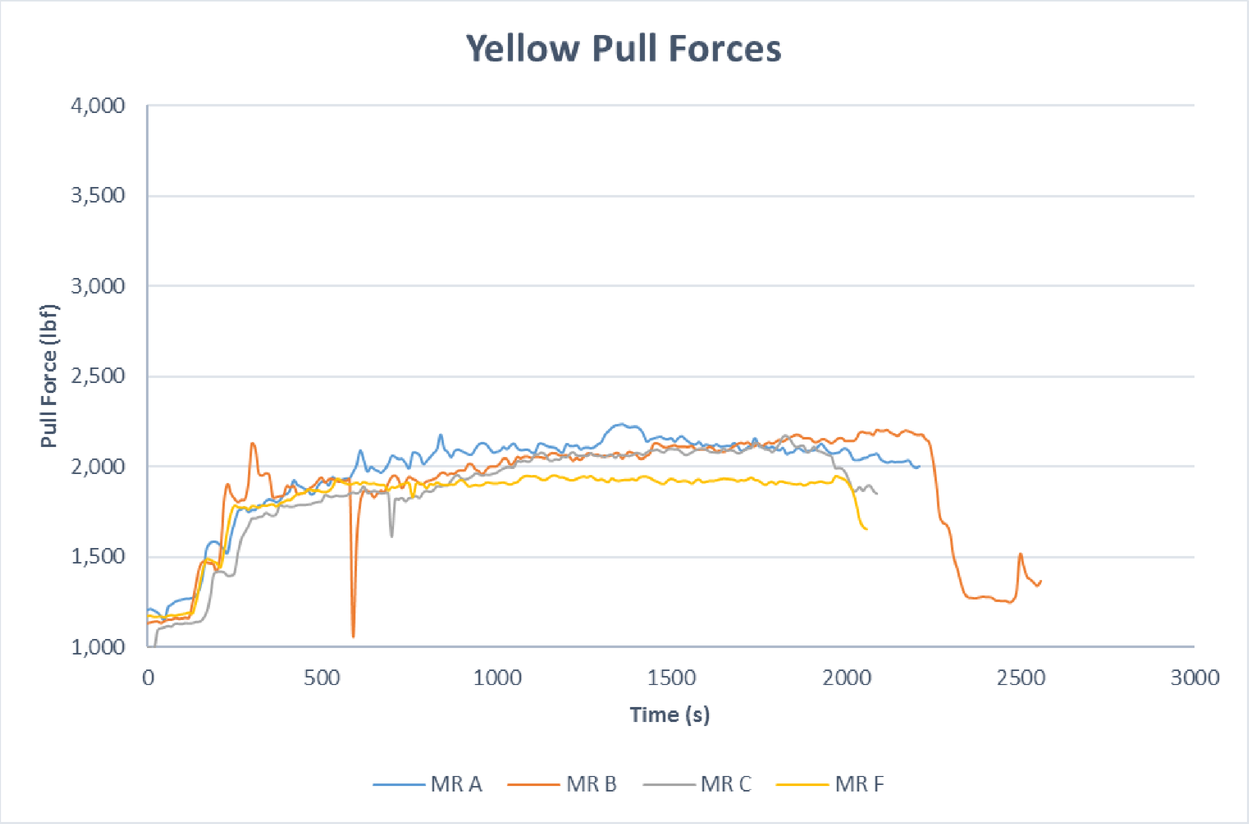


Figure 13 - Yellow Pull Forces

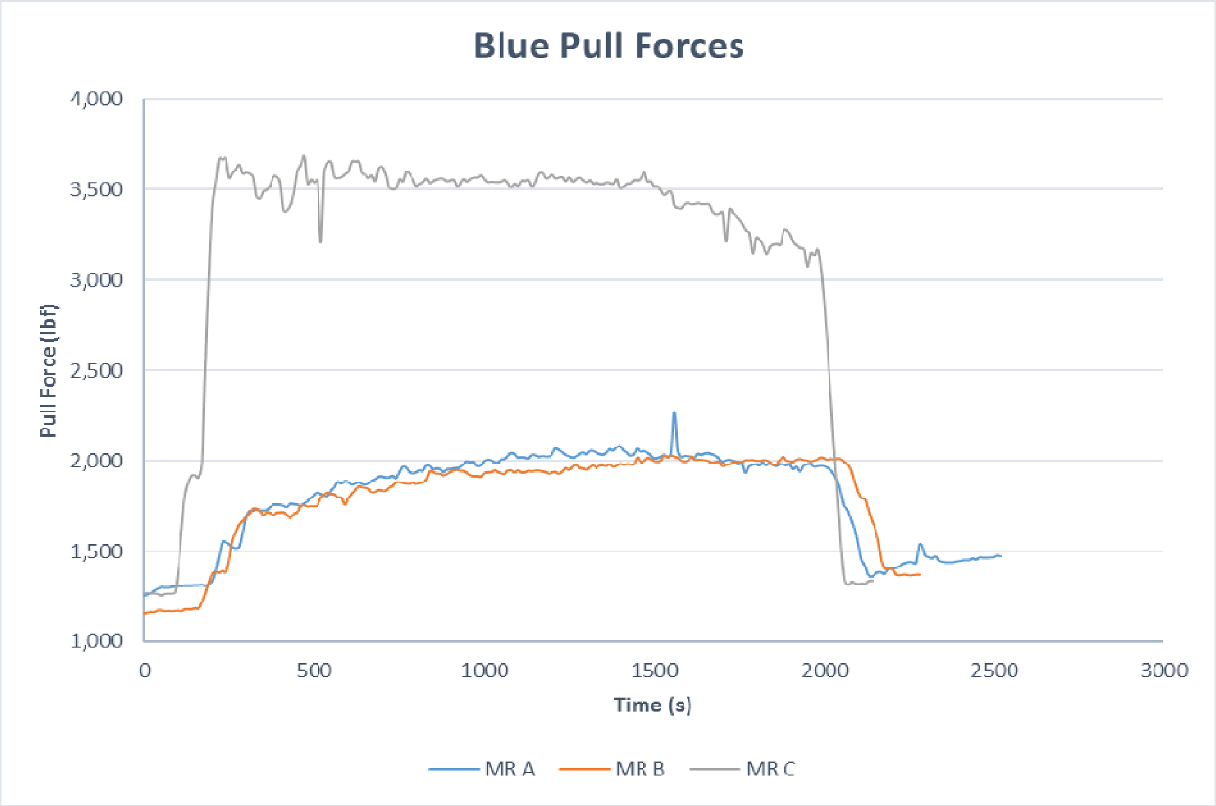


Figure 14 - Blue Pull Forces

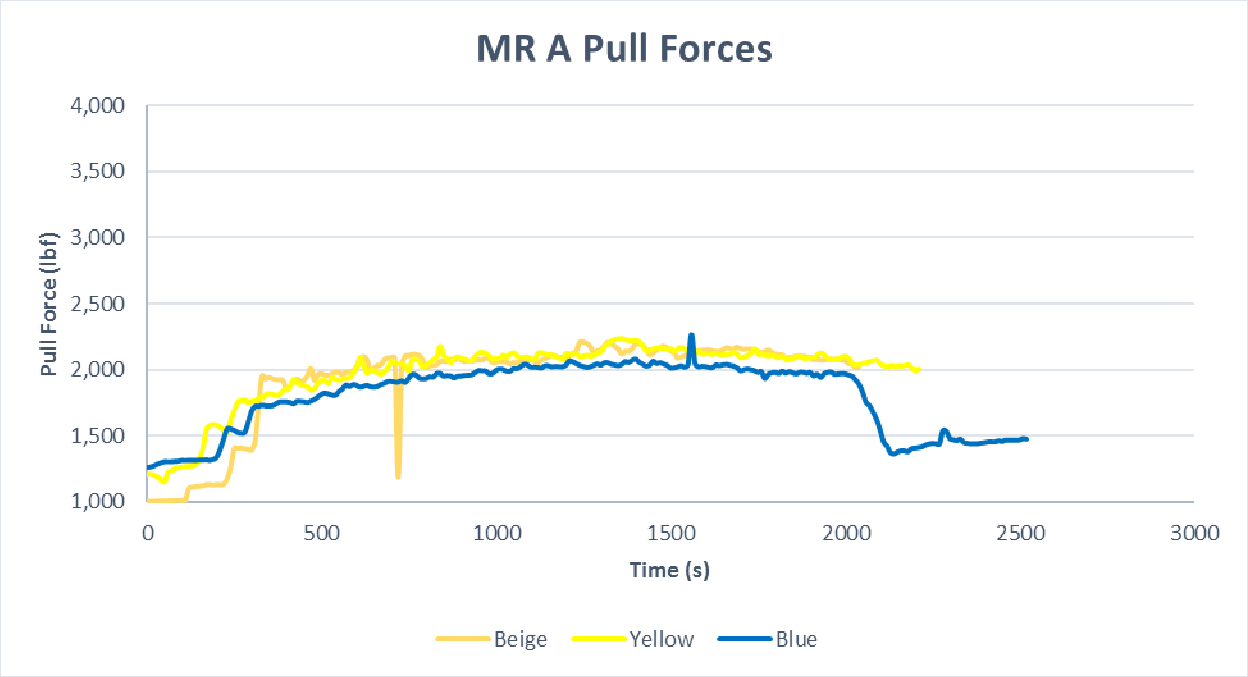


Figure 15 - MR A Forces

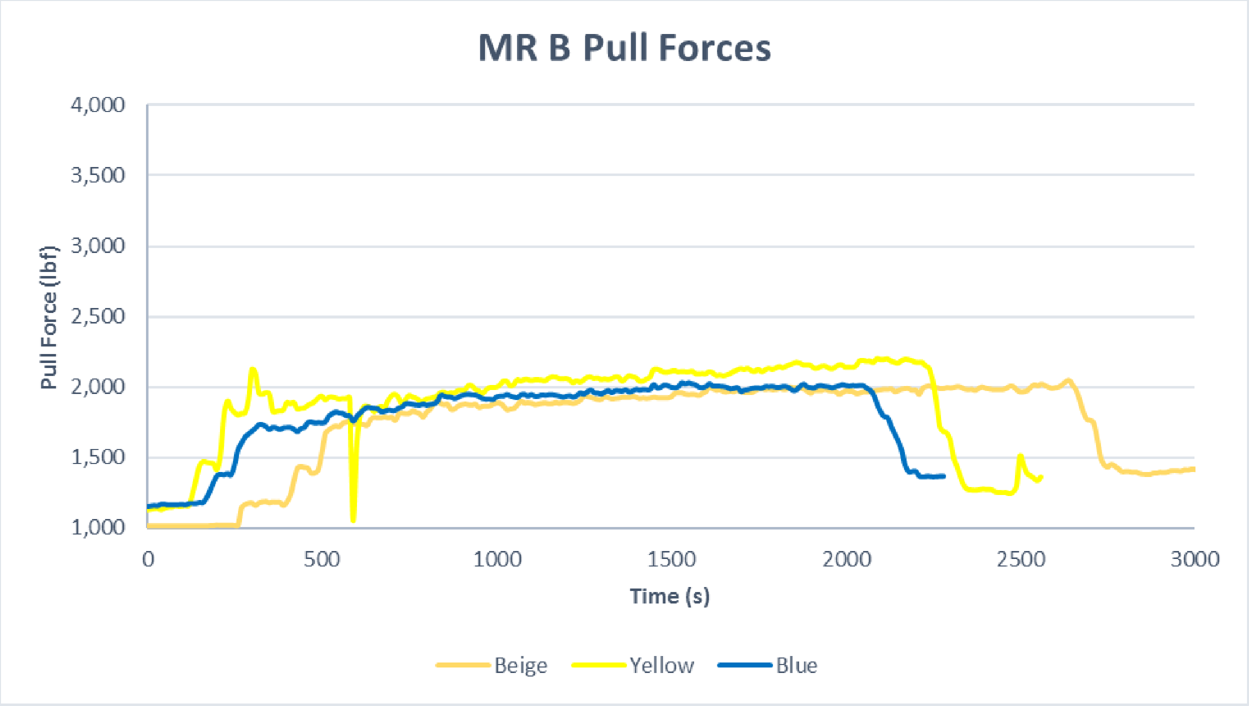


Figure 16 - MR B Pull Forces

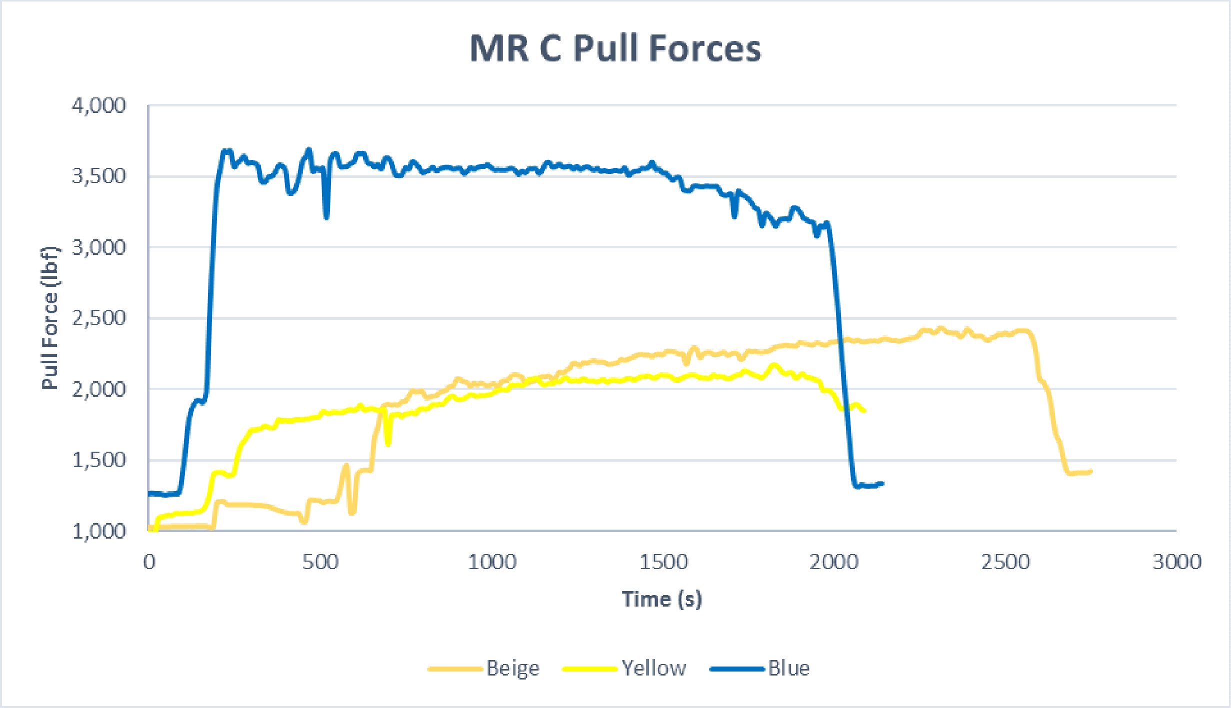
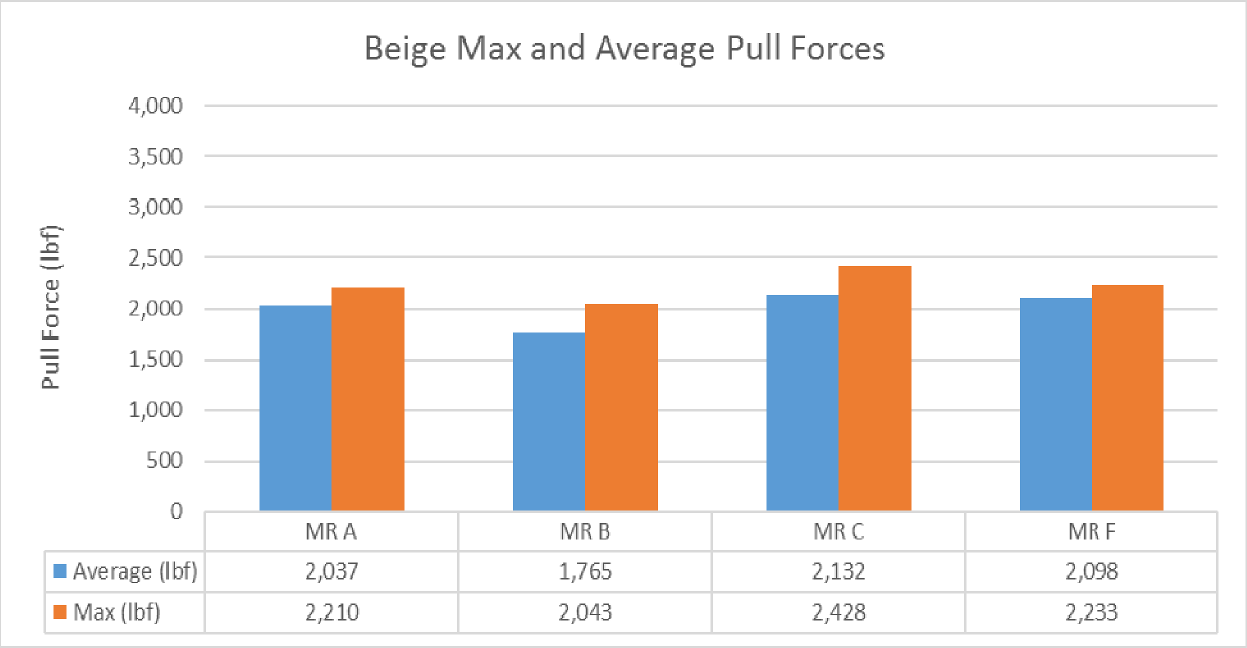


