EFFECT OF WATER AGING IN MECHANICAL PROPERTIES OF COMPOSITE MATERIAL FOR FILAMENT WINDING

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Abstract *The filament winding process is used for the production of pipes of composite materials. A study of the influence of water and temperature on the mechanical properties of these materials is necessary due to their exposition to environmental humidity in operation. In the present work, the mechanical properties of composite materials of polymeric matrix of vinyl ester resin with reinforcement in the form of fiberglass roving, developed for pipe production by filament winding technique were determined through tensile tests. The composites were submitted to a hygrothermal treatment in order to evaluate the influence of water and temperature on modulus of elasticity, tensile strain, tensile strength and toughness. Additionally, a microstructural and fractografic study was carried out aiming a comparative evaluation of the microstructure and fracture surface of the developed material before and after the aging process. The results show that aging caused a drop in tensile strength, tensile strain and led to a marked increase in modulus of elasticity of the material. This can be explained by the effect of post-cure caused by the action of the temperature. Microstructure and fracture surface of the composite showed no alteration with the aging process.*

Keywords*: Composite Material, Aging and Filament Winding*

1. Introduction

Filament winding has emerged as the primary process to manufacture composite cylinders at low cost [1]. Structures of this type are commonly subjected to complex loading conditions, which result from internal pressurization and superimposed axial loads during installation and/or operation of the cylindrical component [2]. It is well know that the performance of a filament wound object depends on such factors as adequate fiber and resin structures and volume fractions throughout the component, and controllable residual stresses after curing [3].

In order to utilize the full potential of composite materials, their performance during and after exposure to high temperature and high humidity environments must be known. For this reason, a great amount of attention has been paid in recent years to the problem of moisture absorption by composite materials [4]. Fiber-reinforced composites offer good resistance to environmental agents such as water and corrosive fluids. One of the obstacles preventing the extensive use of composites, however, has been a lack of long-term durability data [5].

As a common feature of composites, prominent anisotropy in mechanical properties is observed, which has high fracture strength and stiffness along the fiber strengthening component. Yet its potentials are not fully realized due to the moisture affecting long-term life of composite properties [6].

2. Experimental

2.1. Materials

Epoxy vinyl ester resin (Derakene 411-350, Dow Chemical Co) was used as composite matrix. The catalytic system was composed of methyl ethyl ketone peroxide (MEKP) initiator supplied as Butanox LPT (Akzo) activated by a solution of 6 wt% of cobalt octoate in dybutilphthalate supplied as NL51P(Akzo). E-fiberglass (1100 tex) roving (Owens Corning®) was used as reinforcement.

2.2. Laminate Preparation

Unidirectional fiber layers were prepared by winding the fiberglass using a manual device. The resulting layers were then laminated by hand-lay-up method, using vinyl ester resin as matrix, to produce the composites of the study with a nominal 58 vol % fiber content. The composition of the resin mixture was: 150 g of Derakane 411-350, 0.3 wt % Co and 1.5 wt % MEKP. The laminates were cured at room temperature exceeding 15 days before testing and no post cure treatment was applied.

2.3. Testing Procedures

Samples were cut from the laminate and tests were performed with longitudinal (unidirectional) and transverse specimens of nominal dimensions: length 250mm, width 25mm and thickness 2.5 mm. Some samples were subjected to a hygrothermal treatment at temperature of 60ºC for 36 days in distilled water. To study sorption behavior, mass changes for the specimens during aging were recorded at regular time intervals using an analytic balance.

Tensile tests were performed at room temperature for aged and not aged samples using an Instron mechanical testing machine (model TTDML) according to ASTM D3039M.

Optical microscopy was used to characterize composite microstructure and stereo microscopy was used to analyze fracture surface aspect, both before and after the aging process.

3. Results and Discussion

3.1. Microstructural Characterization

The microstructure of the composites show heterogeneity in the dispersion of the fibers, presenting regions of bigger concentration of fibers and others of lager amount of matrix between fibers. This behavior was observed for the composites with both tex of roving. The presence of porosities in the material is also observed, being these more present in the fiber-matrix interface regions.

Another important characteristic observed is the wettability of fibers by the matrix. The absence of wettability doesn't guarantee good properties for composite material causing premature failure of the material when mechanically loaded. Through microstructure photo the wettability in the composite material can be noticed. Optical microscopic images show longitudinal and transverse section of samples. Longitudinal section images for unaged materials show the fibers distributions all along its extension, Fig.1, while transverse sections images of unaged material show the sectionated fibers surface Fig. 2. Figures 3 and 4 show longitudinal and transverse sections for the material after aging. No modification was observed on the microscopy images after exposure to water.

