Journal of Reinforced Plastics and Composites

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Journal of Reinforced Plastics and Composites 2002 21: 983
DOI: 10.1177/073168402128987644

The online version of this article can be found at:
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Hygrothermal Effects on RT-Cured Glass-Epoxy Composites in Immersion Environments. Part A: Moisture Absorption Characteristics

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ABSTRACT: A study on the effect of moisture absorption characteristics of RT-cure glass/epoxy composite and epoxy casting specimens immersed in freshly prepared artificial seawater and distilled water maintained at 333° K are presented in this paper. Results showed that the equilibrium moisture absorption value and the diffusion rate (slope of the moisture absorption curve) are higher in specimens immersed in distilled water than those of artificial seawater. The mixture rule for moisture absorption estimates has been verified for specimens immersed in both environments. These theoretically calculated values compared well with those experimentally obtained. The time of saturation for specimens immersed in seawater was found to be higher than that for those immersed in distilled water.

KEY WORDS: glass/epoxy composite, casting, distilled water, seawater, moisture gain, diffusion coefficient.

INTRODUCTION

THE NEED FOR the polymeric composites has been much felt in the offshore oil industry, submarine and ship building industry, as they offer the advantage of chemical stability and, by and large, are not susceptible to aqueous corrosion.

Innumerable studies on composites conditioned in moist environments were carried out on the rates and levels of absorption, and the consequent degradation in their mechanical properties. In the advanced composites, the fibres being essentially impermeable to moisture and the matrix being permeable in its cast form absorbs moisture often as much as 5% by weight [1] when subjected to severe

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0731-6844/02/01 0983–09 $10.00/0 DOI: 10.1106/073168402024289

hygrothermal factors. The effect of the fibres therefore essentially is to reduce the absorption levels with their increased weight fractions. Glass-epoxy composites are by far among the most researched class of materials in view of the balanced techno-economic benefits that they offer in a majority of applications.

As the hot-wet properties of any composite depend on the type of the matrix, fibre content and the interface, it is essential to know the rate of moisture ingress in these composites, in order to predict their long term behavior [2–7]. In many cases water absorption obeys Fickian law and diffusion is driven by the water concentration gradient between the environment and the exposed material leading to a continuous absorption process until saturation condition is reached.

Moisture absorption effects in polymeric composites are studied to assess the long term serviceability of these materials in aerospace and structural applications. Accordingly, material systems are quite often exposed to a set of varied temperature and humidity combinations rather than to immersion conditions. However, relatively fewer studies have addressed the studies in water immersion and still fewer in seawater environments at ambient or higher temperatures [8]. This paper, therefore, focuses on the moisture absorption behaviour of glass-epoxy composite materials and neat epoxy resin casting specimens immersed in artificial seawater and distilled water, which includes possibly one of the most hostile practical environments for composites in use.

EXPERIMENTAL DETAILS

Materials Used

Epoxy resin LY5052 and RT-cure hardener HY5052 [supplied by M/s Hindustan Ciba-Giegy Ltd., India] and BID glass fabric of 280 gsm [supplied by M/s Arun Fabric Ltd., India] were used.

Test Specimen Preparation

The laminates were fabricated by a wet-lay-up process followed by a vacuum bagging technique and room temperature curing. The resin hardener ratio was 100:40 parts by weight. In the above laminates a fibre weight fraction of 62.0% was maintained. Neat resin castings were also prepared using a two plate mould cavity set up with spacers all around.

Moisture absorption specimens (25 mm × 25 mm × 2 mm) were cut from the RT-cured laminates and castings. The edge coated composite as well as the cast specimens were step post cured at 50°C/1/2 hr, 70°C/1 hr followed by 85°C/2 hrs to ensure maximum glass transition temperature in the system. The artificial seawater was prepared according to ASTM D 1141 using the standard stock solution.

Environmental Conditioning

The post cured composite and casting specimens were weighed and their initial weights noted and were then immersed in baths containing freshly prepared artificial seawater and distilled water maintained at 333º K. The specimens were weighed at regular intervals of time until they reached saturation absorption level. The weight gain of the specimens were recorded as a function of time. The % moisture gain was reported according to the following expression

$$\%\text{Wt gain} = \frac{\text{Wt of specimen at any time} - \text{Wt of dry specimen}}{\text{Wt of dry specimen}}$$

Moisture absorption curves were obtained using the procedure of ASTM D570 for plastics. The % moisture gain values of each set of specimens were plotted against the square root of time in hours. Initially all the curves showed linearity, the slope being proportional to the diffusivity of the material.