Figure 17 - MR C Pull Forces

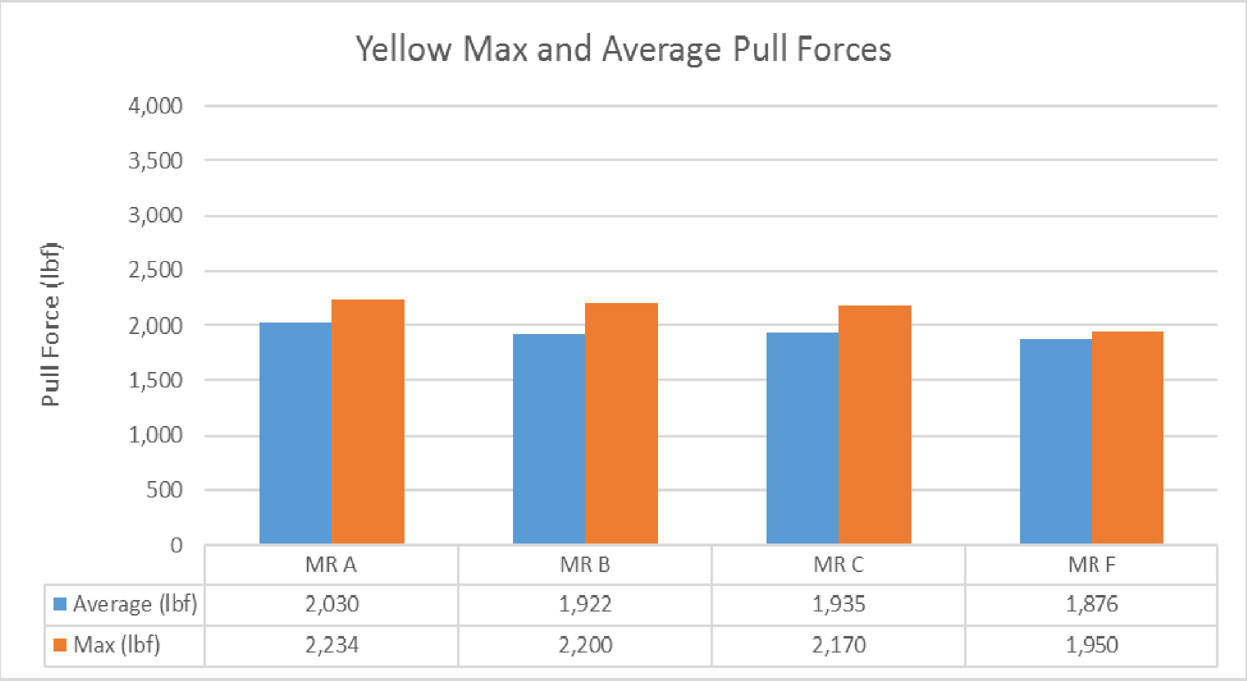
Figure 18 - MR F Pull Forces

Among the most important factors for commercial pultrusion are the average and maximum pull forces present while the composite cures in the die. These forces are a function of the fiber volume, fiber type, resin, catalysts, die temperatures, cure rate, etc. In general, a lower pull force is desired for most resin systems. This ensures the resin will not hang up in the die causing a stoppage of work. For this reason, an understanding of the maximum and average pull forces is desired. The values for maximum and average pull forces for the beige, yellow, and blue runs are shown below in Figures 19-21. A change to a more complex die with larger surface area would exhibit greater pull forces, but the data shown in Figures 19-21 for this 2 ½” x 1/8” rectangular die section allow for direct comparison of these mold release products.



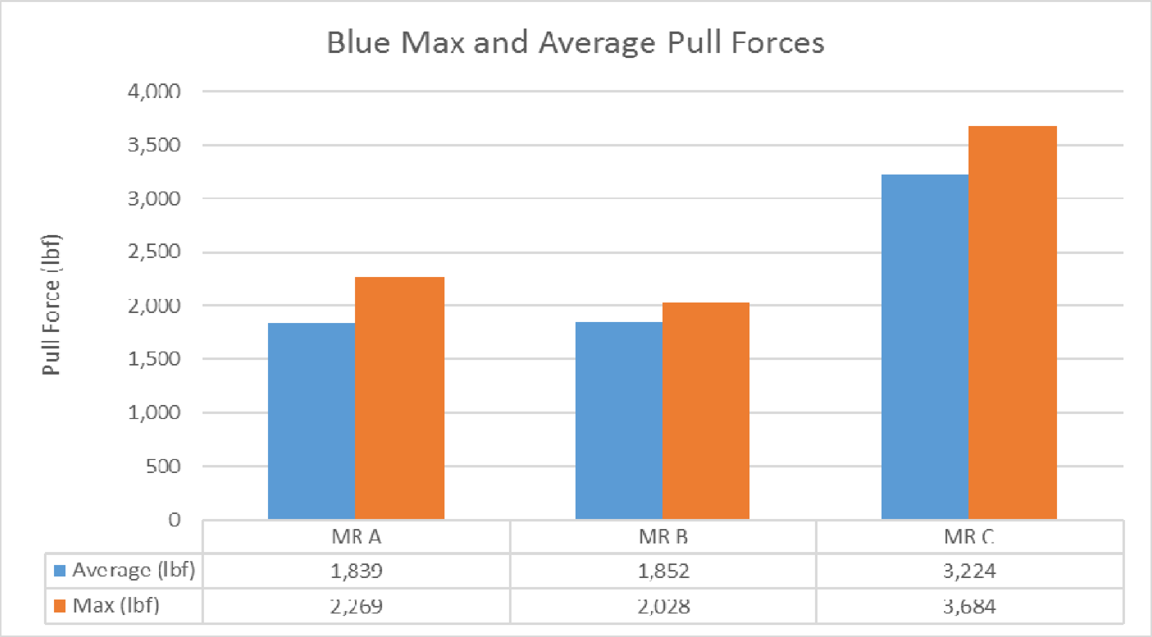
	MR A	MR B	MR C	MR F
🟦 Average (lbf)	2,037	1,765	2,132	2,098
🟧 Max (lbf)	2,210	2,043	2,428	2,233

Figure 19 - Beige Max and Average Pull Forces



	MR A	MR B	MR C	MR F
🟦 Average (lbf)	2,030	1,922	1,935	1,876
🟧 Max (lbf)	2,234	2,200	2,170	1,950

Figure 20 - Yellow Max and Average Pull Forces



	MR A	MR B	MR C
🟦 Average (lbf)	1,839	1,852	3,224
🟧 Max (lbf)	2,269	2,028	3,684

Figure 21 - Blue Max and Average Pull Forces

The first area of concern is determining whether the pigment interaction played a significant factor in the pull force values. There is seemingly little significant deviation due to added pigment. It should be noted, however, that the MR C blue pull force is not in agreement with the values for the yellow and beige. The blue pigment does not significantly affect the values for average pull forces for both the MR A and MR B mold releases. However, a new batch of MR C was used for the blue run which leads to the conclusion that the new MR C mold release was chemically different from the previous one. The surfaces of the composites were analyzed by sight and touch. The composites that had exposed fibers or rough edges were discarded from further mechanical analysis. Using this procedure, the only mold releases that produced an acceptable surface quality were those fabricated with MR A, the commercial product, and MR F. Thus, only composites with MR A and MR F were viable candidates for mechanical testing following these pultrusion manufacturing experiments, and, from these, the beige pieces were selected for mechanical testing as they did not exhibit the color inconsistency that others displayed.

MECHANICAL TEST RESULTS

The number of samples for each test are shown below. With the exception of the "As Pulled" samples, the number of samples for the flex testing are the same as the short beam testing. Table 6 indicates the number of samples tested for each exposure.

Table 6 - Number of Samples

Exposure (Hours)	0	500	1000
As Pulled	4-8	-	-
QUV	-	5	5
Bleach	-	3	5
HCl	-	0-3	0-5
Distilled Water at 125°F	-	3	5
Salt Water	-	3	5

As Pulled

The purpose of this section is to define the strengths of the material as they would present themselves with no environmental exposure. The "As Pulled" samples are defined as the unaged strength values of the composites, and the values here will be used as the t = 0 for the following exposure strengths. It is important to note that this is not an optimized through secondary curing and that further strength increases may be possible. The values shown are the ranges of strengths obtained from testing in Figures 22 and 23. Measured and calculated values for each specific run can be seen in the Appendix in Tables 8-11. The failure mode of these samples when tested for flexural properties was due to interlaminar shear. Cracks propagated from the middle to the outer layer as in Figure 7 (f) indicating a poor bonding between the fibers and resin system. Because this failure mode occurred in the unaged samples, it is unlikely a different failure mechanism will be prevalent in the aged samples. The short beam samples failed in interlaminar shear as well. In both cases, the experimental mold release performed better than its commercially available competitor.

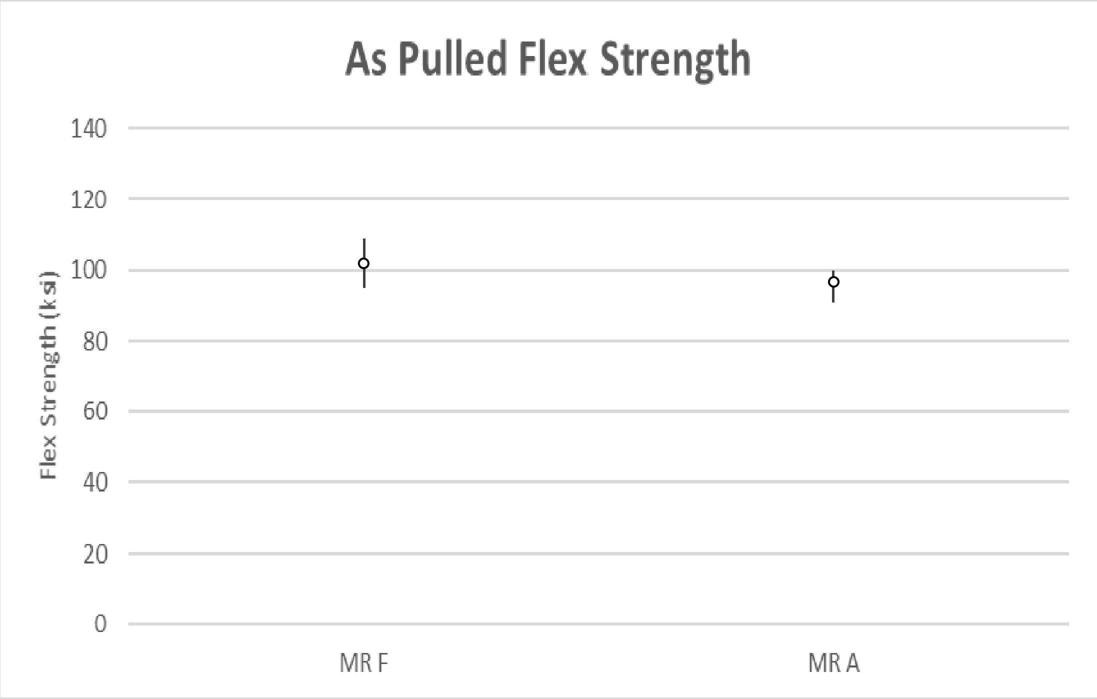


Figure 22 - Unaged Flexural Strength

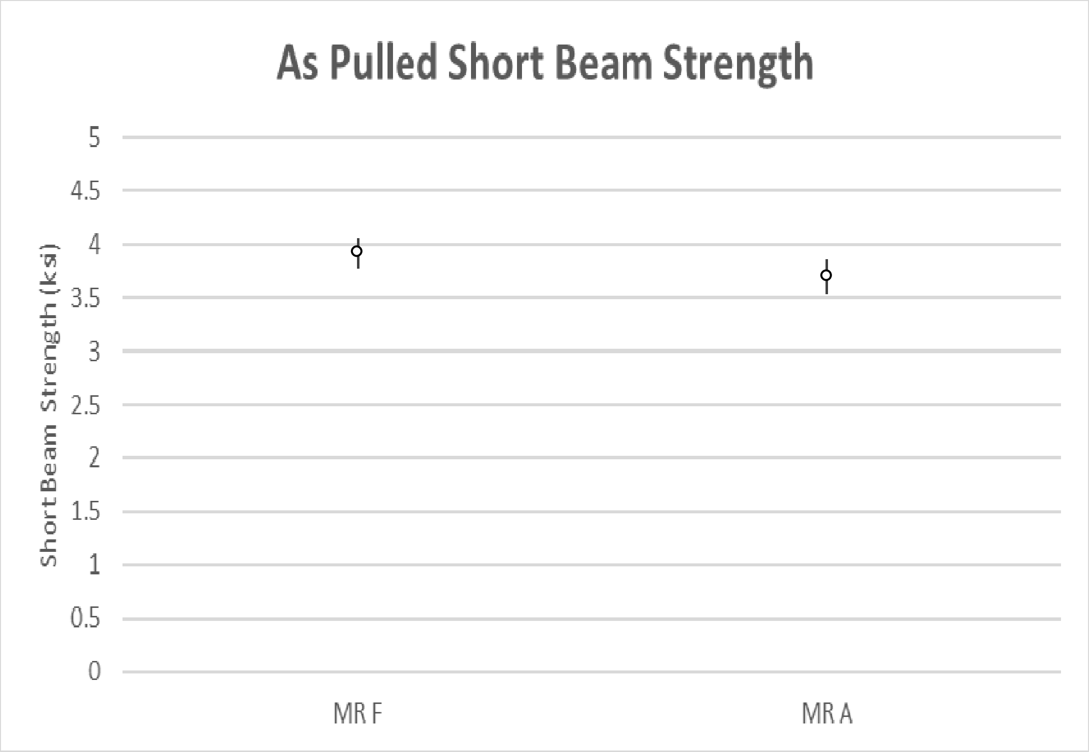


Figure 23 - Unaged Short Beam Strength

QUV

The flexural and short beam strengths for both the MR A and MR F mold releases are shown in Figures 24-27. The flexural and short beam strengths of each mold release increased upon exposure to the UV environment. The flexural properties increased by approximately 20% upon the initial 500 hours of exposure with an evident decline in the further increase of flexural strength. The short beam properties increased as well in the 500 hour mark but decreased during the 1000 hour mark. Often when material strengths increase in hot environments, secondary curing is the most likely reason behind the increase. This secondary curing from both the temperature increase and the UV interaction is particularly known to affect polyester resins. Once the secondary curing completed, a decrease in strengths is seen for each material indicating the maximum increase in initial strength gains has completed. As stated previously, QUV tests tend to affect the outer layers of the composite. Because this was a pultruded sample that contained only unidirectional rovings, it was less likely for significant delamination to occur as is sometimes the case when chopped strand mats or woven fibers are used [13]. Discoloration was also observed on the face of the composite from a light beige to a darker tan color. The differences in mold releases do not seem to significantly affect the trend of the flexural strengths post exposure. Measured and calculated values for each specific run can be seen in the Appendix in Tables 12-19. The flexural and short beam samples also failed in interlaminar shear.

