## Water in Epoxy Coatings

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The absorption of water strongly affects the exploitation characteristics of epoxy coatings and their lifecycles. The models used for describing water absorption to a polymer matrix involve many parameters that can adequately describe the process with parameter tuning. However, when using multiple variable parameters, the physical essence of absorption can be obscured. Hence, one of the promising directions in this field is the development of new models with fixed physically transparent parameters, which will adequately describe the water uptake process in epoxy resins.

Keywords: epoxy coatings ; water uptake ; kinetic study ; water state ; coating performance ; life cycle

### 1. Introduction

Polymer coatings are widely used as the most accessible tool to protect metal and wood. Water on Earth ensures the existence of life on Earth; on the other hand, it is one of the main sources that destroy objects produced by humankind. The infusion of water stimulates corrosion processes in the materials underlying protective coatings. In polar regions, it can result in the destruction of coatings and coated materials as a result of multiple freeze–thaw cycles.

Here, water is considered a complex liquid. The polarity of its molecules in combination with the possibility of a hydrogen bond network being formed results in a multilevel structure of liquid water. Such structures may persist during the infusion of water into the polymer matrix, which influences the diffusion processes and the infiltrate in the polymer bulk. Hence, the study of water infusion in coatings requires a multimethod approach using modeling and spectroscopic structural analysis.

Epoxy resins, with their network structure and excellent mechanical performance, high barrier properties, and thermal and chemical stability, are widely used as coatings and matrices for composite materials. Their high rigidity leads to their fragility and void formation during their preparation, which largely affects their water permeation.

The aim of most studies in this field is to examine the water permeation of common epoxy-based coatings across a range of commercially relevant temperatures (from room temperature to +80 °C). Water absorption can induce damage to the epoxy network, induce distinctive cavity formation in the coating films, and increase the aggregative porosity of the coatings. Hence, the analysis of the processes of water uptake is crucial for protecting transport pipelines, tanks, industrial constructions, and other objects. Vapor transport analysis in a broad range of temperatures from deep freezing to high sunshine is important for epoxy composites used in the aircraft industry because any damage to an aircraft is crucial. Other important engineering plastics are polyamides 6 and 6.6, which are widely used as materials for bearings, shafts, and other construction elements in the ship-building industry. The high uptake of water by these plastics and its influence on the performance characteristics of products is a topic that is not directly related to this discussion and would be the subject of another research. However, the main methods of studying water absorption appear to be common for all plastics and are referred to in the papers discussed.

The study of water uptake to epoxies has a long history <sup>[1]</sup>. The performance of epoxy coatings during moisture absorption has been described to decrease as a result of swelling <sup>[2]</sup>, the fluctuation of bulk mechanical properties <sup>[3]</sup>, the plasticization effect <sup>[4]</sup>, the chemical destruction of the epoxy matrix <sup>[5]</sup>, and other factors. In this study, these changes are considered to involve both experimental <sup>[6]</sup> and molecular simulation <sup>[7]</sup> methods. The complexity of this task requires new insights to advance the molecular-level understanding of the water interaction with the epoxy matrix and justifies the need for new strategies, including 2D IR spectroscopy <sup>[8]</sup> and solid-state NMR using multi-quantum filter techniques <sup>[9]</sup>. Methods based on electrochemical noise measurement have been shown to be promising as reliable quantitative indicators of corrosion <sup>[10][11][12][13]</sup>.

#### 2. Influence of Water on Glass Transition Temperatures of Epoxy Coatings

The glass transition temperature ( $T_g$ ) is a crucial parameter for epoxy coatings as it strongly affects the material's mechanical, thermal, electrical, and chemical properties. Understanding and controlling the  $T_g$  is essential for achieving desirable coating performance and durability. The influence of water on the glass transition temperature of epoxy coatings is one of the most important considerations for designing and formulating coatings for various applications. Understanding the plasticization effect of water can help engineers and coating manufacturers optimize formulations to ensure long-lasting performance, even in water-exposed environments. Effective mitigation strategies produce highly durable and protective epoxy coatings for a wide range of applications.

