

Effect of Ageing on the Mechanical Performance of Thermoset Polymers: A Statistical Approach

Francisco M. Dos Santos^{1,2}, Livia Ávila de Oliveira^{1,3}, Alysson H.S. Bueno², Leandro José da Silva¹, Gilberto García del Pino⁴ and Tulio H. Panzera^{1,*}

¹Centre for Innovation and Technology in Composite Materials (CITeC), Department of Mechanical Engineering, Federal University of São João del Rei (UFSJ), Brazil

²Surface Engineering, Tribology and Electrochemistry Centre, Department of Mechanical Engineering, Federal University of São João del Rei (UFSJ), Brazil

³Department of Natural Science (FQMat/DCNat), Federal University of São João del Rei (UFSJ), Brazil

⁴Department of Mechanical Engineering, State University of Amazonas (UEA), Manaus, Brazil

Abstract: The present work investigates the effect of three different ageing processes (natural, 100% relative humidity and salt spray) on the mechanical performance of two thermoset polymers, epoxy and polyester, commonly used as matrix phase in composite materials. A full factorial design is conducted to evaluate the effect of significant factors and interactions on moisture absorption, tensile and compressive strength and modulus of elasticity of the thermosets. Both polymers reveal a decrease in moisture absorption in the saline environment compared to the completely saturated condition (100% RH). Polyester polymers in harsh environments exhibit higher compressive properties compared to those subjected to natural conditioning. In general, polyester polymers, which are most affected by the positive effect of additional cross-linking, have less moisture absorption and superior mechanical properties compared to epoxy, which is more affected by the negative effect of plasticization.

Keywords: Epoxy, polyester, salt spray, mechanical properties, moisture absorption, plasticization.

1. INTRODUCTION

Polymers are widely used in every area of human pursuits due to their versatility and remarkable performance. Based on their behaviour in relation to thermal treatments, they are classified into thermoplastics and thermosets [1]. The defining characteristic of a thermoset is the presence of covalent intermolecular chemical cross-links that create greater strength and stiffness and reduce creep susceptibility compared to its thermoplastic counterparts. As a result, they become less susceptible to damage by thermal and chemical impulses from the nearby environment, making them highly suitable for use in structural and protective applications, such as adhesives, high-performance coatings, sealants, electronic devices, aerospace components and many others, while the thermoplastics are employed in light-duty utilities [1-3]. Properties like thermal stability and mechanical performance of thermosets depend largely on factors such as chemistry, composition, molecular structure, catalyst, crosslink density, degree of polymerization, thermal and curing conditions [1, 4].

Epoxy is one of the most versatile categories of thermosetting polymers, being widely used for coatings, electronic materials, adhesives, construction and matrices for fibre-reinforced composites. Its outstanding properties, such as good mechanical properties, high adhesion strength, low curing shrinkage, corrosion/chemical resistance and electrical insulation, are attributed to its unique three-dimensional crosslinking network structure originated by the reaction of epoxy groups with high molecular polymers and corresponding curing agents [5, 6]. On the other hand, unsaturated polyester resin is one of the most popular thermoset polymers used in advanced composite structures because of its large range of mechanical properties, good processing characteristics, low cost, low weight and good corrosion resistance. The curing of polyester resin is highly exothermic in nature, in which the resin is transferred from liquid state into a rigid cross-linked molecular structure that becomes insoluble and infusible, ensuring good mechanical performance to the polymer [7, 8].

The wide use of these polymers in tanks, tubes, vessels, outdoor applications and many other aggressive environments, makes it necessary to well understand their long-term stability in the presence of external factors that cause physical and mechanical degradation, such as temperature, oxygen, water,

*Address correspondence to this author at the Centre for Innovation and Technology in Composite Materials (CITeC), Department of Mechanical Engineering, Federal University of São João del Rei (UFSJ), Brazil; Tel/Fax: +55 32 33795879; E-mail: panzera@ufsj.edu.br

chemicals, radiations, etc [9, 10]. In recent years, a large number of studies related to the physical-mechanical properties of epoxy and polyester matrix composites after ageing in different environments have been carried out [11-17]. However, the study of these neat polymers, which can have many other applications in addition to composite matrices, and the comparison of their properties after the ageing process through robust statistical analysis, is still very limited. Based on this, the present work investigates the physical (moisture absorption) and mechanical (tensile and compressive strength and modulus of elasticity) properties of neat polyester and epoxy polymers subjected to three different environmental conditions: natural, 100% relative humidity and salt spray. In addition, a full factorial design of experiment (DoE) is conducted to assess and compare the effect of individual factors and interactions on their properties.