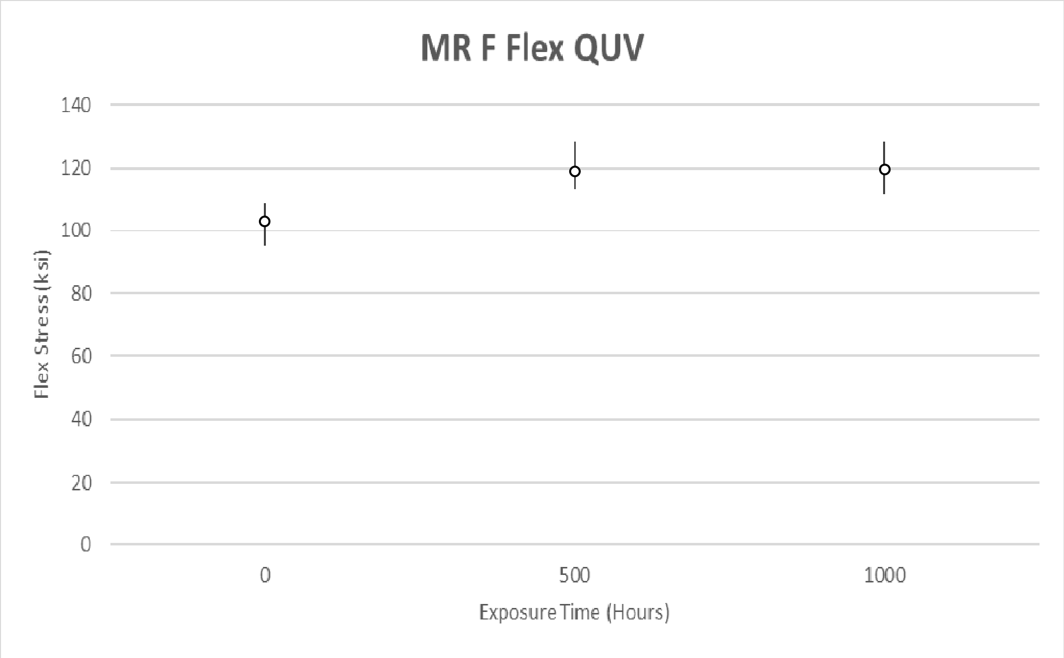


Figure 24 - MR F QUV Exposure Flexural Strength

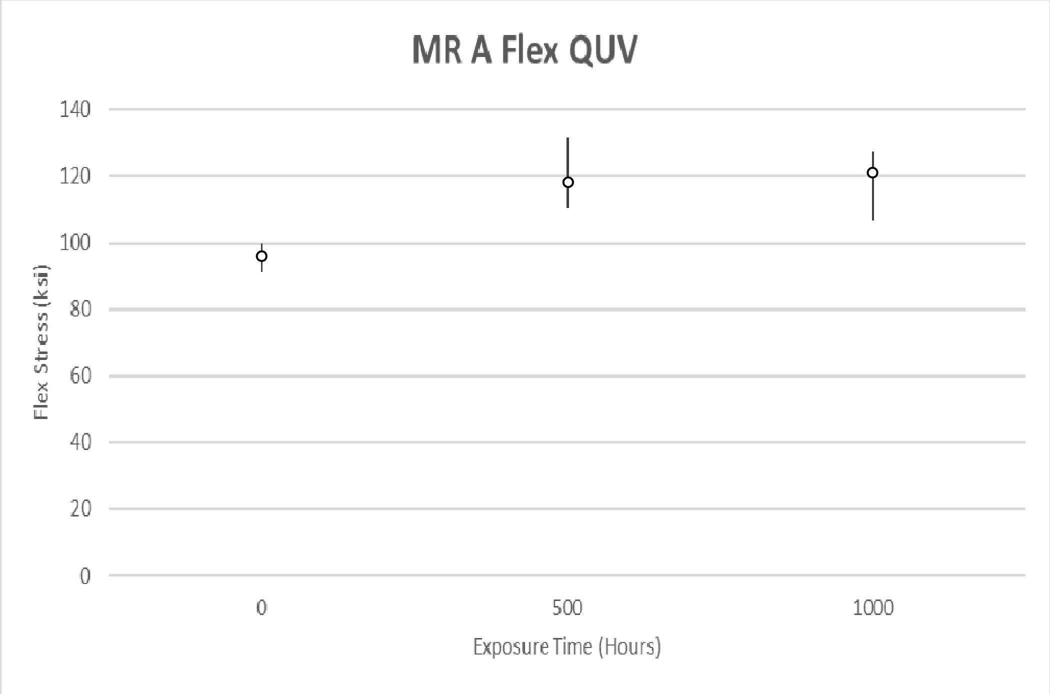


Figure 25 - MR A QUV Exposure Flexural Strength

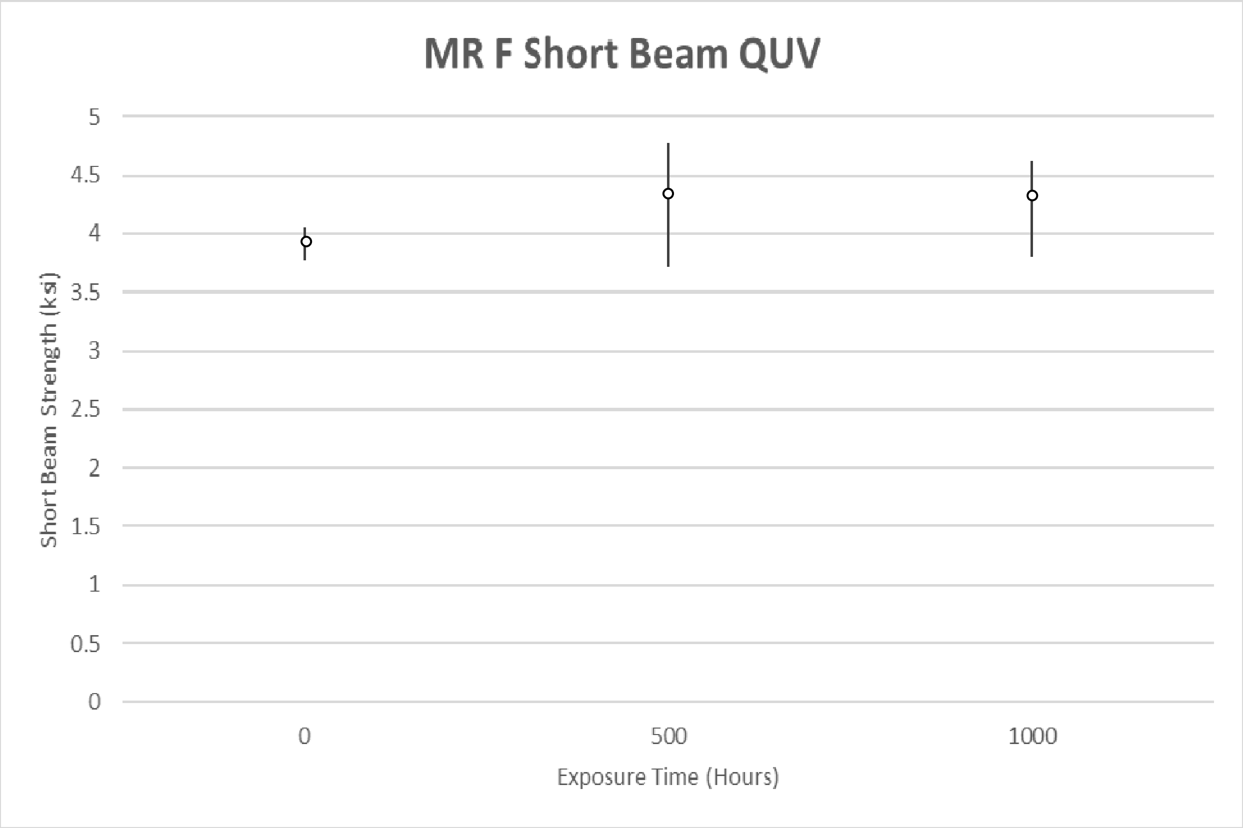


Figure 26 - MR F QUV Exposure Short Beam Strength

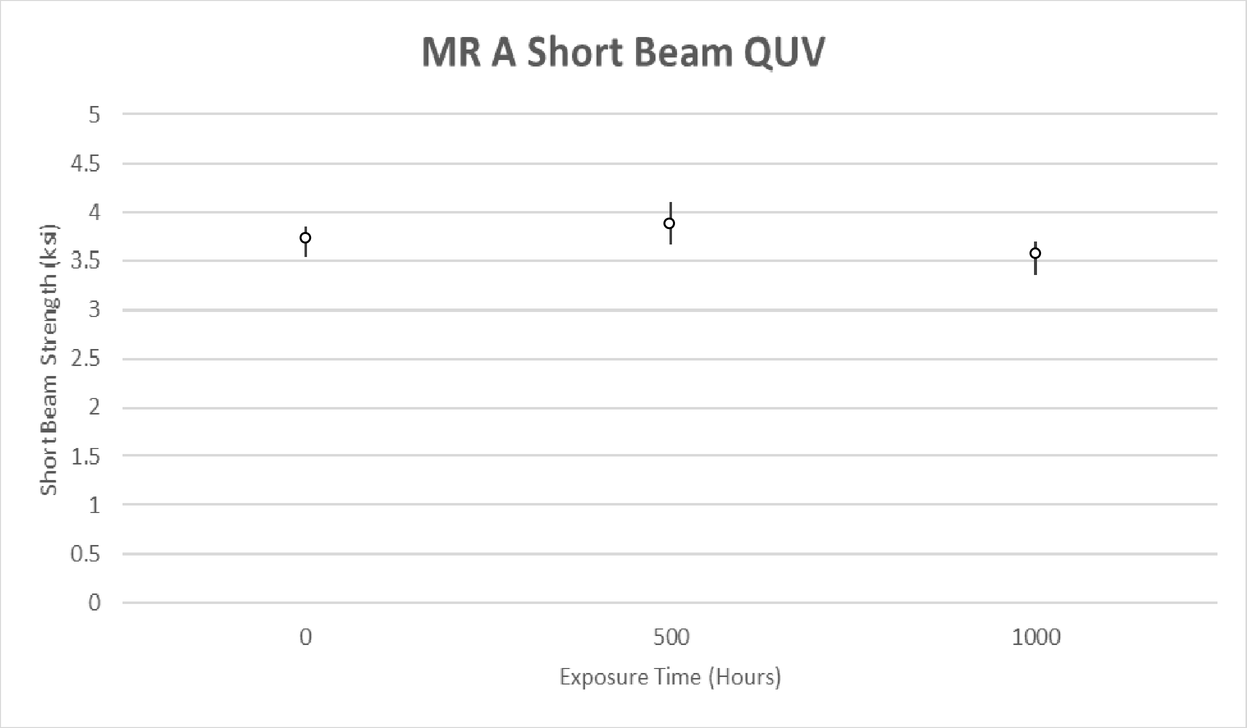


Figure 27 - MR A QUV Exposure Short Beam Strength

Bleach

Exposure to the bleach solution exhibited the most significant and consistent changes in strengths for both the flexural and short beam tests. As shown in the QUV tests, an increase in properties was most likely due to incomplete an initial incomplete cure. This indicates an abundance of sites for the bleach to attack. However, given that the short beam strength lost more than 30% and 45% for MR F and MR A respectively, this indicates a major failure in matrix/ fiber interaction. Because bleach is particularly aggressive in this case, further reduction may still be possible to the point that the matrix is unable to distribute the load to the fibers effectively. The bleach also changed the appearance and surface quality of the composite. While initially beige, the exposed samples became whiter in color while revealing frayed fibers. Figures 28-31 show the ranges for strengths after degradation for both mold releases in flexural and short beam tests. Measured and calculated values for each specific run can be seen in the Appendix in Tables 20-27. The samples all failed in a manner consistent with the previous flexural and short beam failures.