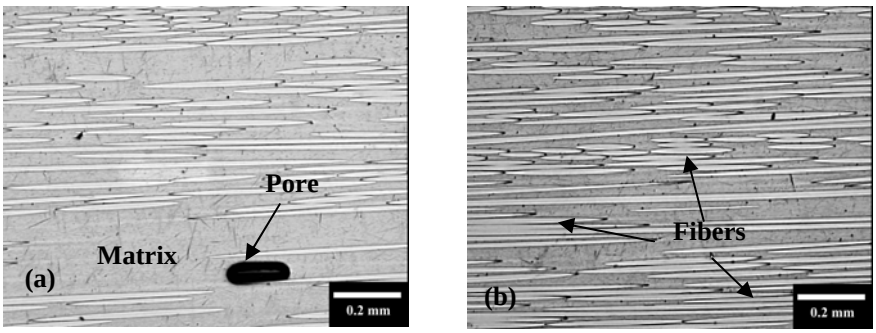

Figure 1. Longitudinal section of unaged material: (a) and (b) magnification: 50 times.

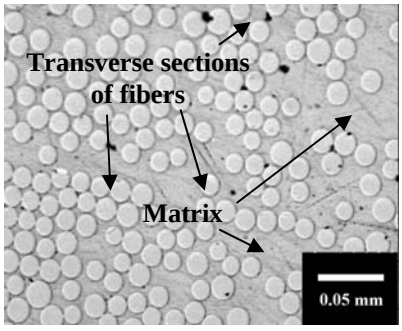

Figure 2. Transverse section of unaged material, magnification: 200 times.

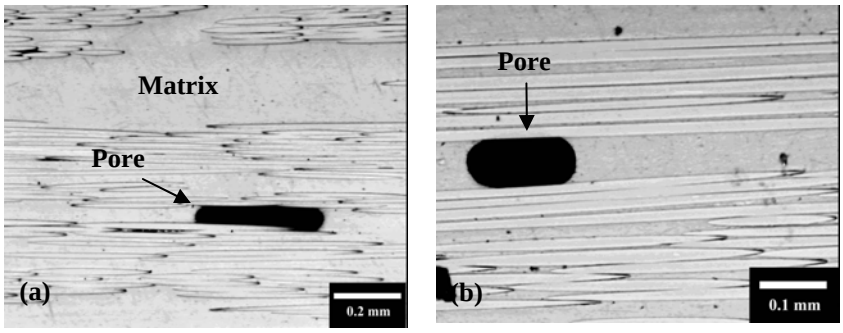

Figure 3. Longitudinal section of aged material: (a) magnification: 50 times (b) magnification: 100 times.

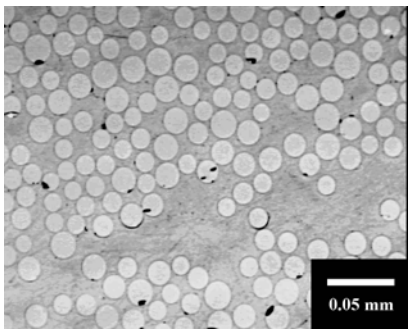

Figure 4. Transverse section of aged material, magnification: 200 times.

3.2 Mechanical Characterization

Figure 5 shows characteristic tensile curves of longitudinal samples of aged and unaged material and Fig. 6 shows the same for transverse samples. Table 1 and 2 show the corresponding average mechanical properties of aged and unaged materials, respectively. The properties evaluated were tensile modulus of elasticity (E), tensile strain (e) tensile strength (σ_R) and, toughness.

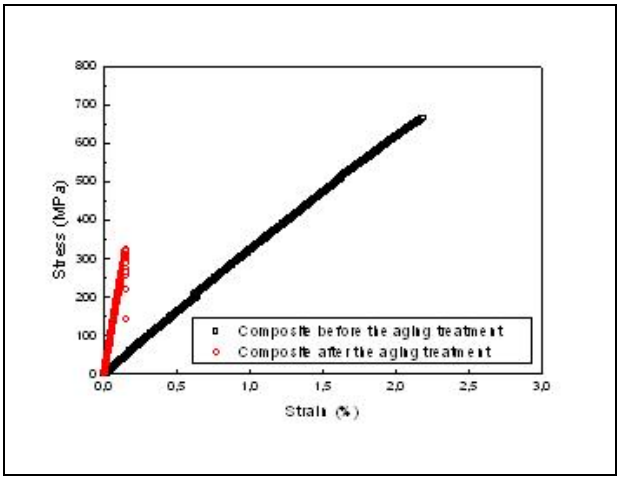

Figure 5. Stress-strain curves of aged and unaged materials. Longitudinal samples.