When water diffuses into an epoxy coating, this can lead to changes in the  $T_g$  of the epoxy. Water molecules can affect the mobility of polymer chains. Water molecules can act as plasticizers (so that polymer chains can move more easily) and reduce the  $T_g$  of the epoxy coating. As water diffuses into the coating, it disrupts the polymer chains, increasing their mobility. This increased mobility lowers the  $T_g$  and can result in a softer and more flexible material. Usually, the decrease in  $T_g$  due to the plasticizing effect ranges from 10 to 20 °C for every percent of water molecules in the epoxy material [6][14] [15][16][17][18].

Water diffusion highly depends on the polymer structure, crosslink density, environmental conditions (including temperature, pressure, humidity, and presence of salts), steric effect, polymer–penetrant interaction, and thermal and mechanical history. Since the relaxation phenomena also have significant effects on the changes in the nature of absorption, Fickian diffusion predominates in an amorphous polymer at temperatures much higher than  $T_g$ , while non-Fickian behavior regularly occurs in polymers at the glass transition temperature and below. This is explained by the relaxation of the polymer, which is essential for penetrant molecules to diffuse into the molecular network. This rearrangement results in a significant macroscopic swelling related to the diffusion of vapors and liquids into polymers <sup>[19]</sup>

When water penetrates into the epoxy matrix, there is a change in the effective crosslink density as a result of the interaction of the polymer and water molecules. This leads to an increase in the distribution of molecular weights and in the relaxation time. The effect of this process can be observed on thermograms when measuring the glass transition temperature using the DMA method. As the water content increases, the tan delta (*tan*  $\delta$ ) peak in the glass transition region broadens towards lower temperatures <sup>[22][23][24]</sup>. Often, the *tan*  $\delta$  peak can become separated into two peaks with low- and high-temperature maxima <sup>[5][6][23][25]</sup>. The high-temperature maximum corresponds to a less plasticized network, while the low-temperature one corresponds to a highly plasticized network. The tan  $\delta$  peak will change until the water saturation is reached. After that point, no evolution in the shape of the peak is observed. This is often explained by the plasticizing effect. Nogueria et al. <sup>[5]</sup> explained the observed phenomenon by the plasticizing effect and two additional factors: the free volume and the epoxy–water interactions. The formation of hydrogen bonds between water molecules and polymer chains requires breaking the interchain hydrogen bonds, which leads to an increased mobility of the chain in the glass transition region and a corresponding decrease in the effective crosslink density.

The extent of the  $T_g$  change depends on the concentration of water diffusing into the epoxy coating. Higher water concentrations typically result in more important reductions in  $T_g$ . The relationship between the water concentration and  $T_g$  change may not be linear, and different epoxy systems may exhibit varying sensitivity to water plasticization. Intramolecular interactions, post-cure, swelling, microcracking, physical aging, hydrolysis, and chain scission at higher temperatures also result in a different  $T_q$  <sup>[26][27][28][29][30]</sup>.

Understanding the effect of water uptake on  $T_g$  and subsequent chemical resistance is essential for selecting appropriate coatings for environments where exposure to chemicals is a concern. Prediction models for the glass transition temperature of epoxy coatings during water diffusion are based on various approaches, ranging from empirical equations to more complex theoretical models. The goal of these models is to estimate how  $T_g$  changes with the water content in the increasing epoxy matrix. Some of the most commonly used methods are the Fox, Kelley-Beuche (free volume model) and Simha–Boyer equations.

The Fox equation is an empirical model that relates the glass transition temperature of a polymer to the weight fractions of various components. The equation can be modified for epoxy–water systems to consider the effect of water (or other low-molecular-weight diluent) uptake [28][31][32]:

$$\frac{1}{T_g} = \frac{w_p}{T_{\rm gp}} + \frac{w_w}{T_{\rm gw}} \tag{1}$$

where  $T_{gp}$  and  $T_{gw}$  are the glass transition temperature of the polymer and water, respectively, and  $w_p$  and  $w_w$  are mass fractions of polymer and water, respectively. According to <sup>[33][34]</sup>, the experimentally obtained value of  $T_g$  of the water is in the range between 124 and 138 K.