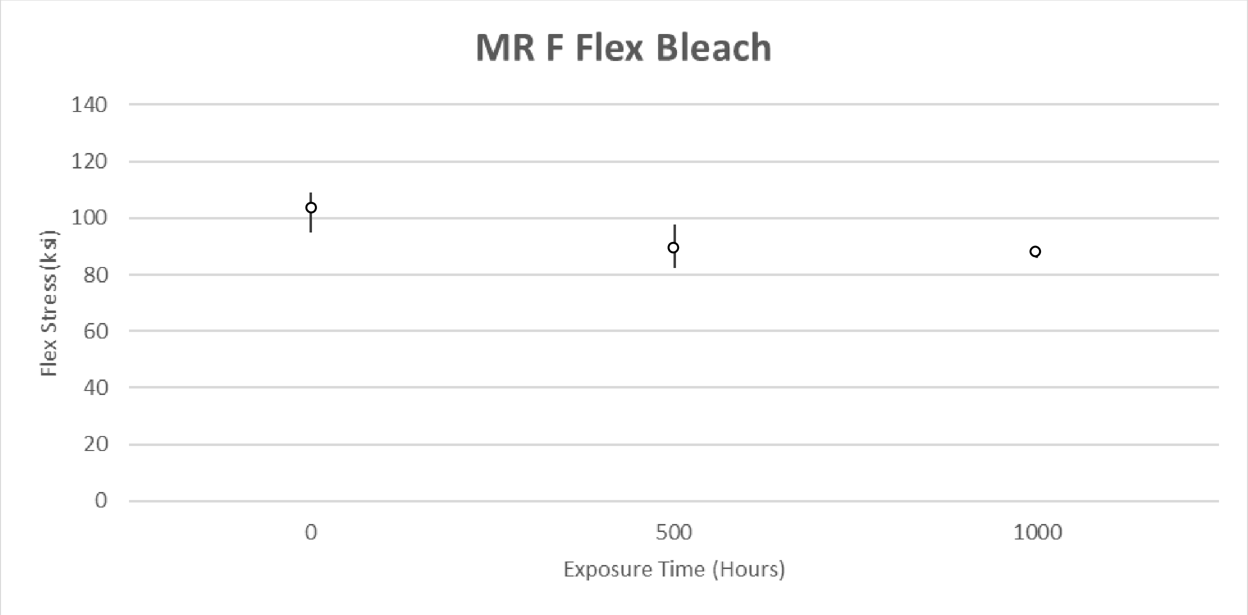


Figure 28 - MR F Bleach Exposure Flexural Strength

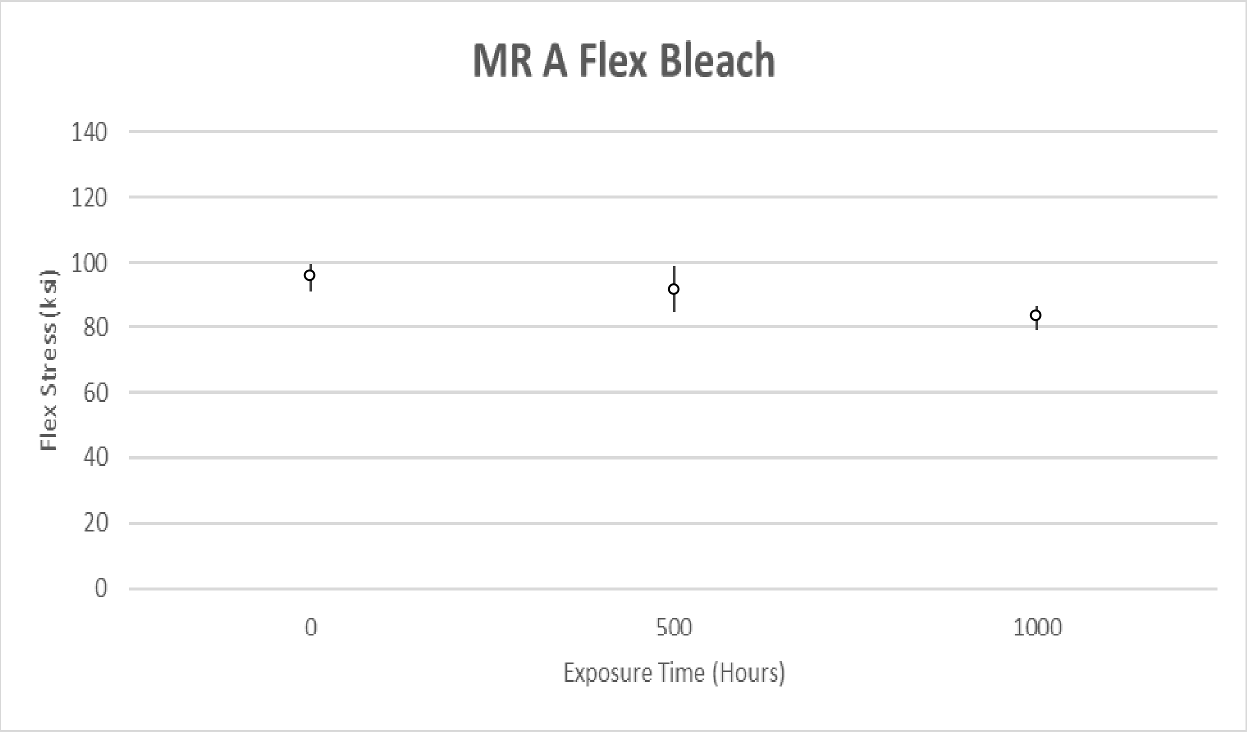


Figure 29 - MR A Bleach Exposure Flexural Strength

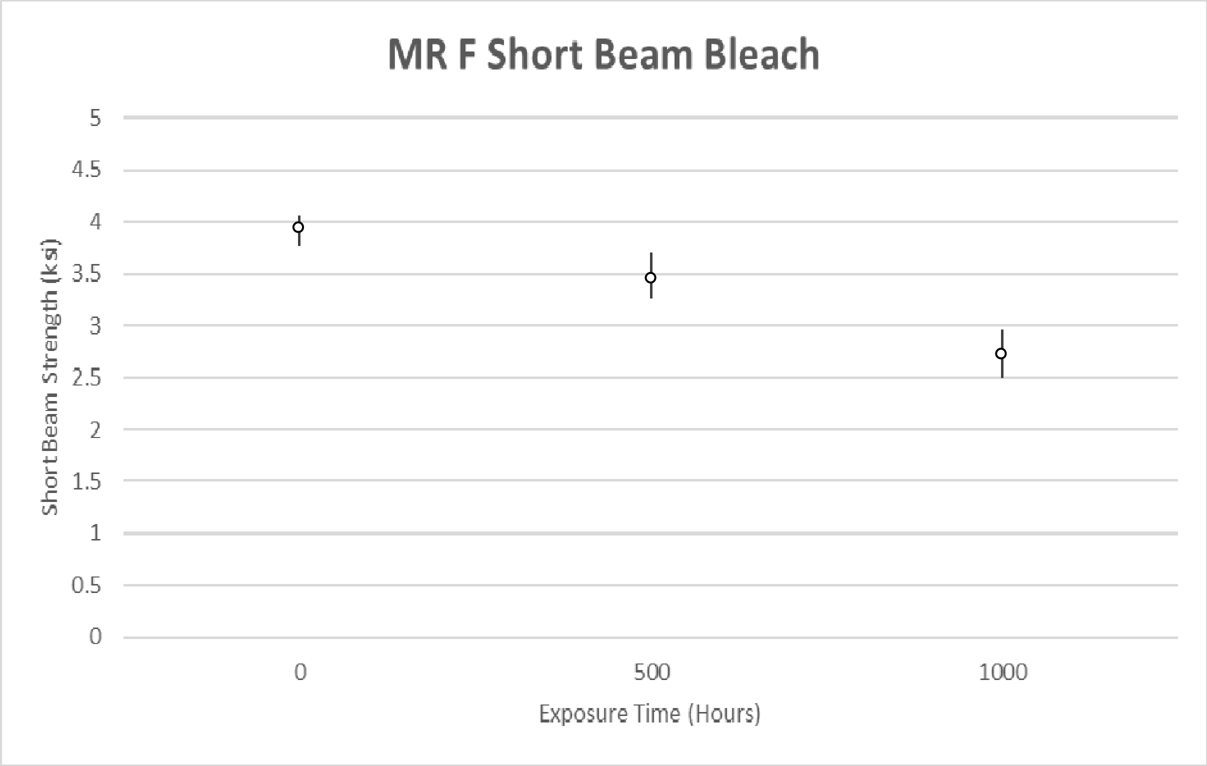


Figure 30 - MR F Bleach Exposure Short Beam Strength

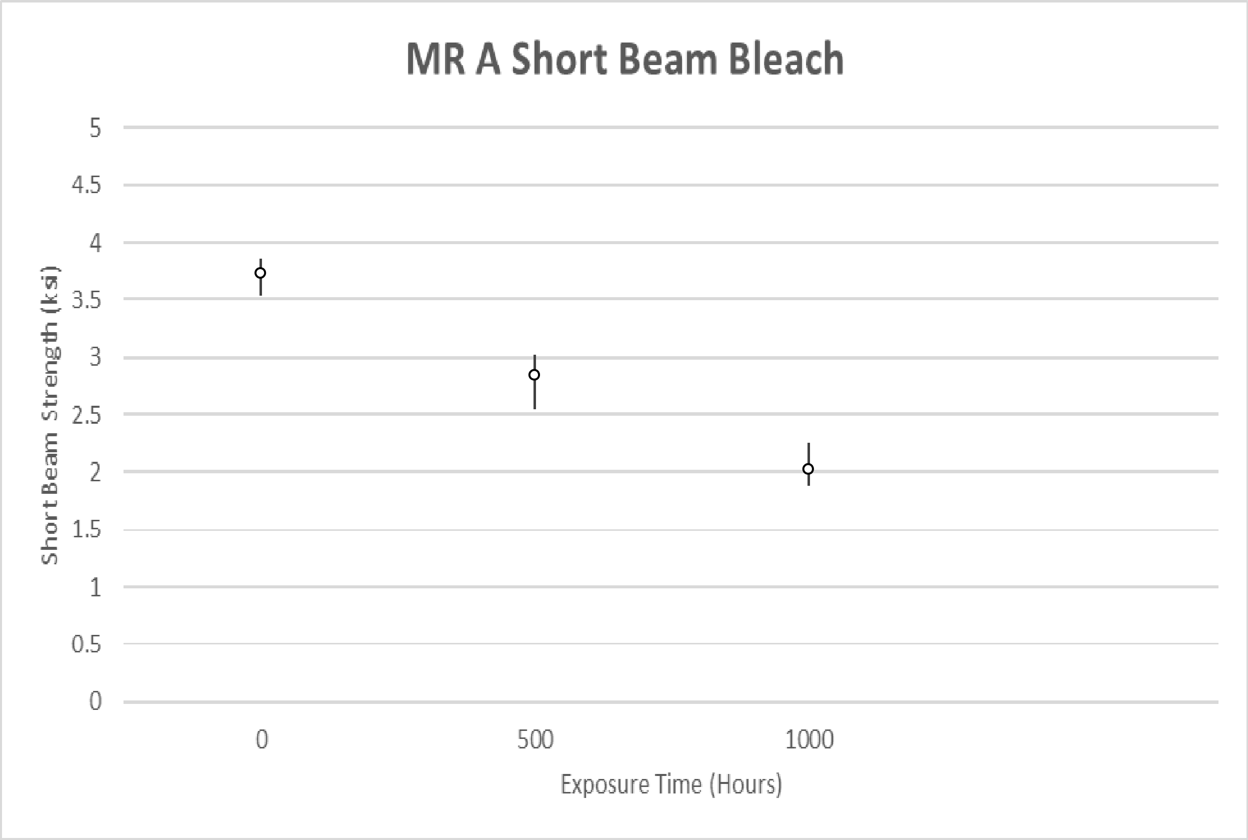


Figure 31 - MR A Bleach Exposure Short Beam Strength

Hydrochloric acid

Flexural samples exposed to hydrochloric acid were only taken at an exposure time of 1000 hours while the short beam samples were only taken at exposure times of 500 hours. These results shown in Figures 32-35. They were chosen to represent the 500 and 1000 hour exposure times for both test types. The flexural tests at 1000 hours show a small increase in average flexural strength for both mold releases while the 500-hour short beam samples were much more vulnerable to decreases in strength. Most notably, the MR A samples were more affected by the hydrochloric acid exposure. This may indicate that the MR A mold release is chemically different enough to react with the HCl in a more negative manner for short beam properties. Measured and calculated values for each specific run can be seen in the Appendix in Tables 28-31. Because the samples increased their average flexural strength over the course of 1000 hours and decreased the short beam strength in only 500 hours of exposure, it is likely that the flexural tests do not accurately indicate material decomposition. This is likely due to the failure mechanism (interlaminar shear) that was prevalent in all aged and unaged flexural samples.

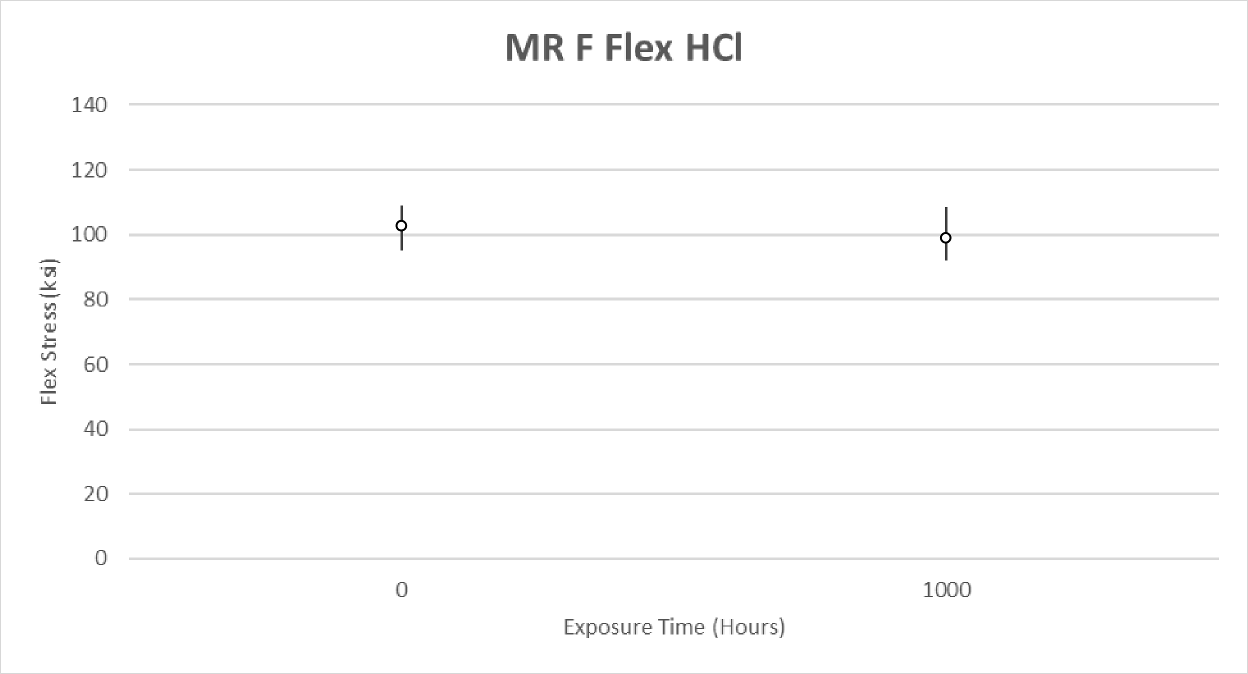


Figure 32 - MR F HCl Exposure Flexural Strength

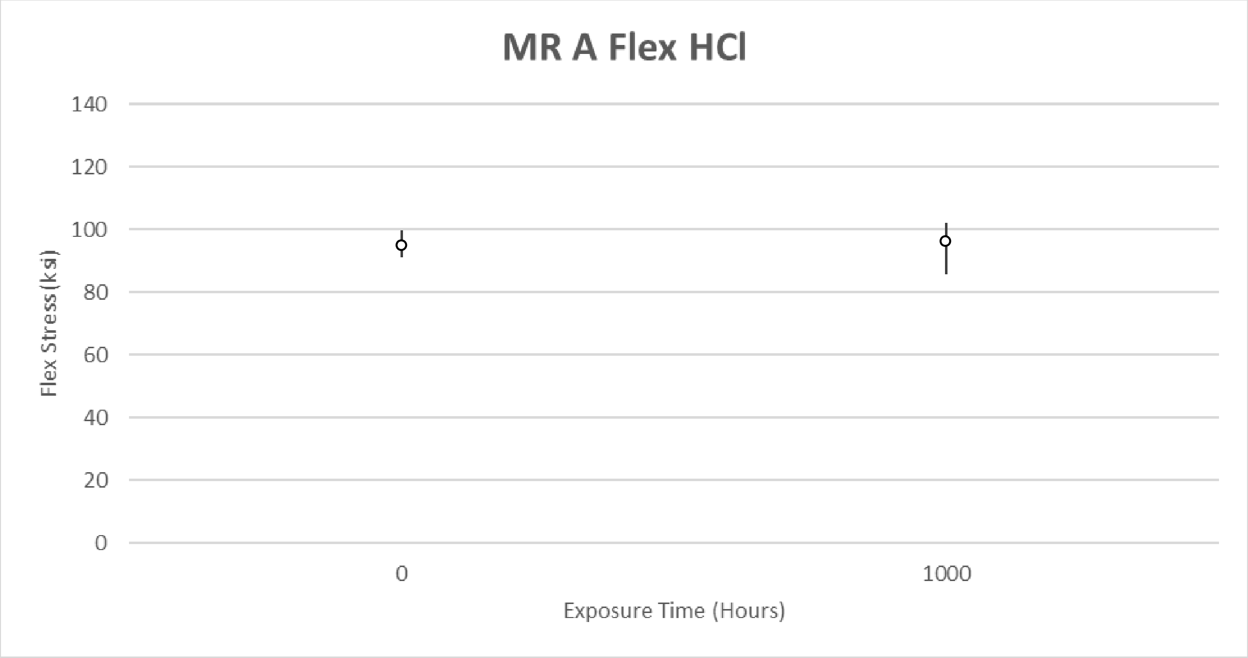


Figure 33 - MR A HCl Exposure Flexural

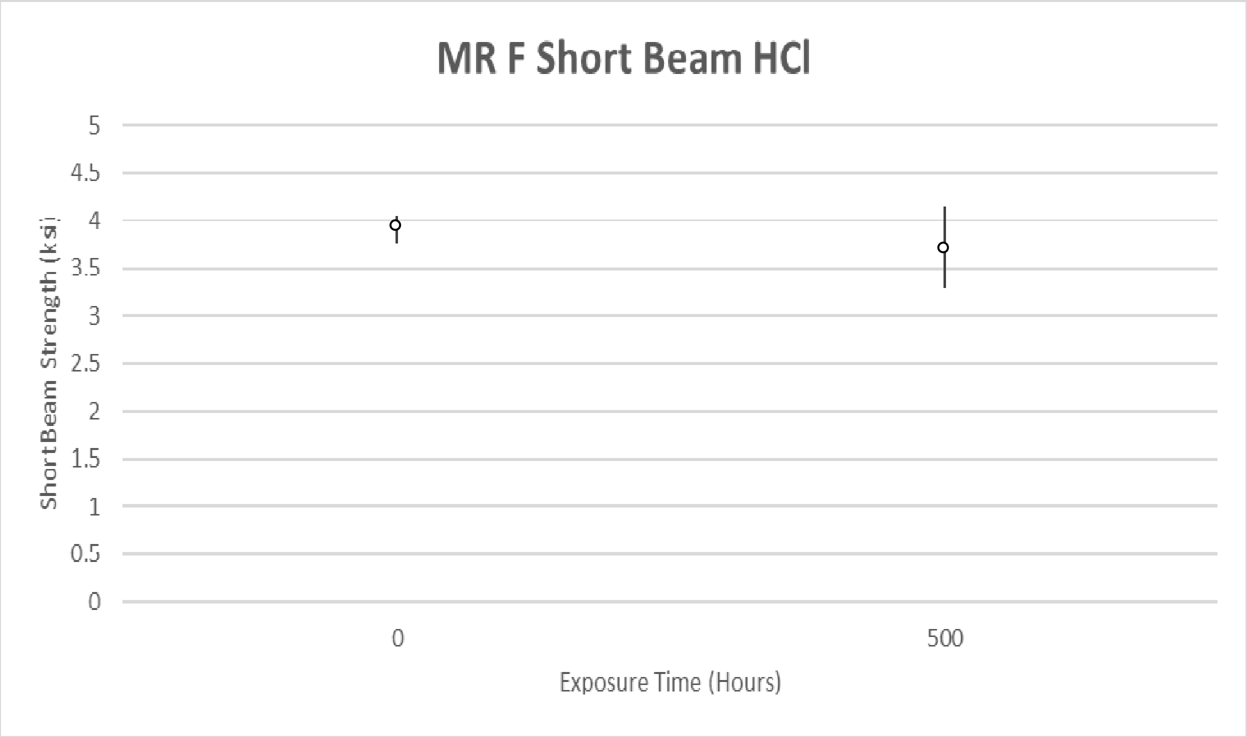


Figure 34 - MR F HCl Exposure Short Beam Strength

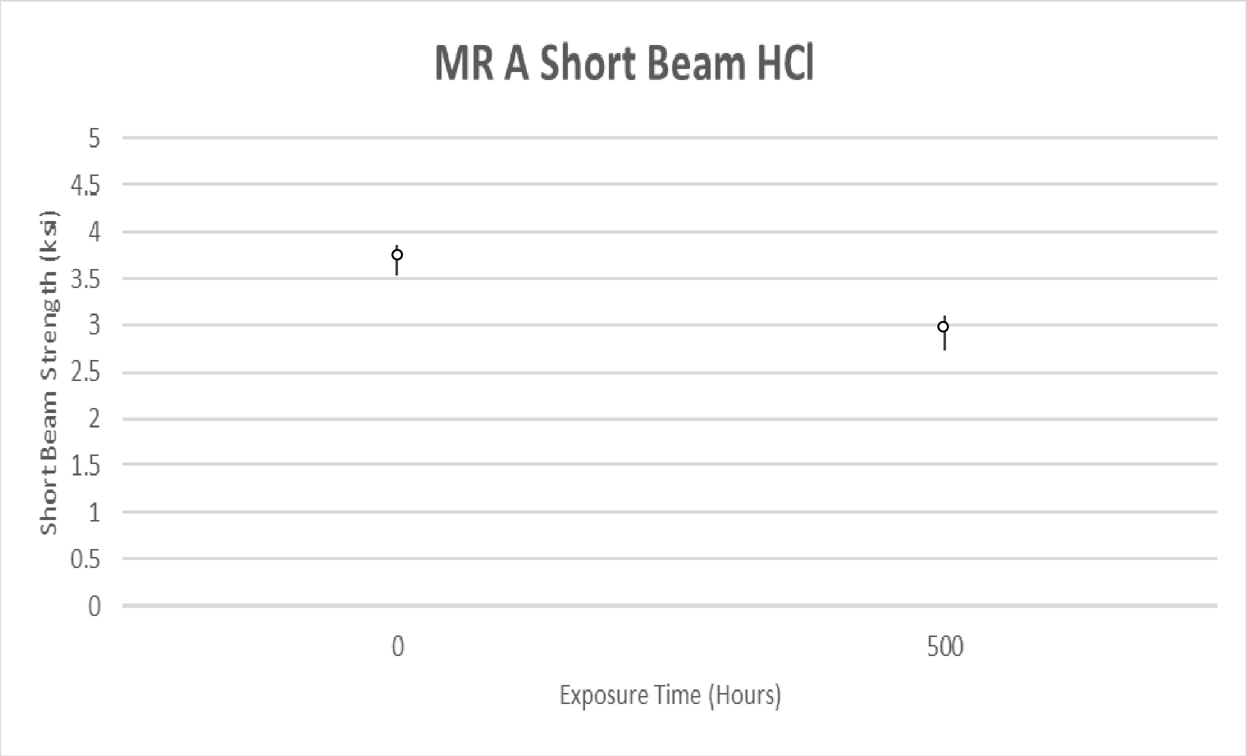


Figure 35 - MR A HCl Exposure Short Beam Strength

Elevated Temperature Distilled Water

The findings for distilled water were similar to those from the QUV exposure. Initially, the short beam and flexural strengths increased likely due to post curing from the 125°F. After the initial exposure, the increases in flexural strengths either stopped or significantly slowed whereas the short beam strengths began to noticeably decrease. The effects of water concentration and temperature are understood with respect to composites. Classical Lamination Theory provides a model that can be used to estimate mechanical properties when the unsaturated, room temperature properties are known [4]. However, this model assumes perfect bonding between laminates and does not accurately account for voids and thus is often used as an upper limit. In this exposure, the differences in mold release are not noticeably apparent. The strengths for this exposure can be seen in Figures 36-39. Measured and calculated values for each specific run can be seen in the Appendix in Tables 32-39. Again the composites failed due to interlaminar shear stresses for both test types.