The diffusion coefficients were calculated from the initial slope of the curve according to the equation:

$$D_c = \pi[h/4M_m]^2[(M_2 - M_1)/(\ t_2 - \ t_1)]^2 \quad (1)$$

where M_m is maximum moisture content, M_1 is moisture content at time t_1, M_2 is moisture content at time t_2 and h is thickness of the specimens.

RESULTS AND DISCUSSIONS

Determination of Diffusion Parameters (M_m, D_c, t_m)

The moisture absorption curves for composites and the casting specimens immersed in distilled water and artificial seawater are shown in Figure 1 and Figure 2 respectively. The nature of the curves indicates the Fickian behaviour in both the immersion media with an initial linear portion and thereafter remaining concave to time axis until maximum moisture content (M_m) is reached.

D_c values for test specimens were calculated using Equation (1). The D_c values obtained for composite test specimens are 7.88×10^{-7} mm^2/sec in distilled water and 6.0×10^{-7} mm^2/sec in artificial seawater, while for casting specimens the D_c values are 32.32×10^{-6} mm^2/sec in distilled water and 32.58×10^{-6} mm^2/sec in artificial seawater. For the same thickness and same test conditions, a small variation in the diffusivity values are noticeable. These results are summarised in Table 1.

The time of saturation for RT-cure glass-epoxy composite and epoxy casting specimens immersed in artificial seawater are found to be higher than those immersed in distilled water.

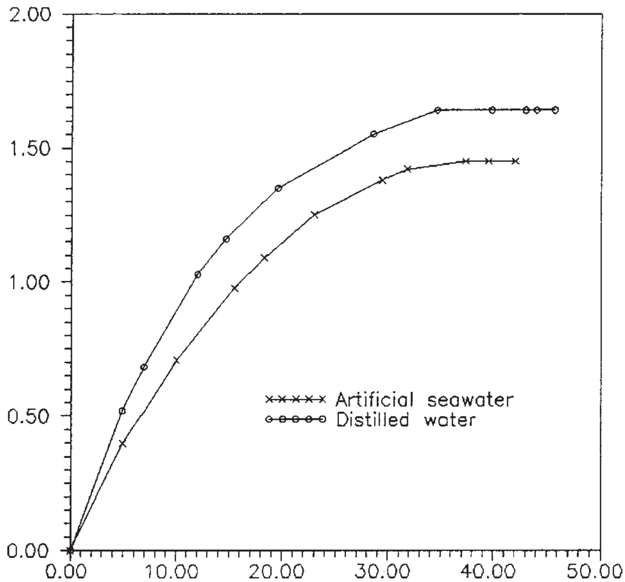

Figure 1. *Moisture absorption curve for glass/epoxy composite specimens in artificial seawater and distilled water immersed at 333° K. x-axis: sq. root of time in hrs and* y-axis: *% moisture gain.*

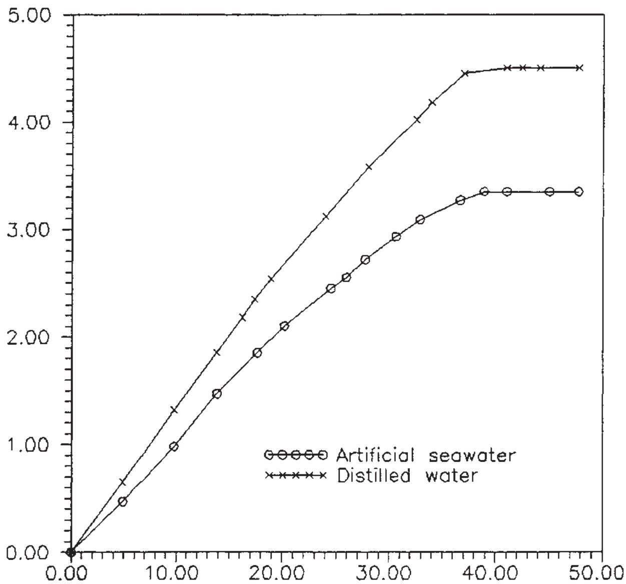

Figure 2. *Moisture absorption curve for epoxy casting specimens in artificial seawater and distilled water immersed at 333° K. x-axis: sq. root of time in hrs and* y-axis: *% moisture gain.*

Table 1. Comparison of diffusion parameters for epoxy casting and RT-cure glass/epoxy composite specimens immersed in artificial seawater and distilled water at 333 K.