The Fox equation is formulated based on the assumption of ideal volumetric additivity at  $T_g$ , and it neglects any specific interactions between the two components. Therefore, this model, in most cases, poorly describes the change in the glass transition temperature during water absorption as there are specific interactions between the water and the polar groups within the epoxy network.

The free volume theory (Kelley-Bueche equation) suggests that the glass transition is related to the available free volume in the polymer matrix. By considering the effect of water uptake on the free volume, the  $T_g$  changes can be estimated  $\frac{[35]}{[36][37]}$ .

$$T_g = \frac{\alpha_{\rm gp} V_p T_{\rm gp} + \alpha_{\rm lw} (1 - V_p) T_{\rm gw}}{\alpha_{\rm gp} V_p + \alpha_{\rm lw} (1 - V_p)}$$
(2)

where  $\alpha_{gp}$  is the difference in the coefficient of thermal expansion (CTE) of a polymer between the glassy and rubbery states;  $\alpha_{lw}$  is the CTE of water; and  $V_p$  is the volume fraction of the polymer. The value of  $V_p$  can be calculated using the following equation [38]:

$$V_p = rac{1}{1+0.01M_m\left(rac{
ho_p}{
ho_w}
ight)}$$
 (3)

where  $M_m$  is the equilibrium water content, and  $\rho_p$  and  $\rho_w$  are the densities of the polymer and water, respectively.

This approach requires knowledge of the epoxy's free volume properties and their variation with water uptake. All parameters required to calculate the glass transition temperature can be obtained from the literature (for example, the CTE and  $T_g$  of water are taken as 4 × 10<sup>-3</sup>/K and 277 K, respectively <sup>[37][38][39]</sup>) or experimentally (for example, the thermal expansion coefficients  $\alpha_{lw}$  and  $\alpha_{gp}$  are determined with a TMA experiment). This model has been used by many authors <sup>[25][36][37][38][39][40]</sup> and has proven to have good agreement with experimental data.

The Simha–Boyer equation is another empirical model that describes  $T_g$  changes with the water uptake. It is given in <sup>[41]</sup> as follows:

$$\frac{1}{T_g} = \frac{1}{T_{\rm gp}} + \left(\frac{1}{T_{\rm gw}} - \frac{1}{T_{\rm gp}}\right) V_w \tag{4}$$

where *V* is the volume fraction of water in the polymer. As mentioned above, the  $T_g$  of water is in the range between 124 and 138 K. It was also mentioned  $\frac{17}{18}$  that this method is able to adequately describe the decrease in the glass transition temperature as a result of the water penetrating into the polymer.

In addition to empirical and theoretical models, there are molecular simulation methods and techniques that can be used to predict the glass transition temperature during water uptake in the epoxy. Molecular dynamics (MD) simulations can provide insights into the behavior of polymers at the molecular level. By simulating the interactions between epoxy and water molecules, MD simulations can offer predictions of  $T_g$  changes during water uptake. However, MD simulations require detailed knowledge of the epoxy molecular structure and parameters, as well as ample computational resources [39][42].

It is important to note that each method has its own limitations and may not accurately capture the specific behavior of the epoxy system selected. Therefore, accurate predictions may require a combination of approaches along with an experimental validation. Additionally, a specific formulation, curing agents, and environmental conditions of the epoxy system can influence the  $T_q$  changes, and accounting for these factors would improve the accuracy of the predictions.

# **3.** The Effect of Water Absorption on Mechanical Properties of Epoxy Materials

Epoxy materials are the basis for preparing composites used in the production of aircrafts, wind, and tidal turbine blades <sup>[43]</sup>. They are in direct contact with atmospheric water (moisture), rain, or sea water. As a result, they undergo humidity damages during their operation. Composite materials were shown to absorb more water from the environment compared to common polymers <sup>[44]</sup>. This has a detrimental effect on the mechanical, electrical, and thermal properties of epoxy composites and reduces their service lives <sup>[45]</sup>.

A high moisture uptake can lead to obvious mechanical degradation and even severe failure and damage <sup>[46]</sup>. The inclusion of fillers was found to decrease the moisture absorption, increase the glass transition temperature, and slightly reduce the detrimental effect on the mechanical properties after hygrothermal conditioning <sup>[47]</sup>. However, no long-term durability of epoxy resins seems to have been comprehensively documented.