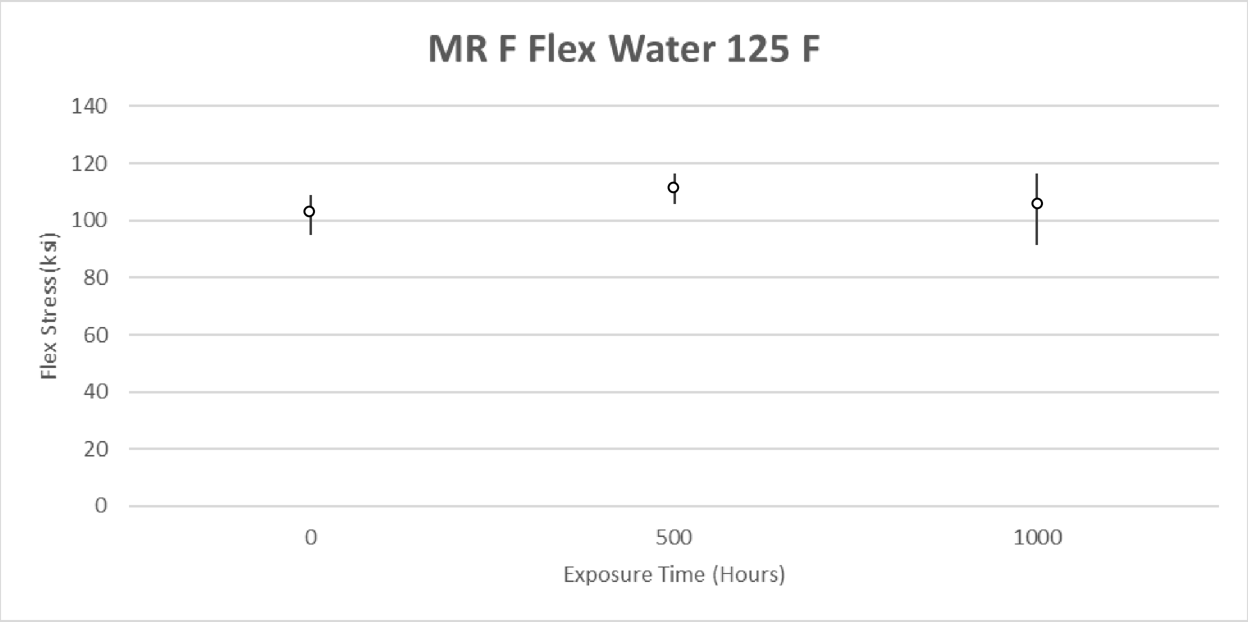


Figure 36 - MR F 125°F Water Exposure Flexural Strength

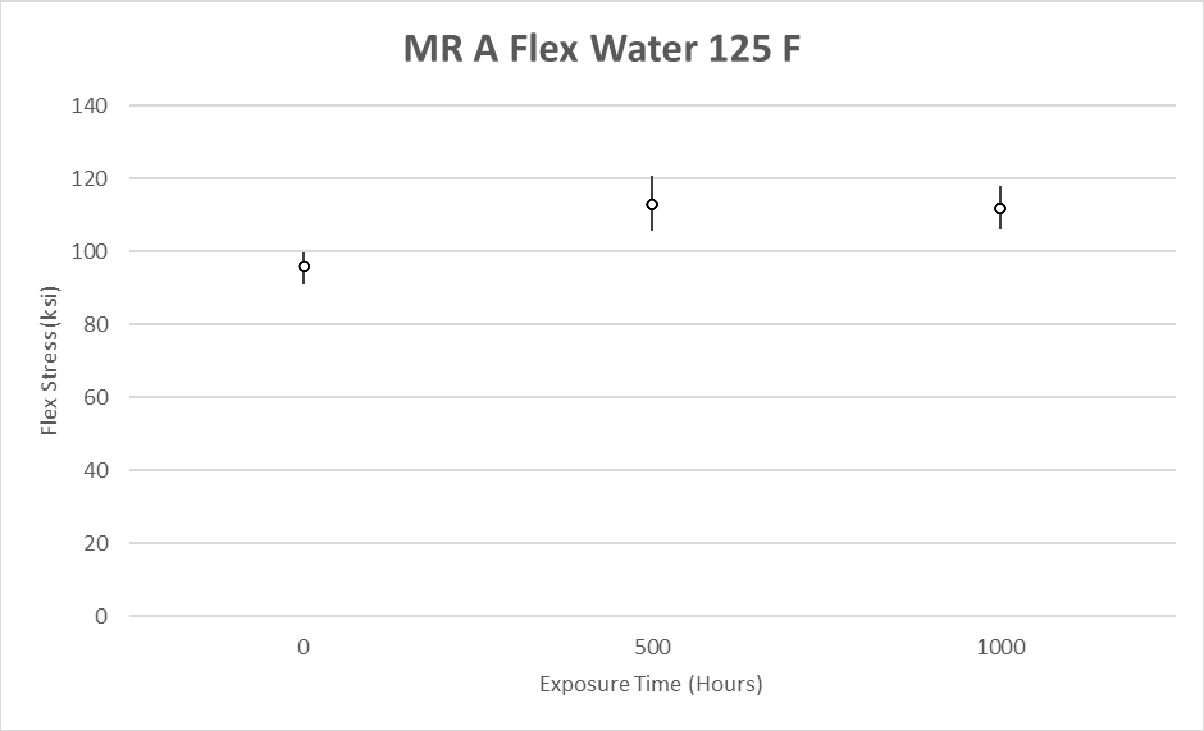


Figure 37 - MR A 125°F Water Exposure Flexural Strength

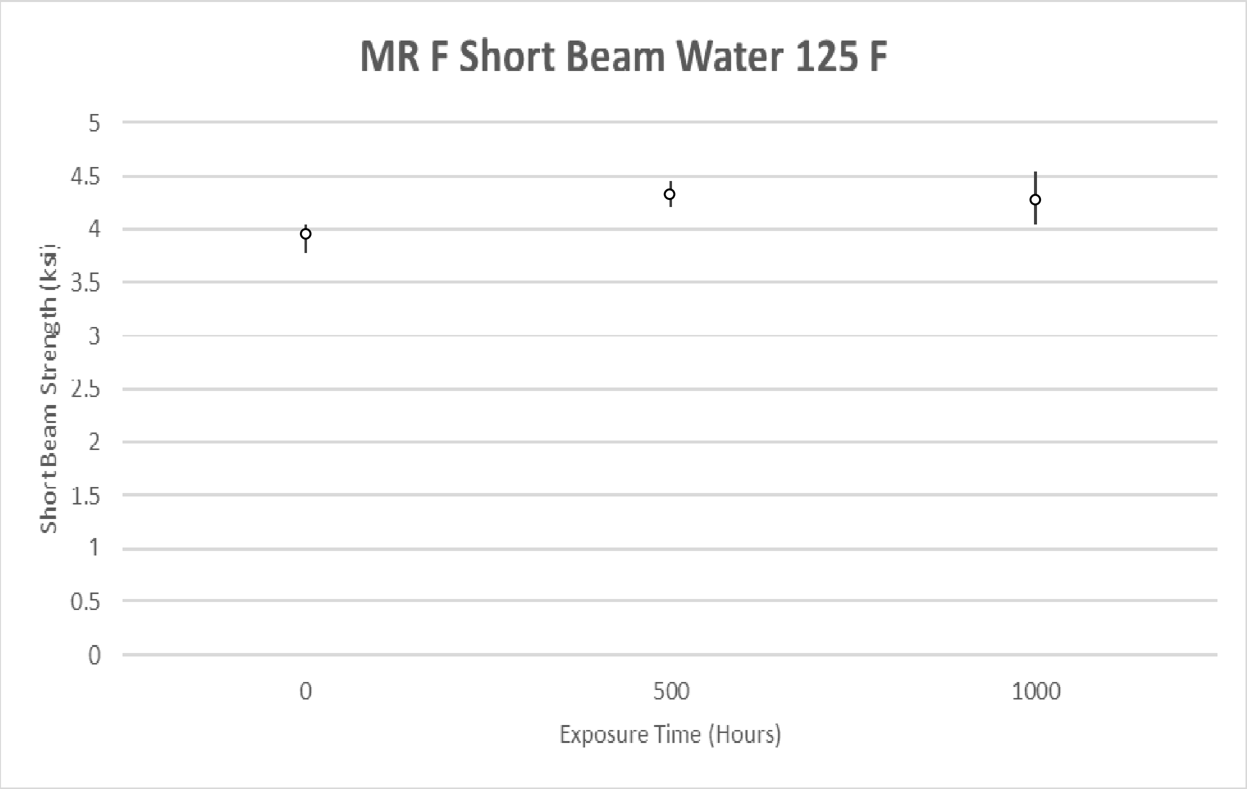


Figure 38 - MR F 125°F Water Exposure Short Beam Strength

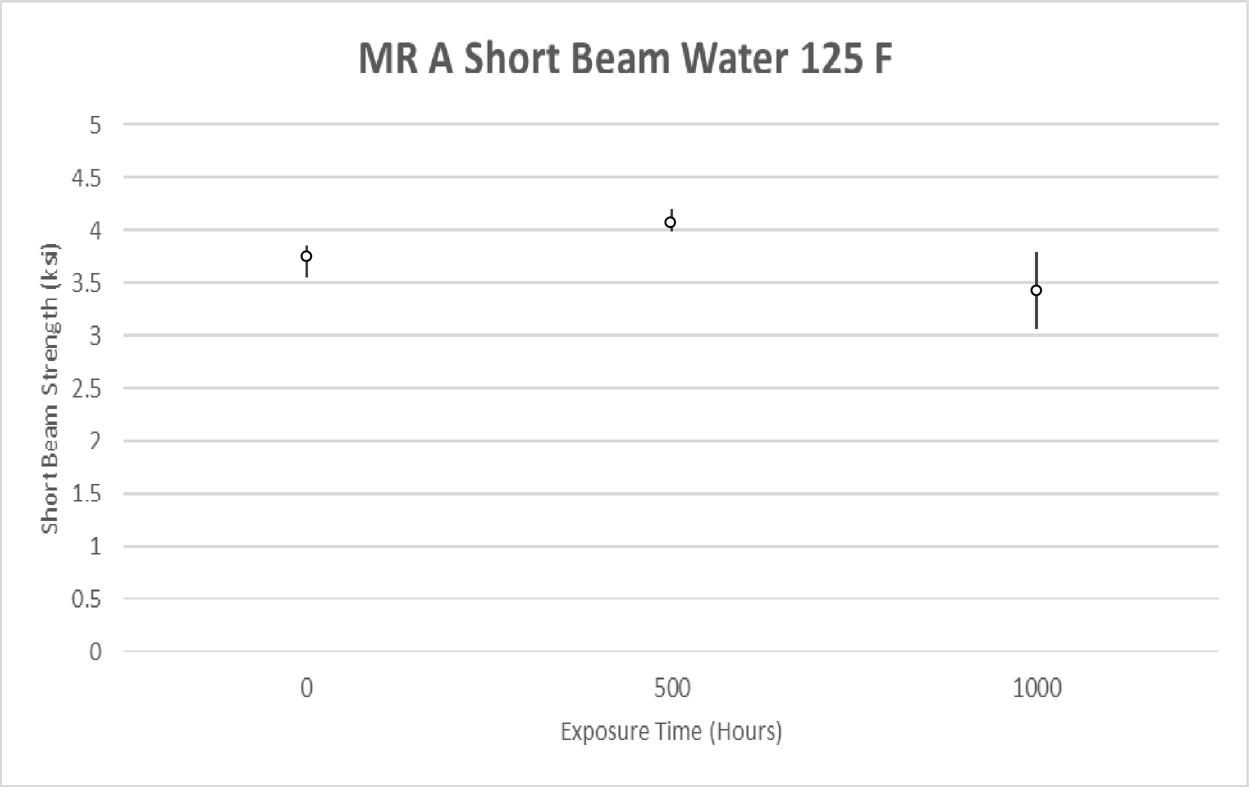


Figure 39 - MR A 125°F Water Exposure Short Beam Strength

Salt Water

The estimations for salt water are similar to those of distilled water. However, if the composite is susceptible to salt concentrations, then a salt gradient is likely to form and suffer attack via osmosis. The data indicates the saltwater did not differ significantly from the distilled water significantly. Specimens would likely display a higher variability in strengths if a UV source were coupled with the salt exposure. This would be a more accurate representation of the conditions these composites will find themselves if they are subject to real world applications. This result conforms with expectations that the salt water will behave similarly to distilled water for short term exposure times. The most noticeable physical difference was due to the accumulation of brown salt molecules on the surface of the samples. Figures 40-43 show the strengths after exposure to a salt water environment. Measured and calculated values for each specific run can be seen in the Appendix in Tables 40-47.