2. MATERIALS AND METHODS

2.1. Materials

Two different types of polymers are tested: a low viscosity epoxy resin (Araldite LY 5052) combined with the Aradur 5052 hardener, supplied by Huntsman, and an unsaturated polyester resin (Polylite 10316-10) combined with a methyl ethyl ketone peroxide hardener (MEK-P), supplied by Reichhold.

2.2. Fabrication Process

Initially, the hardeners are weighted according to the manufacturer's recommendation (38 wt.% for the epoxy and 2 wt.% for the polyester system) and added to their respective resins. The system is manually mixed for five minutes and poured into silicon moulds (Figure 1a) for the curing process. The dimensions of the tensile and compressive specimens are based on the recommendations of ASTM D638-14 [18] and ASTM D695-15 [19], respectively. Epoxy samples are left at room temperature ($\sim 22^\circ\text{C}$ and 55% of relative humidity) for 7 days, while polyester samples are left for 12 hours at room temperature, followed by 48 hours in an oven at 60°C , based on the manufacturer's recommendation. After curing, the samples are removed from the mould (Figure 1b) and conditioned by the ageing process.

2.3. Ageing Process

The polymers are submitted to three different environmental conditions after curing: natural (no degradation), 100% relative humidity (RH) and salt spray (fog), as shown in Table 1. In the first condition, the samples are left at room temperature for a natural ageing process, while for the second and third conditions, the samples are placed in a climatic chamber model Solab SL – 206 and subjected to two

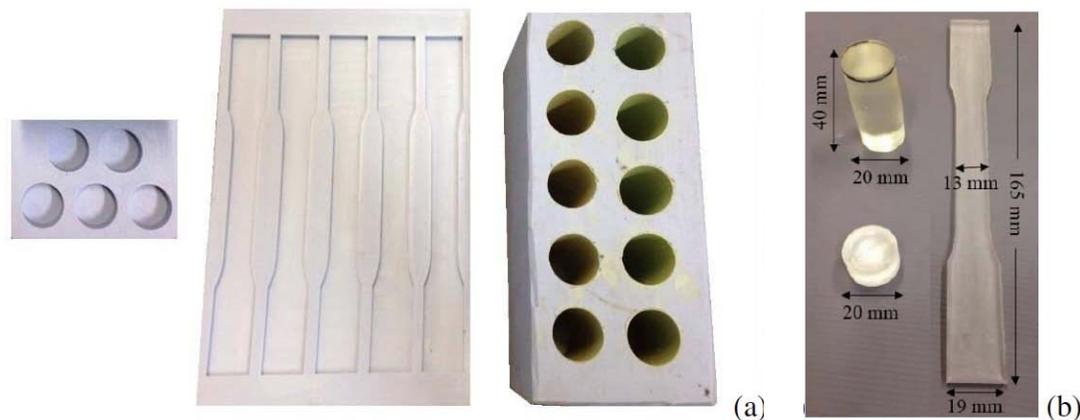


Figure 1: (a) Silicon mould and (b) samples for moisture absorption, tensile and compressive tests.

Table 1: Parameter of the Ageing Process

Environmental Condition	Temperature and relative humidity	Sodium Chloride (NaCl)	Standard	Ageing Time (days)
Natural	$\sim 22^\circ\text{C}$, 55%	-	-	7
100% RH	40°C , 100%	-	ASTM D2247-15 [20]	7
Salt spray	35°C , 100%	5 wt. %	ASTM B117-19 [21]	7

different harsh environments: (i) water saturation and (ii) sodium chloride solution. After seven days of ageing, all samples are dried in a blow drier and subjected to physical and mechanical tests.

2.4. Characterisation

Polymers are characterised by moisture absorption, tensile and compressive tests. Moisture absorption is determined only for samples in a harsh environment, since room temperature polymers absorb insignificant moisture. The relative moisture uptake (M) is calculated using the Archimedes principle and the ASTM D5229-20 [22] recommendations, as shown in Eq. 1.

$$M = \frac{w - w_o}{w_o} \times 100\% \quad (1)$$

where W_o is the weight of the dry specimen and W is the weight of the wet specimen after seven days in the chamber.