Specimen Type	Distilled Water Immersion				Artificial Seawater Immersion			
	% M_m	Slope	D_c mm²/sec	t_m in Days	% M_m	Slope	D_c mm²/sec	t_m in Days
Casting	4.5	0.099	32.32×10^{-6}	57	3.35	0.074	32.58×10^{-6}	63
Composite	1.64	0.100	7.88×10^{-7}	48	1.41	0.075	6.00×10^{-7}	57

Fickian Correlation Curves

Figure 3 shows the correlation curve for composite specimens subjected to the distilled water and artificial seawater immersion. Figure 4 shows the correlation curve for casting specimens subjected to the distilled water and artificial seawater immersion. These figures indicate good correlation between the analytical and experimental values.

It is interesting to note here that for the composite case (Figure 3), the experi-

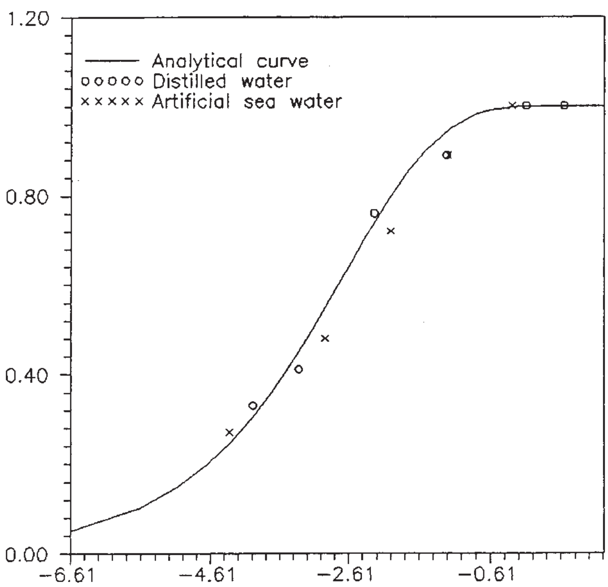

Figure 3. *Comparison of analytical and experimental values for glass/epoxy composite specimens in artificial seawater and distilled water immersed at 333° K. x-axis: ln*$(D_c t/h^2)$ *and y-axis:* $G = (M_t/M_m)$.

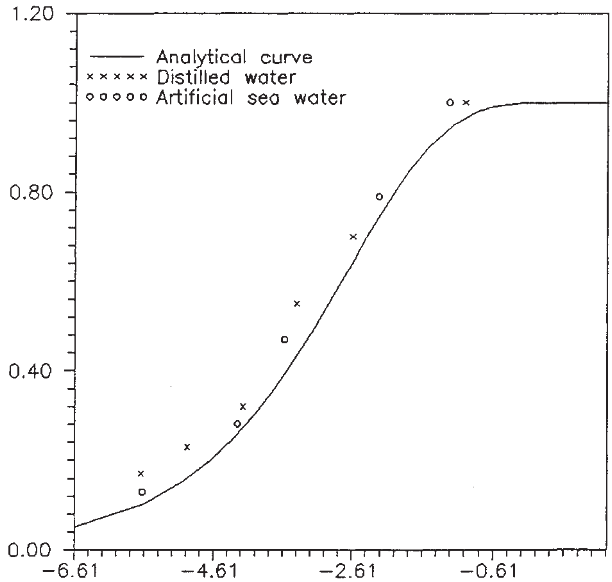

Figure 4. *Comparison of analytical and experimental values for epoxy casting specimens in artificial seawater and distilled water immersed at 333° K. x-axis: ln(*$D_c t/h^2$*) and y-axis:* G = (M_t/M_m).

mental data for both environments are distributed closely above and below the analytical curve, while for the castings (Figure 4) the experimental data fall close to, but above the analytical curve. Figure 5 and Figure 6 are conceived and drawn to quantify these deviations with respect to the analytical values (represented as a horizontal zero line for reference).

Verification of Mixture Rule for Moisture Absorption

The maximum moisture content of the composite (M_m) can also be computed from the experimentally obtained maximum moisture content of the neat resin casting $(M_m)_r$. The M_m and $(M_m)_r$ are related by the following expression representing the mixture rule

$$M_m = (M_m)_r (W_r) \qquad (2)$$

where W_r is the weight fraction of the resin in the composite.