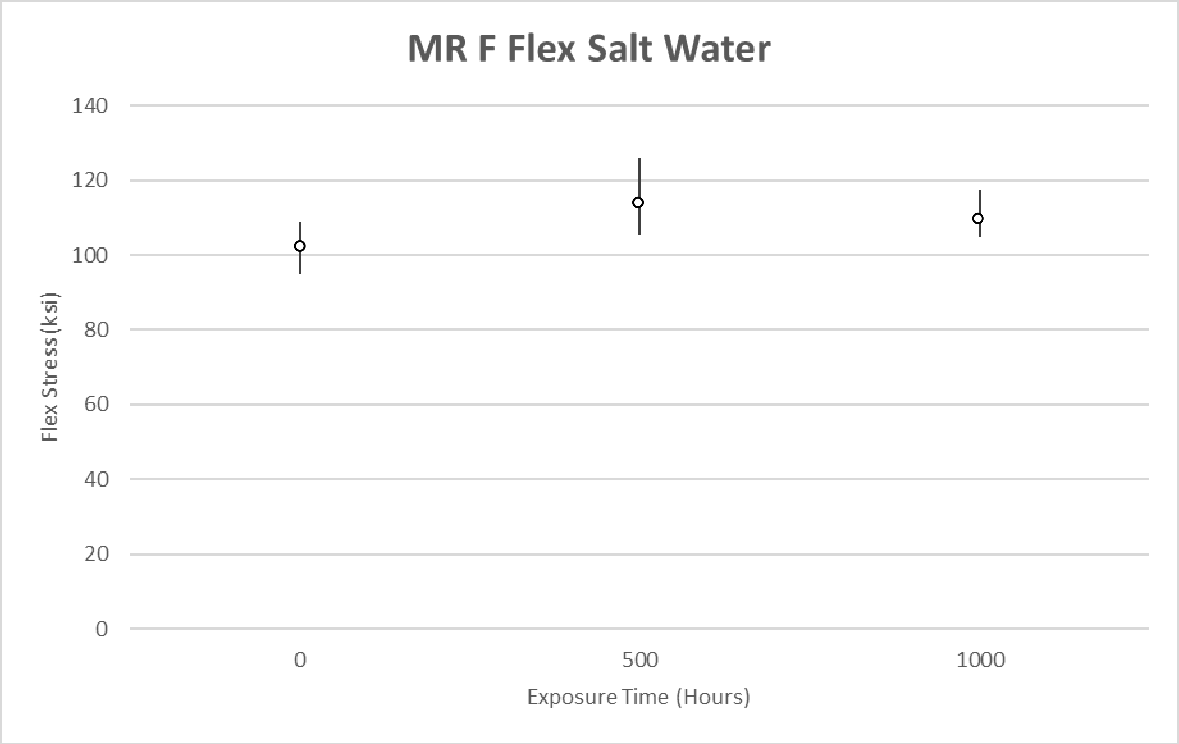


Figure 40 - MR F Salt Water Exposure Flexural Strength

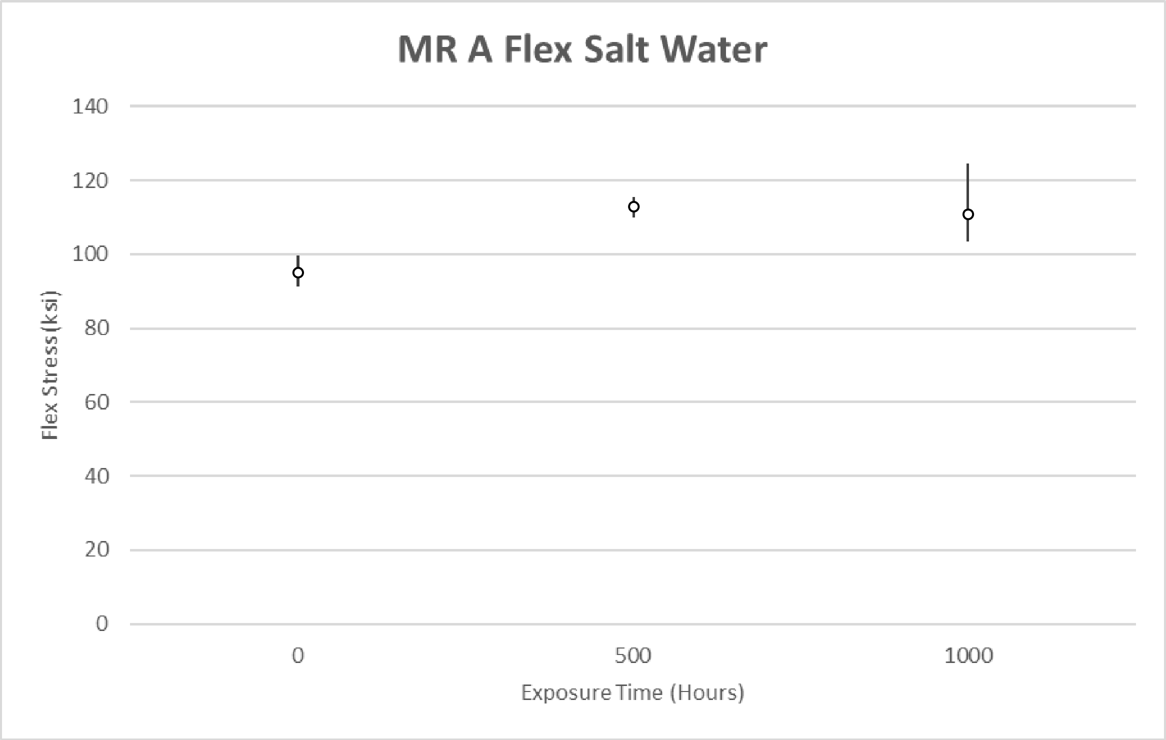


Figure 41 - MR A Salt Water Exposure Flexural Strength

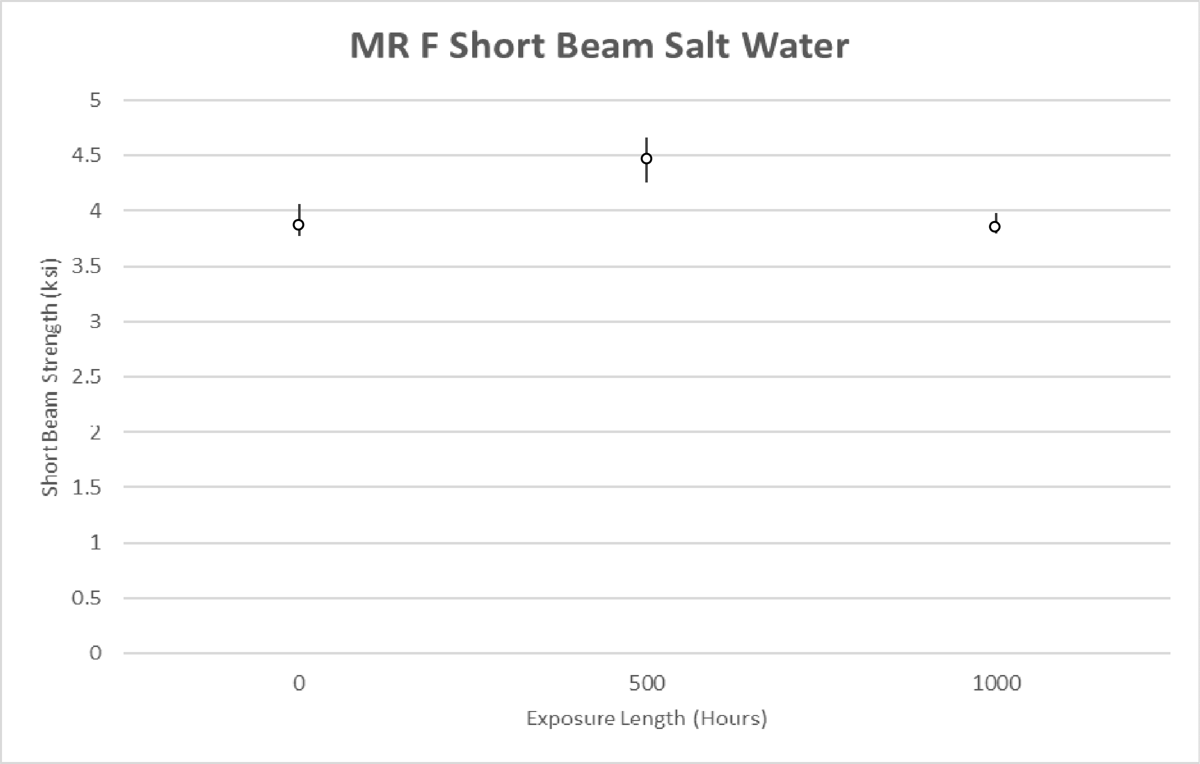


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Figure 42 - MR F Salt Water Exposure Short Beam Strength

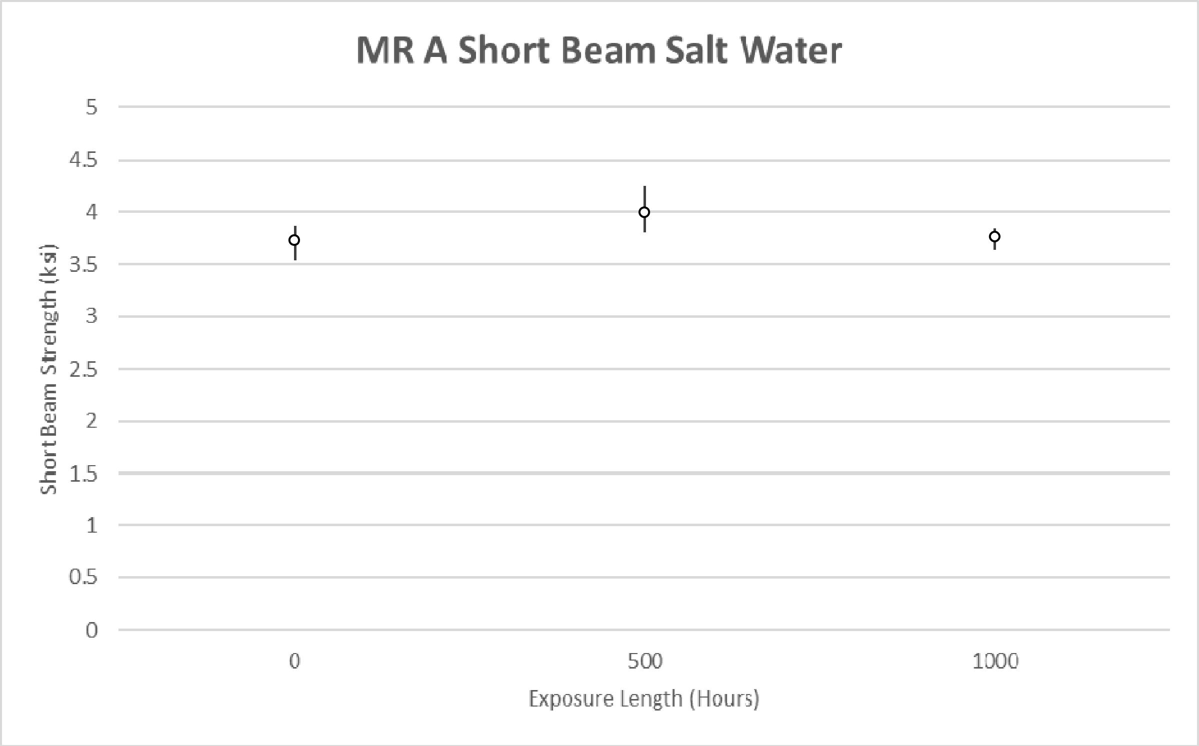

Figure 43 - MR A Salt Water Exposure Short Beam Strength

Comparison of Exposure Tests

A side by side comparison allows for a direct comparison between the environments. The comprehensive results of the effects of environmental exposure can be seen below in Figures 44-47. These show each material's average strength before and after exposure as a percentage of the unaged specimen. With the exception of bleach, the flexural strengths did not decrease as initially imagined. The increase in strength for these cases is a strong indication that the failure modes had a significant role in the mechanical characterization. Because the flexural samples all failed in interlaminar shear, the cause of failure is most likely the bonding between the fibers and the matrix. The UV had the most significant increase in strength increase which is indicative of secondary curing. However, increases after exposure to HCl, water and salt water were not expected and do not display the pattern recognized by most studies.

The most obvious trend in short beam strength is the decline in bleach environments. This is to be expected with an uncured resin. The results from any environment that had elevated temperature or UV exposure also show that some increases in strength may be seen for some composites in the short term. The decline in short beam strength from the HCl environment differs from the flexural results. Because the failures of the short beam samples were mostly acceptable, this is a better indication of degradation. This data should not be extrapolated to determine lifecycle as the effects of longer exposure could lead to exponential degradation of the fiber/ matrix interface. Interestingly, the MR F performs better in short beam shear tests while the MR A maintains its strength better in the flexural tests.

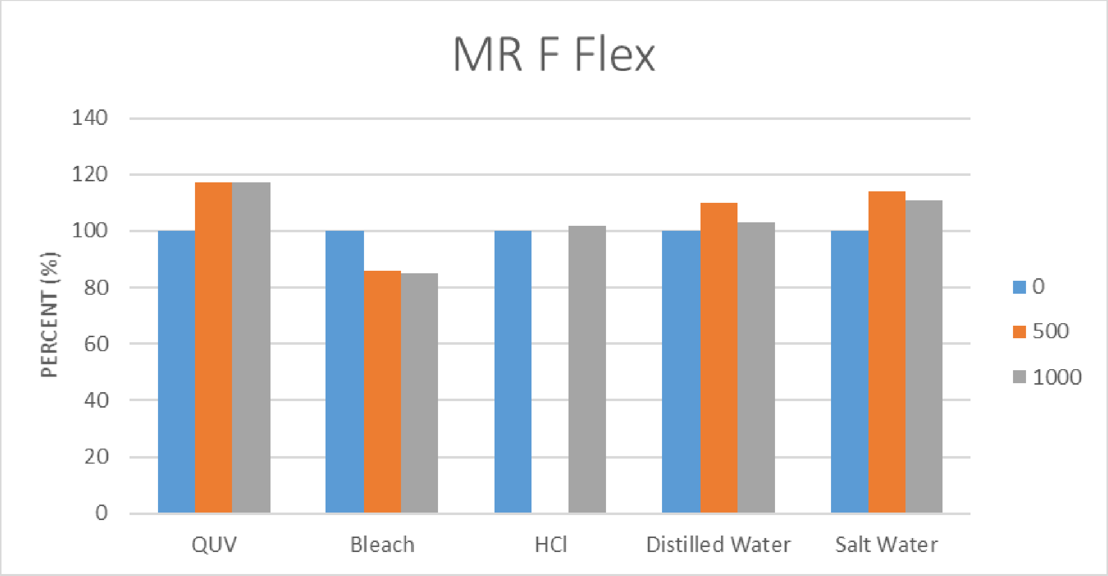

Figure 44 - MR F Flexural Strength Retention

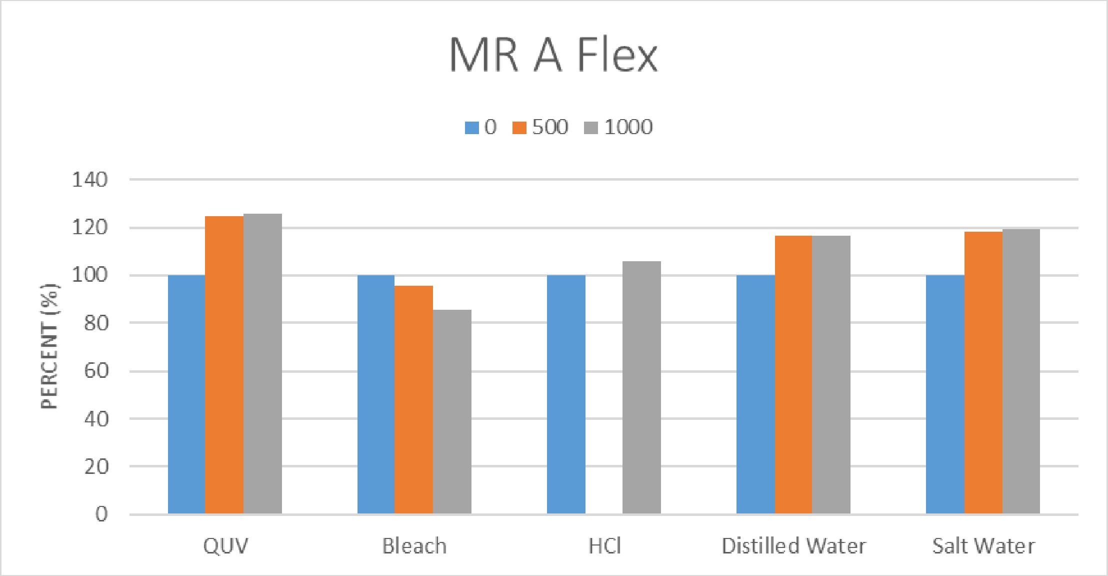

Figure 45 - MR A Flexural Strength Retention

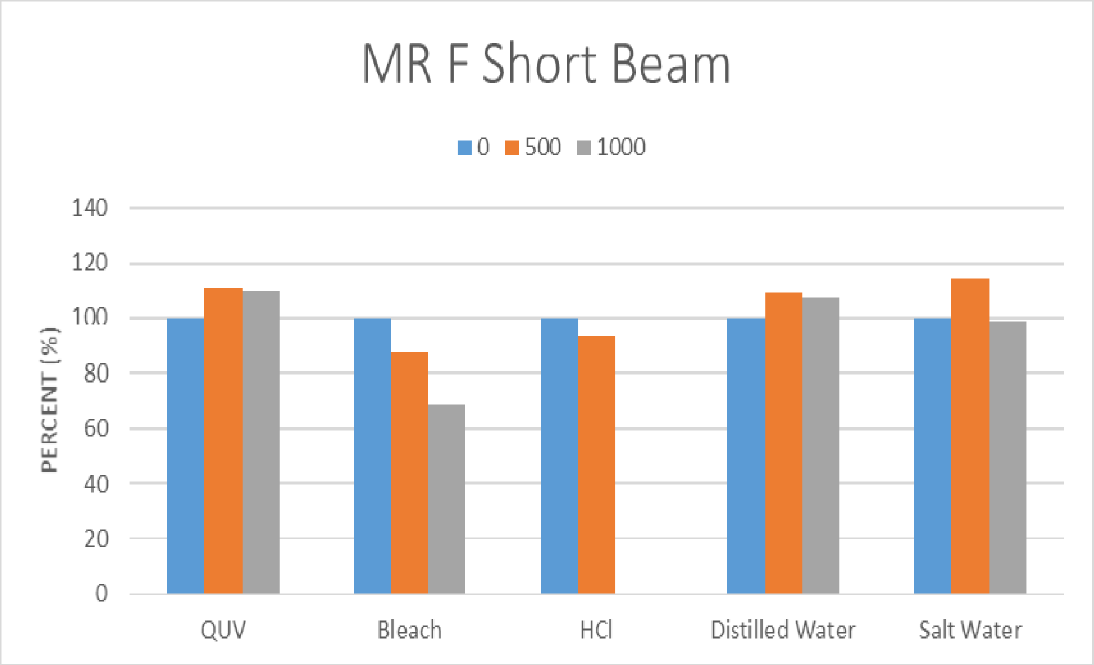

Figure 46 - MR F Short Beam Strength Retention

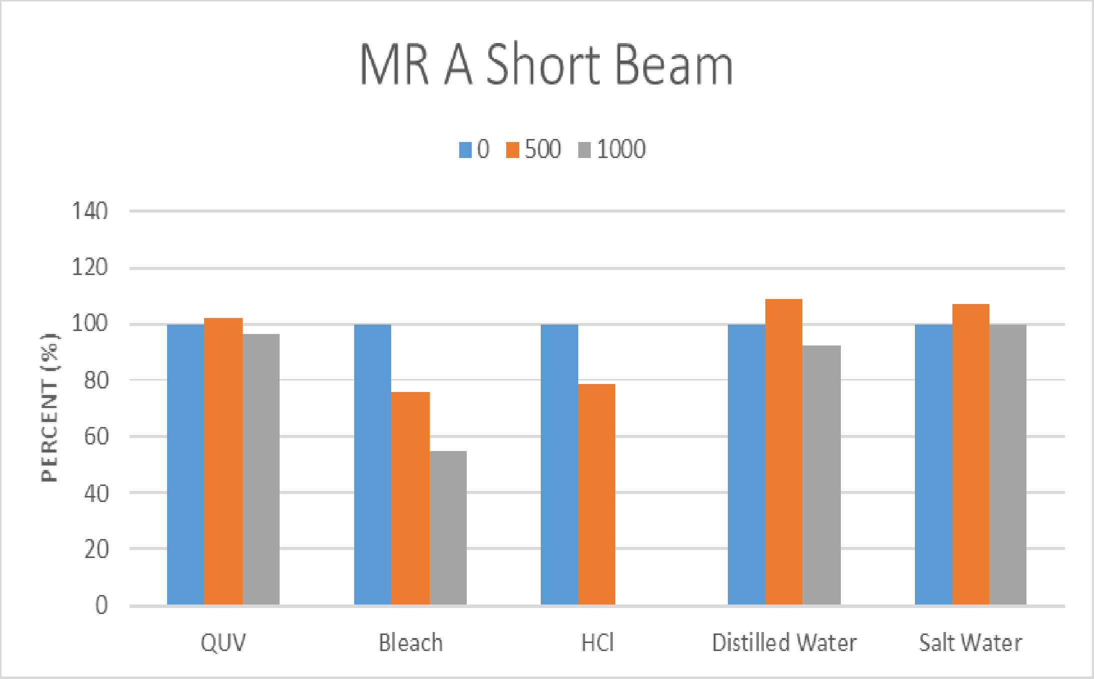

Figure 47 - MR A Short Beam Strength Retention

THERMAL TEST RESULTS

This section introduces the findings from TGA and DMA tests. Explanations for data are provided and trends are described where possible.