Tensile and compressive tests are conducted for all three environmental conditions following ASTM D638-

14 [18] and ASTM D695-15 [19], respectively. Prior to the characterisation, cylindrical samples are turned on a lathe to ensure the perfect parallelism required in the compression test. Both experiments are conducted on a universal test machine Shimadzu AG-X Plus, equipped with 100 kN load cell at 2 mm/min, as shown in Figure 2. The tensile tests are performed using a video-extensometer. The investigated responses are tensile and compressive strength and modulus.

2.5. Statistical Design

A full factorial design ($2^1 3^1$) is used to identify the effects of the type of polymer (epoxy and polyester) and the environmental condition (natural, 100% RH and salt spray-100% RH) on moisture absorption, compressive and tensile properties of the polymers, resulting in six experimental conditions (ECs), as shown in Table 2. Ten specimens are fabricated for each EC in two different replicates (five for each) to estimate the experimental error, resulting in 60 specimens for each test (moisture absorption, tensile and compressive test), totalling 180 specimens. The techniques of Design of Experiment (DoE) and

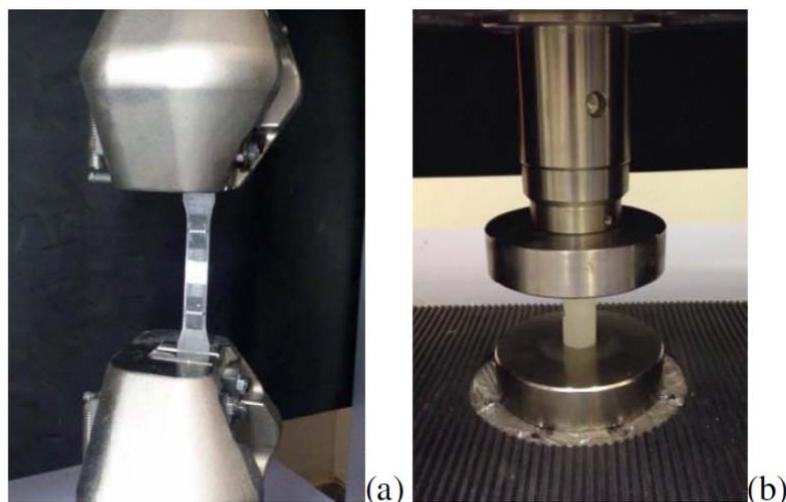


Figure 2: (a) Tensile and (b) compressive tests.

Table 2: Full Factorial Design ($2^1 3^1$)

EC	Polymer Type	Environmental Condition
1	Epoxy	Natural
2	Epoxy	100% RH
3	Epoxy	Salt spray 100% RH
4	Polyester	Natural
5	Polyester	100% RH
6	Polyester	Salt spray 100% RH

Analysis of Variance (ANOVA) are performed in Minitab® v. 18.

3. RESULTS

3.1. Experimental Data

Table 3 shows the mean values and standard deviation of DoE responses for each replicate. In general, polyester polymers have less moisture absorption and superior mechanical properties, except for tensile strength. These results are in accordance with the literature [23-25], in which the mechanical properties increase with the decrease of the water content. The data will be better assessed in the statistical design described in the following section.

3.2. Statistical Design

Table 4 presents the DoE/ANOVA analysis of the responses, exhibiting statistically significant effects (in bold) for the main and interaction factors within a 95%

confidence level, i.e., the P-values must be less than or equal to 0.05 ($P\text{-values} \leq 0.05$). Higher-order effects (underlined in Table 4) are interpreted using the effect plots (Figures 3-7) obtained from Minitab®. The $R^2\text{-adj}$ ranges from 89.24% to 99.77%, indicating models of high predictability for approaching 100%. ANOVA is validated by the Anderson-darling normality test; in this case, a normal distribution is indicated when the P-values are greater than 0.05 (0.088 – 0.954, Table 4).