Using the experimentally obtained data (Table 1), $(M_m)_r = 4.5$ in distilled water and $(M_m)_r = 3.35$ in artificial seawater and $W_r = 0.38$ for the above equation, the composite moisture contents "M_m" were found to be 1.71% in distilled water and

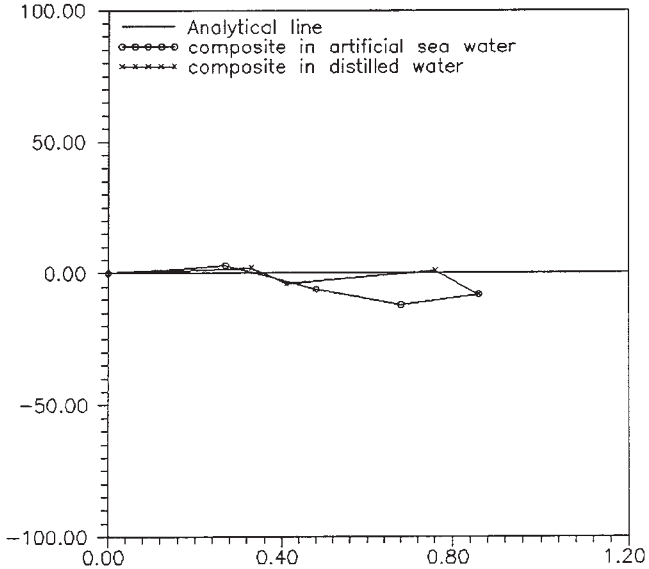

Figure 5. *Deviation of the experimental values from the analytical line for glass/epoxy composite specimens in artificial seawater and distilled water.* x-axis: G = (M_t/M_m) *and* y-axis: % *Deviation.*

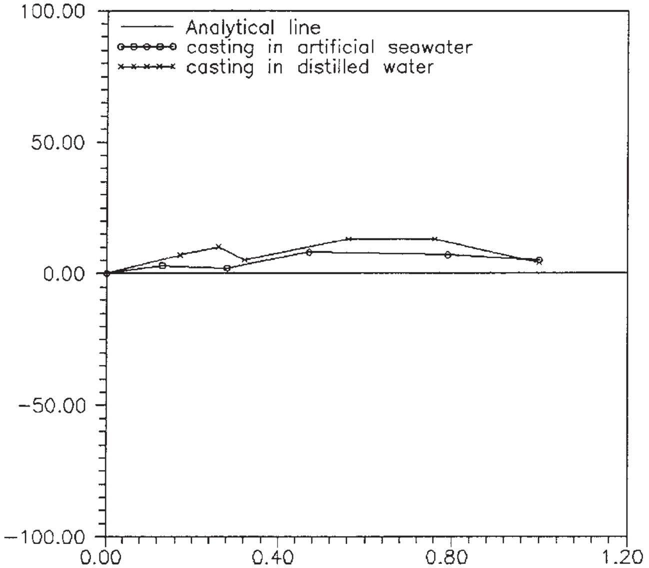

Figure 6. *Deviation of the experimental values from the analytical line for epoxy casting specimens in artificial seawater and distilled water.* x-axis: G = (M_t/M_m) *and* y-axis: % *Deviation.*

1.30% in artificial seawater immersion. These compared satisfactorily with the experimentally obtained value of 1.64% for distilled water and 1.41% for artificial seawater immersion environments as shown in Table 1. This shows that the deviations between the theoretically calculated and experimentally obtained "M_m" values are within 4–7% for both environments.

CONCLUSIONS

Under immersion conditions at 333° K, an RT-cured bi-directional glass/epoxy composite system exhibited saturation moisture level of 1.41% in artificial seawater and 1.64% in distilled water indicating higher absorption in the latter case. The diffusion rates (slopes) are seen to be higher for specimens immersed in distilled water than those immersed in artificial seawater, which can probably be due to the heavier NaCl molecules present in the artificial seawater media. The composite and the neat resin casting specimens immersed in distilled water and artificial seawater exhibited Fickian behaviour. The time of saturation for specimens immersed in seawater is higher than that for those immersed in distilled water, confirming an overall sluggish diffusion processes under seawater immersion conditions.

The mixture rule for moisture absorption estimates has been verified for both the immersion environments. The deviations between the experimentally obtained values are 4% lower than the theoretically calculated values for distilled water immersion and 7% higher than the theoretically calculated values for seawater immersion.

ACKNOWLEDGEMENTS

The authors are thankful to Dr. T. S. Prahlad, Director, NAL for all the support received during the investigations. They also acknowledge the assistance rendered by Mr. Rajendra Prakash in the preparation of test specimens.

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