TGA

Further tests on the MR F and MR A were conducted to determine thermal properties. For the TGA, the heating of the sample started from room temperature and ended at 800°C at a rate of 10°C/min. The sample was heated under a Nitrogen atmosphere to ensure no combustion. Shown in Figures 48 and 49 are the TGA results for the two composites. The remaining masses for both the MR A and MR F were 81.02% and 81.68% respectively. Figure 50 shows a comparison between the two graphs for both percentage mass remaining and the derivatives of the mass remaining curves. The derivatives indicate the highest change in mass and occur near the same temperature, hinting to the chemical closeness of the two mold releases. The value for the onset temperature of decomposition is roughly 375°C. This can be compared with the previously mentioned results from Bai et al. showing that the increased fiber volume has a large effect on the onset temperature [17]. This does not ensure that the product will maintain its material integrity until the onset temperature as increased plasticization may cause the mechanical properties of the material to decline.

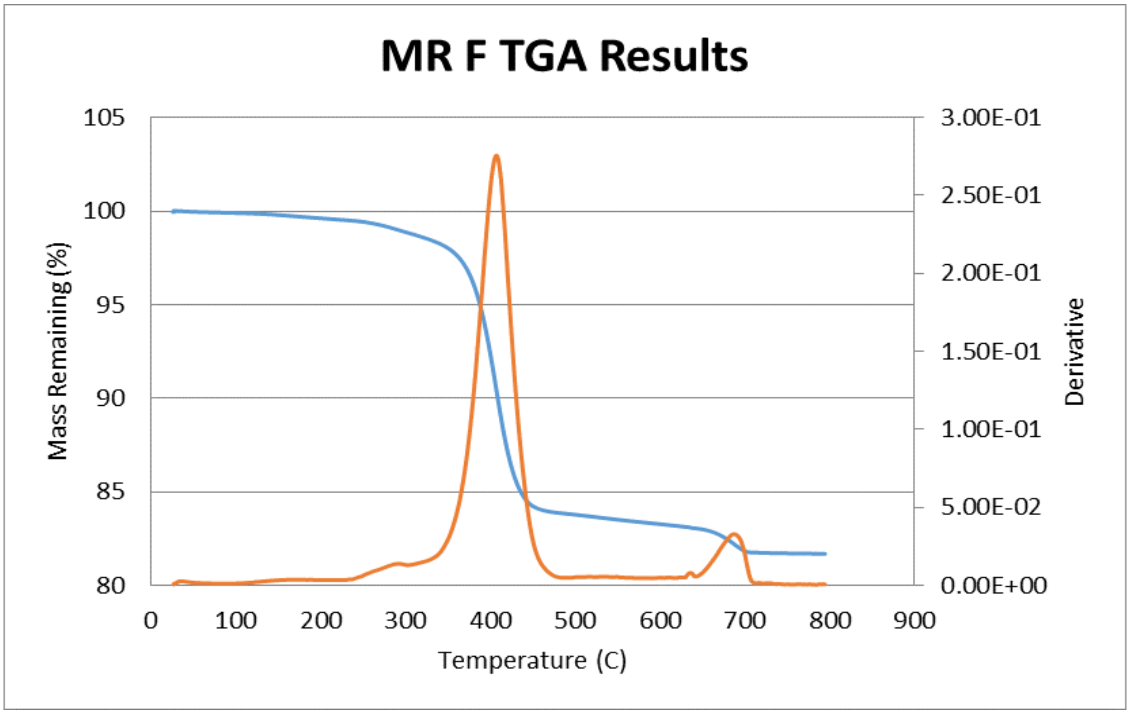

Figure 48 - MR F TGA Results

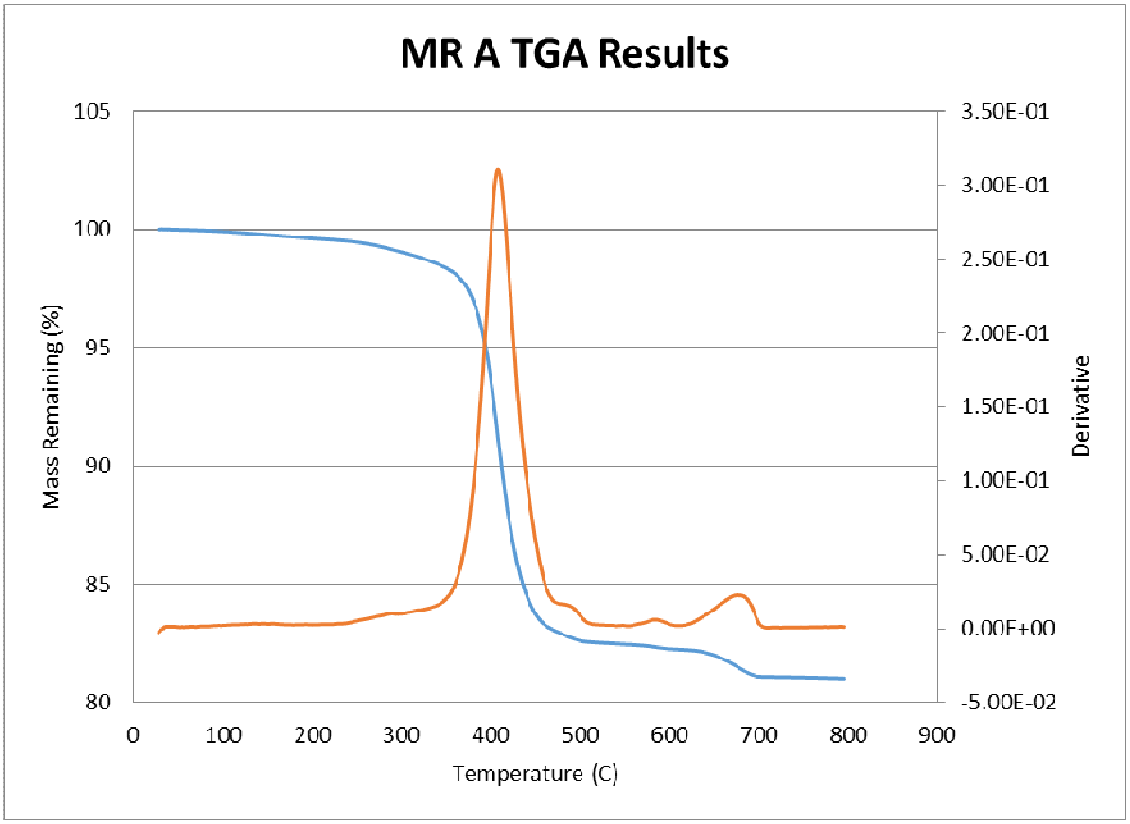

Figure 49 - MR A TGA Results

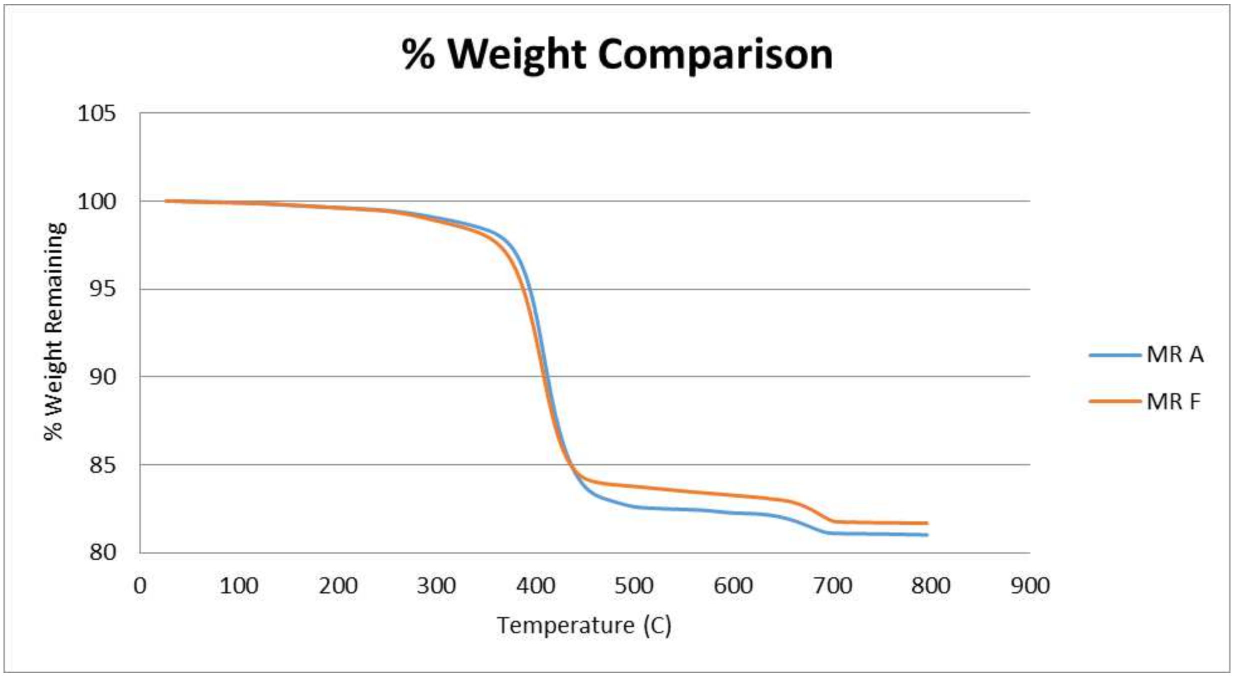

Figure 50 - TGA Comparison

DMA

The second thermal experiment performed was a DMA using a dual cantilever. This test allows an approximation of T_g. However, it should be noted that the T_g is dependent on the frequency at which the specimen is oscillated as well the heating rate. The heating rate for each test was set at 10°C/min as in the previous TGA tests. The composites were tested under 1 Hz oscillation to determine storage and loss modulus as well as the tan delta (which is the criterion under which the T_g was estimated). Because the initial test for MR F was only performed until 200°C, an additional DMA test was run for the same material at 10 Hz until 300°C. The values of 1 and 10 Hz were selected to ensure no premature rupture would result from excessive applied oscillatory force. The additional 10 Hz test was to determine the oscillation effect on the T_g value. Figure 51 shows the tan delta values for each of the three tests while Table 7 shows the maximum value for tan delta as well as the corresponding temperatures for those maximums.

The data indicates an increase in oscillation does have a noticeable effect on the storage and loss moduli and subsequent T_g values.

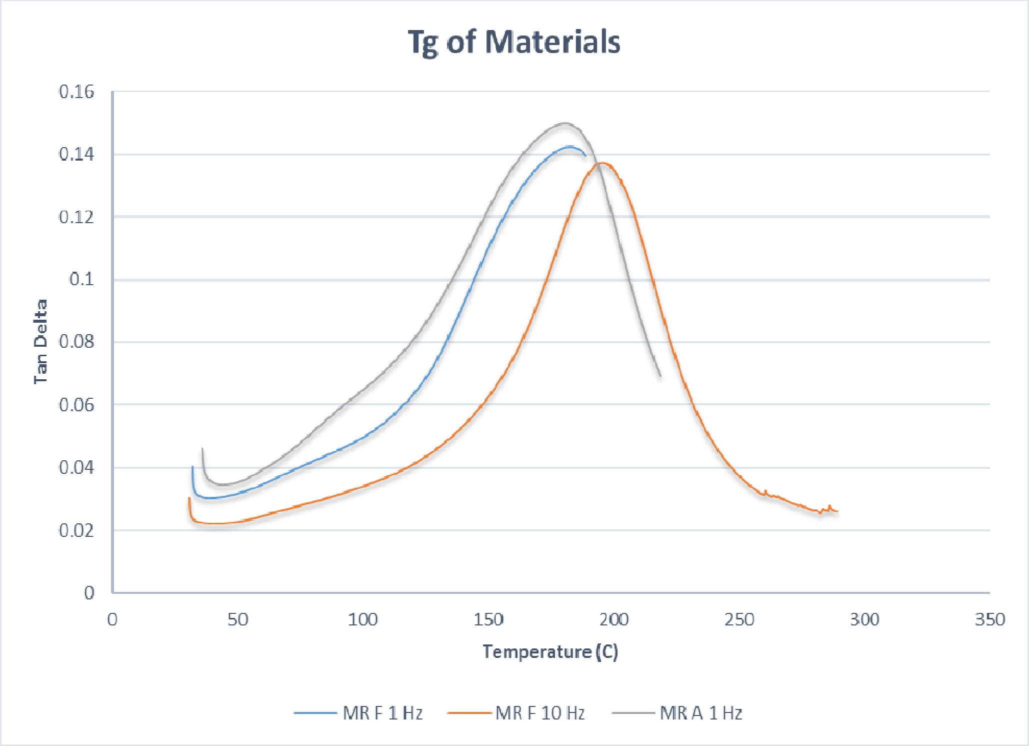

Figure 51 - Tan Delta Values

Table 7 - T_g Values from Tan Delta

	MR F 1 Hz	**MR F 10 Hz**	**MR A 1 Hz**
Max Tan δ	0.14254	0.13739	0.14973
T_g from Tan Delta (°C)	183	195	181

CONCLUSIONS AND RECOMMENDATIONS

Three different experimental mold releases with three different added pigments were pultruded after being mixed with an unsaturated polyester resin to determine their pull forces and surface qualities in comparison to a commercially available mold release. The experimental mold release that exhibited the best surface quality and pull force average was used for further mechanical evaluation. This included subjecting flexural and short beam samples to UV, bleach, HCl, elevated temperature distilled water, and salt water for time periods of 500 and 1000 hours. The properties most notably decreased for both the bleach and HCl samples and increased for those that had elevated temperature environments which was most likely due to secondary curing. With the exception of the exposure to HCl, the mold releases did not seem to directly account for significant bonding issues – that is, the difference in mold release was not detrimental in the bonding of matrix to the fiber during exposure. Further DMA and TGA tests were completed on the unaged samples to determine that the glass transition temperature and onset temperature values were nearly identical for both composites.

There are two significant recommendations to this experiment that would yield a more comprehensive examination of the environmental effects. The first is an increase in the span length for the flexural tests. Because the materials failed in shear rather than in a pure flexural mode, this indicates the fibers did not bond well with the resin. Increasing the span or modifying the resin to increase bonding with the silane coated fibers will improve flexural strength values. An increased span length will increase the likelihood of failure in flexure and should yield a more accurate representation of a material's response to environmental exposure. The failure should occur due to tensile forces shown in Figure 8.

The second improvement would be completing further crosslinking within the composite. Because strength properties for the QUV and elevated water on average increased over the first 500 hours of exposure, it is difficult to determine the effects these environments had in the first few hundred hours of exposure. There are a few ways to increase the crosslinking density during pultrusion. The easiest and most efficient are:

1) Operate the machine at a lower pull speed – This allows the material more time inside the die to cure.
2) Increase die zone temperatures – Increasing the temperature can allow for the reactions to occur more quickly than previously.
3) Modify the catalyst – By changing the amount or type of initiator, better results can be observed.
4) Include secondary cure – This could be an added zone where temperature is increased, or it could be as simple as placing the composite in an oven afterwards. These options can be risky, though, as too much heat or time in an oven may cause other issues such as crack development.

These methods, or a combination of them, may be utilized to ensure that the composite has undergone as much crosslinking as possible. However, it is unrealistic to assume 100% cure for any resin system. With a more complete cross linking, the results from exposures will be more apparent.

Further recommendations would be to include larger ranges of exposure time and the addition of DMA testing of composites post-exposure. The former would give a larger data set that allows for a more apparent trend to be seen while the latter provides T_g data that would indicate plasticization of the matrix. The glass transition temperature may expose trends in the material property reduction from the environmental degradation and may be useful for modeling predicted strengths.