3.2.1. Moisture Absorption

Moisture absorption ranges from 0.21% (polyester, salt spray) to 0.49% (epoxy, 100%RH). Figure 3 shows the second-order interaction effect plot for the mean of moisture absorption. The letters in the figure refer to the Tukey's comparison test (at 95% confidence interval), in which equivalent means are represented by equal letters. Polyester polymers result in less moisture absorption after both ageing processes when compared to epoxy, revealing reductions of up to 38%

Table 3: DoE Response Data for Replicates 1 and 2

	EC	Moisture Absorption [%]	Tensile Strength [MPa]	Tensile Modulus [GPa]	Compressive Strength [MPa]	Compressive Modulus [GPa]
Replicate 1	1	-	40.11 (± 5.92)	3.21 (± 0.01)	112.78 (± 2.12)	3.05 (± 0.13)
	2	0.49 (± 0.02)	48.32 (± 6.35)	3.26 (± 0.41)	110.34 (± 0.55)	2.94 (± 0.05)
	3	0.34 (± 0.01)	54.97 (± 7.04)	3.13 (± 0.13)	112.35 (± 1.45)	3.05 (± 0.08)
	4	-	22.55 (± 2.26)	3.71 (± 0.27)	115.17 (± 4.83)	3.04 (± 0.09)
	5	0.30 (± 0.01)	25.03 (± 3.60)	2.97 (± 0.06)	130.23 (± 1.94)	3.17 (± 0.05)
	6	0.22 (± 0.01)	21.43 (± 3.68)	3.06 (± 0.22)	128.43 (± 4.34)	3.15 (± 0.09)
Replicate 2	1	-	45.21 (± 6.79)	3.13 (± 0.12)	115.75 (± 4.84)	3.09 (± 0.04)
	2	0.48 (± 0.01)	43.03 (± 6.69)	3.02 (± 0.23)	109.79 (± 0.25)	2.96 (± 0.001)
	3	0.34 (± 0.01)	54.63 (± 5.96)	3.18 (± 0.16)	112.19 (± 0.34)	3.05 (± 0.03)
	4	-	22.63 (± 1.29)	3.91 (± 0.25)	118.11 (± 4.78)	3.09 (± 0.17)
	5	0.30 (± 0.01)	20.58 (± 4.61)	2.87 (± 0.24)	124.56 (± 4.44)	3.12 (± 0.06)
	6	0.21 (± 0.01)	23.11 (± 2.86)	3.10 (± 0.27)	127.01 (± 2.87)	3.15 (± 0.06)

Table 4: Analysis of Variance (ANOVA) for Polymer Samples

P-value ≤ 0.05					
Experimental Factors	Moisture Absorption	Tensile Strength	Tensile Modulus	Compressive Strength	Compressive Modulus
Polymer Type (PT)	0.000	0.000	0.090	0.000	0.000
Environmental Condition (EC)	0.000	0.039	0.001	0.051	0.033
PT x EC	0.001	0.030	0.002	0.004	0.002
$R^2\text{-adj}$	99.77%	96.64%	89.24%	92.24%	90.76%
Anderson Darling (P-value ≥ 0.05)	0.088	0.373	0.954	0.937	0.832

(when considered 100% RH). In addition, both types of polymers in the salt spray condition have less moisture absorption when compared to 100% RH, revealing reductions of up to 30% (when epoxy polymer is considered). Similar results are observed by [26-28]. According to Tan *et al.* [28], the decrease in moisture absorption in a saline environment is attributed to the change in driving force, since the chemical potential of water is decreased in a saline solution. This phenomenon generates an osmotic pressure that inhibits the water uptake by the polymer.

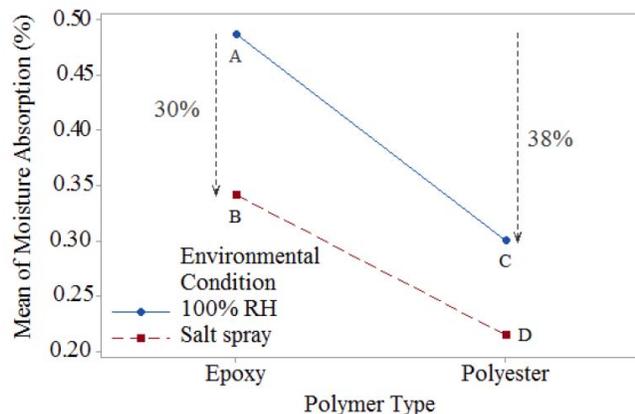


Figure 3: Second-order interaction effect plot for the mean (average) moisture absorption.