Water attacks epoxy-based composites at every level. The fibers, matrix, fiber–matrix interface, and adhesives are all susceptible to deterioration. Absorbed water can increase creep and relaxation, introduce residual stresses, cause osmotic pressure, and degrade the epoxy matrix, fillers, and filler/matrix interfaces via hydrolysis and chemical nucleophilic attack and acid or alkali catalysis. Water accelerates the fatigue degradation of composites and shortens their fatigue life. Additionally, fatigue damage offers new paths for moisture ingress and significantly increases the rate of moisture-related damage. However, the effect of fluids tends to be contradictory. For example, fluids result in improved impact resistance. This may be due to the impacting object being resisted by a larger volume of fibers. However, over time, impact damage may allow more routes for moisture to penetrate into the composite, and, thus, impair its properties over time <sup>[48]</sup>.

Moisture damage begins near the surface of the material and spreads inward over time, with cracks tending to grow parallel to the free surface. This damage is often localized, resulting in a small number of large cracks <sup>[49]</sup>. Crack growth is dominated by different effects depending on the level of loading. At lower load levels, cracking is most influenced by chemical reactions. At moderate load levels, cracking is most affected by diffusion. At higher load levels, stress-assisted corrosion controls crack growth <sup>[50]</sup>. The rate of mechanical performance degradation of epoxy-based composites has been observed to be directly correlated with the rate of moisture absorption <sup>[51]</sup>. Moisture is attracted to areas of air entrainment such as voids and delaminations. Thus, these areas can collect water over time <sup>[52]</sup>. Cracks and voids, even the microscopic ones, allow for easier penetration of water into the composite system via capillary action and diffusion. Therefore, a generous application of epoxy resin can potentially render moisture-related effects negligible. After an initial period of seeking out and filling cracks and voids, moisture begins to swell the composite. One study observed a linear relationship between strain and water uptake from the beginning of swelling <sup>[53]</sup>. Notably, water has been shown to diffuse more slowly through epoxy composites than through polyester composites, to give an example <sup>[54]</sup>.

Water uptake results in microcrack formation in the bulk of the epoxy and in the amplified voids, which tend to become linked together, generating paths, which promotes further penetration of water in the epoxy depth. The absorbed water may prove damaging to installed wraps. The tendency of composite materials to suffer damage when operating in harsh humidity environments shows that the humidity issue for epoxy-based composites should not be underestimated. It needs, instead, a rigorous and deep study to evaluate the impact on the system performance and assess the reduction in the operative life.

The moisture history of the composite is important. Both the maximum load at failure and the fracture toughness have been observed to decrease linearly as the time of moisture exposure increases. A higher average moisture content over the life of the material will result in a higher level of damage, and desorption appears to be more destructive than absorption. Additionally, wet–dry cycling has been observed to decrease the ultimate load of various epoxy-based composites. Typically, glass-fiber-reinforced composites suffer much more than carbon-fiber-reinforced composites.

Epoxy matrices are the primary victims of moisture absorption. If unchecked, moisture absorption by the resin will result in plasticization, swelling, hydrolysis, and fiber debonding from the matrix <sup>[55]</sup>. By plasticizing the polymer and lowering the glass transition temperature, moisture can soften a polymer and increase creep effects. This is typically reversible with drying. The hydrolysis of the epoxy resin, however, is irreversible, and can change both the stiffness and the strength of the polymer. Degradation is substantially slowed if the resin is fully cured prior to exposure <sup>[Z]</sup>. Additionally, water-resistant epoxies have been shown to increase the reliability of glass-fiber-reinforced epoxy composites by 200%–300%. Moisture absorption is known to encourage fibers to debond from the surrounding matrix. Matrix cracking allows further paths for moisture penetration, thus accelerating damage growth. Additionally, fluid ingress can result in swelling, which can cause

stresses between the fibers and the matrix and deteriorate the bond between them, resulting in tiny debondings. Epoxy resin swelling and osmotic pressure due to moisture uptake can cause debonding stresses between the fibers and resin.

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