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APPENDIX

Table 8 - As Pulled MR F Flexural Values

Sample Name	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
90	3.03	1.027	0.127	451	102.10
91	3.0275	1.023	0.127	422	95.91
92	3.013	1.017	0.127	442	101.05
93	3.034	1.021	0.127	417	94.96
94	3.026	1.024	0.127	479	108.76
30	3.03	1.0175	0.126	447	103.77
36	3.013	1.013	0.126	467	108.89
Average					102.20

Table 9 - As Pulled MR A Flexural Values

Sample Name	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
95	3.0195	1.0225	0.127	428	97.32
96	3.036	1.027	0.127	417	94.40
97	3.036	1.0255	0.127	402	91.14
98	3.0265	1.021	0.127	438	99.74
Average					95.65

Table 10 - As Pulled MR F Short Beam Values

Sample Name	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
90	0.7485	0.2475	0.1275	166.6	3.96
91	0.752	0.248	0.1275	171	4.06
92	0.75	0.253	0.1275	162	3.77
93	0.752	0.249	0.1275	169.9	4.01
Average					3.95

Table 11 - As Pulled MR A Flexural Values

Sample Name	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
95	0.752	0.2485	0.127	149	3.54
96	0.752	0.247	0.127	158.3	3.78
97	0.7515	0.2465	0.1265	160.5	3.86
98	0.747	0.2495	0.1265	160.3	3.81
Average					3.75

Table 12– 500 Hour QUV MR F Flex Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
37	13.096	13.093	-0.0229	3.021	1.008	0.1265	523	121.59
38	13.111	13.108	-0.0228	3.024	1.0065	0.126	513	120.39
39	13.104	13.099	-0.0381	3.014	1.009	0.126	485	113.54
40	13.15	13.147	-0.0228	3.012	1.01	0.126	488	114.13
41	13.182	13.178	-0.0303	3.014	1.013	0.126	550	128.25
Average			-0.0274					119.58

Table 13 – 1000 Hour QUV MR F Flex Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
7	13.12	13.09	-0.2286	3.0125	1.005	0.1265	536	124.98
8	13.115	13.077	-0.2897	3.0115	1.0035	0.1275	509	117.01
9	13.11	13.086	-0.1830	3.012	1.0035	0.127	483	111.91
10	13.167	13.12	-0.3569	3.0175	1.005	0.127	555	128.40
11	13.228	13.21	-0.1360	3.0155	1.012	0.127	511	117.40
Average			-0.2388					119.94

Table 14 – 500 Hour QUV MR A Flex Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
79	13.0911	13.09	-0.0084	3.008	1.009	0.1265	477	110.78
80	13.125	13.127	0.0152	3.014	1.009	0.1265	565	131.22
81	13.134	13.134	-0.0000	3.014	1.01	0.1265	512	118.80
82	13.204	13.206	0.0151	3.017	1.012	0.126	511	119.27
83	13.264	13.263	-0.0075	3.018	1.015	0.1265	504	116.36
Average			-0.0029					119.29

Table 15 – 1000 Hour QUV MR A Flex Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
49	13.177	13.151	-0.1973	3.015	1.013	0.126	526	122.65
50	13.212	13.158	-0.4087	3.0175	1.013	0.126	522	121.72
51	13.198	13.178	-0.1515	3.015	1.0135	0.1265	461	106.59
52	13.155	13.11	-0.3421	3.0155	1.011	0.1265	550	127.49
53	13.138	13.105	-0.2512	3.0145	1.01	0.126	521	121.84
Average			-0.2702					120.06

Table 16 – 500 Hour QUV MR F Short Beam Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
37	0.7993	0.7993	0	0.756	0.252	0.1265	158.1	3.72
38	0.805	0.8046	-0.0496	0.753	0.253	0.127	181.2	4.23
39	0.8008	0.8001	-0.0874	0.7515	0.2535	0.1265	195.9	4.58
40	0.8026	0.8026	0	0.7565	0.253	0.127	204.5	4.77
41	0.8261	0.8252	-0.1089	0.749	0.2585	0.1265	200.4	4.60
Average			-0.0492					4.38

Table 17 – 1000 Hour QUV MR F Short Beam Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
7	0.828	0.8264	-0.1932	0.7495	0.259	0.1265	196.1	4.49
8	0.7979	0.7963	-0.2005	0.7485	0.252	0.1265	193.3	4.55
9	0.8051	0.8041	-0.1242	0.7535	0.253	0.127	162.8	3.80
10	0.8005	0.8001	-0.0499	0.749	0.2535	0.126	197.1	4.63
11	0.8043	0.8013	-0.3729	0.752	0.2535	0.127	177.2	4.13
Average			-0.1881					4.32

Table 18 – 500 Hour QUV MR A Short Beam Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
79	0.8059	0.8057	-0.0248	0.755	0.253	0.127	165.8	3.87
80	0.8031	0.8039	0.0996	0.76	0.251	0.1265	173.5	4.10
81	0.7975	0.7963	-0.1504	0.7505	0.2535	0.127	160.2	3.73
82	0.8093	0.8103	0.1235	0.7545	0.254	0.127	158.2	3.68
83	0.813	0.8124	-0.0738	0.74	0.2585	0.127	163.5	3.74
Average			-0.00518					3.82

Table 19 – 1000 Hour QUV MR A Short Beam Test

Sample Name	Initial Mass (g)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
49	NA	0.751	0.2565	0.126	156.4	3.63
50	NA	0.7555	0.251	0.127	155.9	3.67
51	NA	0.7505	0.253	0.127	143.5	3.35
52	NA	0.756	0.259	0.127	160.1	3.65
53	NA	0.749	0.2485	0.127	155.9	3.70
Average						3.60

Table 20– 500 Hour Bleach MR F Flex Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
1	13.225	13.248	0.0017	3.016	1.013	0.1265	422	97.62
2	13.214	13.255	0.0031	3.024	1.0095	0.127	358	82.45
3	13.154	13.185	0.0024	3.0125	1.0085	0.127	359	82.76
Average			0.0024					87.61

Table 21 – 1000 Hour Bleach MR F Flex Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
13	13.115	13.183	0.0052	3.01	1.01	0.127	382	87.94
14	13.098	13.172	0.0056	3.008	1.0075	0.127	374	86.31
15	13.207	13.28	0.0055	3.014	1.0125	0.1265	383	88.64
16	13.258	13.337	0.0060	3.02	1.012	0.127	374	85.92
17	13.149	13.219	0.0053	3.0185	1.012	0.127	377	86.61
Average			0.0055					87.06

Table 22 – 500 Hour Bleach MR A Flex Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
43	13.283	13.319	0.0027	3.0175	1.0195	0.127	397	90.54
44	13.324	13.363	0.0029	3.028	1.0175	0.127	434	99.17
45	13.254	13.288	0.0026	3.021	1.015	0.1265	366	84.50
Average			0.0027					91.40

Table 23 – 1000 Hour Bleach MR A Flex Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
55	13.27	13.337	0.0050	3.016	1.015	0.1265	372	85.89
56	13.303	13.378	0.0056	3.017	1.0155	0.1265	375	86.54
57	13.21	13.286	0.0058	3.0115	1.015	0.127	346	79.26
58	13.253	13.332	0.0060	3.024	1.013	0.1265	355	82.12
59	13.197	13.274	0.0058	3.0165	1.012	0.1265	361	83.59
Average			0.0056					81.66

Table 24 – 500 Hour Bleach MR F Short Beam Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
1	0.7964	0.7888	-0.0095	0.752	0.251	0.1265	145	3.42
2	0.785	0.8	0.0191	0.7505	0.2495	0.1265	155.8	3.70
3	0.8035	0.808	0.0056	0.755	0.251	0.126	137.5	3.26
Average			0.0051					3.46

Table 25 – 1000 Hour Bleach MR F Short Beam Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
13	0.7836	0.7888	0.0066	0.752	0.248	0.1265	105.3	2.51
14	0.7978	0.8031	0.0066	0.7435	0.254	0.1265	106.6	2.48
15	0.7836	0.7882	0.0059	0.755	0.2475	0.1255	112.4	2.71
16	0.798	0.8029	0.0061	0.7555	0.2505	0.126	124.5	2.95
17	0.7749	0.7805	0.0072	0.7425	0.248	0.1265	116.8	2.79
Average			0.0065					2.69

Table 26 – 500 Hour Bleach MR A Short Beam Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
43	0.787	0.7933	0.0080	0.7495	0.251	0.1275	128.7	3.01
44	0.8063	0.8113	0.0062	0.7515	0.2545	0.127	127.7	2.96
45	0.7997	0.807	0.0091	0.7465	0.2555	0.127	110.2	2.54
Average			0.0078					2.84

Table 27 – 1000 Hour Bleach MR A Short Beam Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
55	0.7944	0.8023	0.0099	0.741	0.254	0.127	96.9	2.25
56	0.7956	0.8034	0.0098	0.741	0.2545	0.127	81.4	1.88
57	0.7838	0.791	0.0092	0.748	0.249	0.1275	82	1.93
58	0.7801	0.7866	0.0083	0.753	0.247	0.1275	86.9	2.06
59	0.7825	0.7892	0.0086	0.755	0.2475	0.127	88.4	2.10
Average			0.0092					2.05

Table 28 – 1000 Hour HCl MR F Flex Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
19	13.05	13.002	-0.0037	3.0065	1.008	0.127	416	95.95
20	13.02	12.98	-0.0031	3.004	1.007	0.127	436	100.67
21	13.046	13.004	-0.0032	3.0085	1.0075	0.127	399	92.08
22	13.076	13.04	-0.0028	3.015	1.0075	0.127	436	100.62
23	13.069	13.02	-0.0037	3.009	1.007	0.127	471	108.75
100	13.3	13.272	-0.0021	3.027	1.019	0.127	469.1	107.03
101	13.367	13.33	-0.0028	3.026	1.0215	0.1265	512	117.46
102	13.44	13.41	-0.0022	3.028	1.025	0.127	476.2	108.02
Average			-0.0029					103.82

Table 29 – 1000 Hour HCl MR A Flex Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
61	13.078	13.02	-0.0044	3.0085	1.0085	0.1265	420	97.59
62	13.058	13.003	-0.0042	3.005	1.0075	0.127	372	85.85
63	13.059	12.99	-0.0053	3.0095	1.008	0.1265	440	102.29
64	13.1	13.013	-0.0066	3.0125	1.008	0.1265	426	99.04
65	13.093	13.024	-0.0053	3.009	1.008	0.127	439	101.26
103	13.34	13.3	-0.0030	3.028	1.023	0.1265	469.1	107.46
104	13.32	13.28	-0.0030	3.028	1.022	0.127	469	106.70
105	13.417	13.39	-0.0020	3.031	1.025	0.127	486.5	110.35
Average			-0.0042					101.32

Table 30 – 500 Hour HCl MR F Short Beam Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
100	0.8011	0.7927	-0.0105	0.752	0.253	0.127	148.4	3.46
101	0.806	0.7981	-0.0098	0.752	0.254	0.127	159.2	3.70
102	0.817	0.809	-0.0098	0.751	0.257	0.127	172.1	3.95
19	0.7862	0.7764	-0.0125	0.743	0.2505	0.126	149.2	3.55
20	0.7911	0.7803	-0.0137	0.7565	0.2475	0.126	153.4	3.69
21	0.7976	0.7897	-0.0099	0.753	0.251	0.126	138.6	3.29
22	0.778	0.7678	-0.0131	0.7545	0.248	0.1265	173.2	4.14
23	0.782	0.7733	-0.0111	0.752	0.2485	0.1265	157.9	3.77
Average			-0.0113					3.69

Table 31 – 500 Hour HCl MR A Short Beam Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
103	0.7878	0.7764	-0.0145	0.749	0.2505	0.127	121.6	2.87
104	0.8016	0.7904	-0.0140	0.7485	0.255	0.1275	134.5	3.10
105	0.7982	0.7914	-0.0085	0.748	0.2545	0.127	117.2	2.72
61	0.7907	0.7823	-0.0106	0.752	0.25	0.127	122.8	2.90
62	0.7956	0.7878	-0.0098	0.758	0.249	0.1275	124.1	2.93
63	0.8061	0.7977	-0.0104	0.743	0.2555	0.127	125.2	2.89
64	0.7913	0.7827	-0.0109	0.746	0.2525	0.1275	131.8	3.07
65	0.7989	0.7912	-0.0096	0.757	0.251	0.127	131	3.08
Average			-0.0110					2.95

Table 32– 500 Hour Water MR F Flex Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
4	13.123	13.175	0.0040	3.011	1.0065	0.1275	462	105.89
5	13.08	13.16	0.0061	3.0085	1.005	0.127	503	116.37
6	13.082	13.141	0.0045	3.008	1.004	0.127	497	115.09
Average			0.0049					112.45

Table 33 – 1000 Hour Water MR F Flex Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
25	13.058	13.139	0.0062	3.013	1.006	0.127	504	116.48
26	13.103	13.189	0.0066	3.018	1.0095	0.127	455	104.79
27	13.156	13.238	0.0062	3.016	1.0115	0.127	486	111.71
28	13.213	13.304	0.0069	3.0155	1.013	0.127	447	102.59
29	13.381	13.468	0.0065	3.02	1.024	0.1275	406	91.46
Average			0.0065					105.40

Table 34 – 500 Hour Water MR A Flex Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
46	13.207	13.268	0.0046	3.02	1.014	0.1265	458	105.85
47	13.181	13.266	0.0064	3.013	1.012	0.1265	466	107.91
48	13.1669	13.224	0.0043	3.0095	1.0125	0.1265	522	120.82
Average			0.0051					111.52

Table 35 – 1000 Hour Water MR A Flex Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
67	13.158	13.2532	0.0072	3.015	1.01	0.1265	485	112.53
68	13.151	13.2446	0.0071	3.01	1.0095	0.127	461	106.17
69	13.139	13.23	0.0069	3.0125	1.011	0.126	505	117.99
70	13.177	13.271	0.0071	3.012	1.011	0.1265	468	108.48
71	13.194	13.288	0.0071	3.009	1.014	0.1265	467	107.93
Average			0.0071					111.46

Table 36 – 500 Hour Water MR F Short Beam Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
4	0.7747	0.7808	0.0079	0.747	0.2495	0.1275	178.3	4.20
5	0.7981	0.8035	0.0068	0.7465	0.255	0.127	183.5	4.25
6	0.7966	0.8017	0.0064	0.75	0.2525	0.1275	191.2	4.45
Average			0.0070					4.30

Table 37 – 1000 Hour Water MR F Short Beam Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
25	0.812	0.8204	0.0103	0.755	0.254	0.1265	185.9	4.34
26	0.7932	0.8	0.0086	0.7555	0.249	0.127	172.9	4.10
27	0.7955	0.8046	0.0114	0.752	0.25	0.1265	171.1	4.06
28	0.799	0.8059	0.0086	0.7565	0.2495	0.126	175.9	4.20
29	0.8063	0.8129	0.0082	0.755	0.2525	0.127	194.2	4.54
Average			0.0094					4.25

Table 38 – 500 Hour Water MR A Short Beam Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
46	0.7873	0.7932	0.0075	0.7545	0.2495	0.127	168.4	3.99
47	0.7809	0.7863	0.0069	0.7535	0.2465	0.127	175.3	4.20
48	0.7804	0.7849	0.0058	0.7525	0.2465	0.1265	168.6	4.06
Average			0.0067					4.08

Table 39 – 1000 Hour Water MR A Short Beam Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
67	0.8056	0.8142	0.0107	0.753	0.2535	0.1265	152.2	3.56
68	0.7975	0.8054	0.0099	0.7485	0.253	0.127	149.5	3.49
69	0.8053	0.8142	0.0111	0.7575	0.2525	0.1275	163	3.80
70	0.7873	0.7952	0.0100	0.748	0.252	0.1275	131	3.06
71	0.784	0.792	0.0102	0.7555	0.2485	0.1275	144.9	3.43
Average			0.0104					3.47

Table 40– 500 Hour Salt Water MR F Flex Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
12	13.281	13.477	0.0148	3.02	1.013	0.1275	463	105.43
18	13.103	13.184	0.0062	3.005	1.0065	0.127	511	118.04
24	13.071	13.389	0.0243	3.011	1.0055	0.127	545	126.02
Average			0.0151					116.50

Table 41 – 1000 Hour Salt Water MR F Flex Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
31	13.311	13.43	0.0089	3.022	1.025	0.127	518.3	117.57
32	13.248	13.42	0.0130	3.0125	1.022	0.1275	519.3	117.21
33	13.211	13.476	0.0201	3.0125	1.01	0.127	455.6	104.88
Average			0.0140					113.22

Table 42 – 500 Hour Salt Water MR A Flex Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
54	13.319	13.366	0.0035	3.031	1.021	0.1275	512	115.68
60	13.364	13.387	0.0017	3.034	1.022	0.127	483	109.88
66	13.383	13.433	0.0037	3.037	1.025	0.1275	506	113.88
Average			0.0030					113.15

Table 43 – 1000 Hour Salt Water MR A Flex Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
73	13.178	13.46	0.0214	3.017	1.012	0.1265	538.3	124.65
74	13.121	13.35	0.0175	3.008	1.01	0.127	449.5	103.47
75	13.086	13.31	0.0171	3.008	1.0065	0.127	493.3	113.95
Average			0.0187					114.03

Table 44 – 500 Hour Salt Water MR F Short Beam Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
12	0.8208	0.8232	0.0029	0.753	0.256	0.126	198.2	4.61
18	0.787	0.79	0.0038	0.75	0.249	0.1265	178.9	4.26
24	0.7908	0.7921	0.0016	0.747	0.251	0.127	198.1	4.66
Average			0.0028					4.51

Table 45 – 1000 Hour Salt Water MR F Short Beam Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
31	0.8125	0.7838	-0.0353	0.7445	0.257	0.127	170.8	3.92
32	0.8046	0.7798	-0.0308	0.752	0.2535	0.127	170.8	3.98
33	0.8018	0.7781	-0.0296	0.7485	0.2545	0.127	163.4	3.79
Average			-0.0319					3.90

Table 46 – 500 Hour Salt Water MR A Short Beam Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
54	0.8158	0.8176	0.0022	0.755	0.2545	0.127	183	4.25
60	0.8133	0.817	0.0045	0.7555	0.2545	0.127	163.8	3.80
66	0.7964	0.7984	0.0025	0.7545	0.251	0.127	167.8	3.95
Average			0.0031					4.00

Table 47 – 1000 Hour Salt Water MR A Short Beam Test

Sample Name	Initial Mass (g)	Post Exposure Mass (g)	Weight Change (%)	Length (in)	Width (in)	Thickness (in)	Peak Load (lb)	Peak Stress (ksi)
73	0.8106	0.7983	-0.0152	0.7545	0.2545	0.1265	162.4	3.78
74	0.8096	0.7922	-0.0215	0.7565	0.2535	0.127	164.6	3.83
75	0.7806	0.7984	0.0228	0.752	0.248	0.127	152.7	3.64
Average			-0.0046					3.75

VITA

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