3.2.2. Tensile Properties

Tensile strength data range from 21.43 MPa (polyester, salt spray) to 54.97 MPa (epoxy, salt spray). Figure 4 shows the second-order interaction effect plot for the mean tensile strength, in which the epoxy polymer reaches higher values compared to the polyester, revealing increases of up to 146%. Moreover, samples under salt spray show strength 20% higher than other environmental conditions, as shown by Group A. An opposite behaviour in relation to tensile strength is found in the literature [23-25], revealing a recovery with increased content of water. According to several authors [27, 29-32], two types of bound water are found in these polymers, depending on the difference in the bond complex and activation energy. Zhou and Lucas [29, 30] classify it as type I and type II bonding, in which type I corresponds to a water molecule that forms a single hydrogen bond with the resin network, while type II is the result of a water molecule forming multiple hydrogen bonds with the resin network creating a secondary crosslink network. In type I, the water molecules disrupt Van der Waals interchain forces and hydrogen bonds, resulting in greater mobility of the chain segment and decreased T_g (glass transition temperature, i.e., the temperature

beyond which the adhesive changes from a rigid and strong material to one exhibiting high levels of flexibility and low mechanical performance); thus, the polymer acts as a plasticizer [33]. On the other side, in type II there is an increase in T_g and mechanical properties due to the additional cross-linking effect [27]. According to Guen-Geffroy [34], type I bonding is observed mainly in amine-based epoxy materials due to a large amount of absorbed water, as shown in Figure 3. However, an opposite behaviour is shown for the epoxy polymer under tensile strength, which is more dominated by type II effect; thus, exhibiting an increase in response. The tensile strength of polyester polymers is not affected by the environmental condition, revealing similar results, as revealed by Group C.

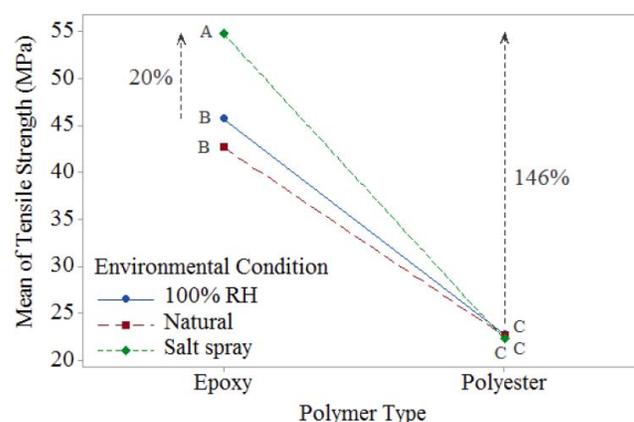


Figure 4: Second-order interaction effect plot for the mean (average) tensile strength.

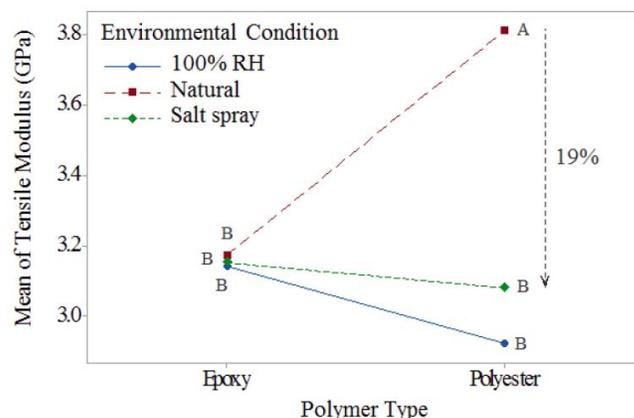


Figure 5: Second-order interaction effect plot for the mean (average) tensile modulus.

The tensile moduli of the polymers vary from 2.87 GPa (polyester, 100% RH) to 3.91 GPa (polyester, natural). Figure 5 shows the second-order interaction effect plot for the mean tensile modulus. In contrast to the strength behaviour (Figure 4), epoxy polymers have similar stiffness (Group B) in all experimental

conditions, while polyester polymers achieve a 19% reduction under ageing conditions, as shown by Group B. This behaviour can be attributed to the negative effect of plasticization (type I bonding), reducing the tensile modulus of polyester polymers.

3.2.3. Compressive Properties

The compressive strength values range from 109.79 MPa (epoxy, 100% RH) to 130.23 MPa (polyester, 100% RH). Figure 6 shows the second-order interaction effect plot for the mean compressive strength. The results of epoxy are similar to those of polyester in a natural environment, as shown by the same Group B. Although the epoxy polymer presents similar behaviour in compressive strength for all environmental conditions, as revealed by Group B, there is a tendency to reduce this response under a harsh environment, which is attributed to the plasticization effect of the epoxy (type I bonding), especially under high temperature and moisture content, as reported by Ashcroft *et al.* [33]. On the other hand, the compressive strength of polyester polymers is 16% higher than of epoxy under the same ageing condition. Moreover, a 10% increase is observed for polyester polymers after ageing in a harsh environment. This fact is attributed to the positive effect of the additional crosslinking (type II bonding), which is activated by raising the temperature through adequate post-cure [27].

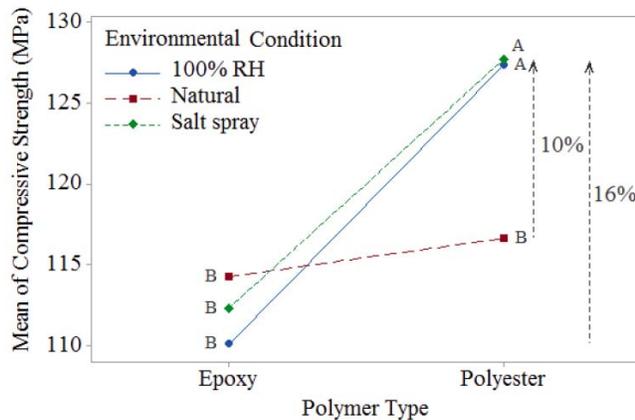


Figure 6: Second-order interaction effect plot for the mean (average) compressive strength.

The compressive modulus of elasticity ranges from 2.94 GPa (epoxy, 100% RH) to 3.17 GPa (polyester, 100% RH). Figure 7 shows the second-order interaction effect plot for the compressive modulus. A similar trend to compressive strength is observed for this response, showing increases of 3% and 7% for the polyester polymer in hostile environments and when

compared to epoxy polymer under the same environmental condition, respectively, attributed to the crosslinking effect (type II bonding). On the other hand, a decrease of 3% is observed for the epoxy polymer under 100% RH, attributed to the plasticization effect (type I bonding).

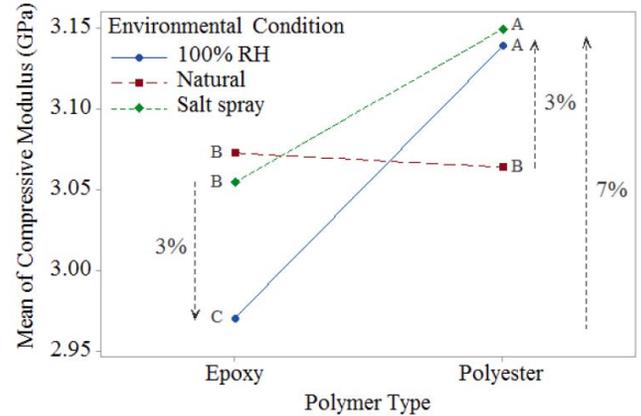


Figure 7: Second-order interaction effect plot for the mean (average) compressive modulus.

These findings reveal that the crosslinking effect (type I or type II), obtained by water saturation, is affected not only by the type of polymer, but also by the loading directions (tension or compression). In the elastic regime, the polyester polymer reduces its stiffness under tension (type I) (Figure 5), but also increases under compression (type II) (Figure 7). Meanwhile, the epoxy polymer does not change under tension (Figure 5), but reduces its compressive modulus (Figure 7), implying the presence of type I crosslinking. There was a correlation effect between the plastic and elastic regimes only under compressive efforts (Figures 6-7), when the epoxy and polyester polymers are dominated by type I and II, respectively. While the 100% RH level leads to less compressive strength and modulus of epoxy polymers, the level of salt-spray leads to higher compressive properties of polyester polymers (Figures 6-7).

4. CONCLUSIONS

The present work investigated, through a statistical design, the effect of three different environmental conditions (natural, 100% RH and salt spray) on moisture absorption, tensile and compressive properties of epoxy and polyester polymers in neat condition. The main conclusions are:

- The interaction between the environmental condition and the type of polymer significantly affects all responses;

- In general, polyester polymers have less moisture absorption and superior mechanical properties compared to epoxy, except for tensile strength;
- Both polymers reveal a decrease in moisture absorption in the saline environment compared to the completely saturated condition (100% RH);
- Polyester polymers in harsh environment exhibit higher compressive properties compared to those subjected to natural conditioning;
- In compression, the epoxy polymer is more affected by the negative effect of plasticization (type I bonding), while polyester is more affected by the positive effect of additional crosslinking.
- The findings indicate that the crosslinking effect (type I or type II) is also affected by the loading directions.

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