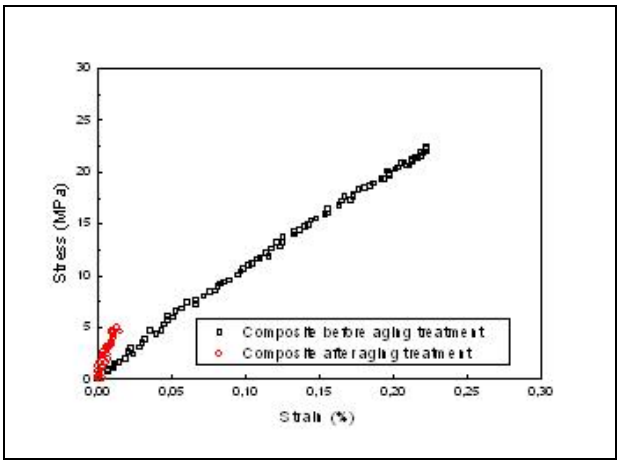

Figure 6. Stress-strain curves of aged and unaged materials. Transverse samples.

Table 1. Average mechanical properties of composite materials before the aging process.

Samples/ Standard Deviations	$\overline{E}$ [GPa]	$\overline{\sigma_R}$ [MPa]	$\overline{e}$ [%]	$\overline{Toughness}$ [J]
Longitudinal	31,20	629,81	2,09	21,58
Standard Deviation	0,76	79,22	0,33	6,52
Transverse	9,82	20,48	0,23	0,06
Standard Deviation	1,54	3,48	0,03	0,03

Table 2. Average mechanical properties of composite materials after the aging process.

Kind of Samples/Standard Deviations	$\overline{E}[GPa]$	$\overline{\sigma_R}[MPa]$	$\overline{e}[\%]$	$\overline{Toughness}$ [J]
Longitudinal	225,23	348,27	0,15	0,81
Standard Deviation	11,79	34,90	0,02	0,25
Transverse	27,40	4,63	0,013	0,003
Standard Deviation	4,77	1,97	0,005	0,002

The results show a marked fall of tensile strength and tensile strain and a considerable increase in modulus of elasticity of the material after the aging treatment, which is possibly related with post-cure of the resin, originated from the hygrothermal treatment of the material at 60ºC. It was noticed that room temperature wasn't sufficient to the complete cure of composite material. Then, post-cure take place during the aging treatment by the application of higher temperature.

3.3. Sorption Behavior

In terms of water absorption the composite material after aging for 36 days at 60ºC presented a behavior similar to the one presenting the Fick's law, reaching saturation after 14 days of immersion and presenting maximum value of 0,82% absorption. Figure 7 illustrates experimental behavior and behavior according to Fick Law, allowing the comparative analysis of the absorption process.
The absorbed percentage values were obtained using Eq. (1):

$$M = \frac{m_f - m_i}{m_i} x100\% \quad (1)$$

Where m_i and m_f are initial and final mass of samples before and after some time of aging , respectively.

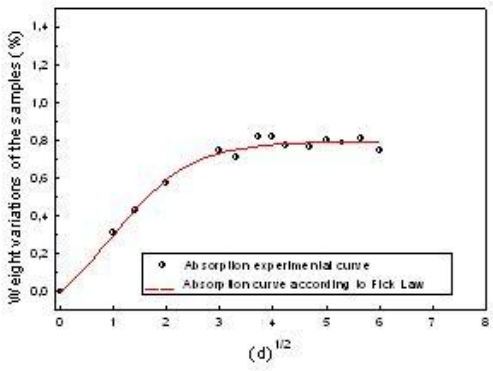

Figure 7. Weight change of specimens as a function of time of aging. Solid curve is theoretical.

Diffusion coefficient of water was calculated through two different models proposed in the literature. First model used is represented by Eq. (2) and second model by Eq. (3):

$$\frac{M_t}{M\infty} = 4\left(\frac{Dt}{\pi l^2}\right)^{1/2} \tag{2}$$

Where M_t is the amount of fluid that has been diffused in a period of time t, $M\infty$, is the value of fluid absorption in the beginning of the saturation, l is the thickness of the sample and D is the diffusion coefficient.

$$D = \frac{D_A}{x} \tag{3}$$

Where,

$$D_A = \pi\left|\frac{l^2}{16(M_\infty)^2}\right|\left|\frac{(M_2 - M_1)}{\sqrt{t_2} - \sqrt{t_1}}\right|^2$$

And

$$x = 1 + \frac{l}{L} + \frac{l}{W}$$

Where D_A is the apparent diffusion coefficient, x is the correlation geometric factor, $M\infty$ é is the equilibrium absorption value, M_1 e M_2 are the weight gain for the t_1 and t_2 times and L, W and *l* are length , width and thickness of the sample, respectively.

Results of diffusion coefficients are given in Tab. 3.

Table 3. Calculated values of diffusion coefficients.

	D (m^2/s)
Model 1	5,13 x 10^{-13}
Model 2	3,47 x 10^{-13}

It's difficult to discuss the mechanical behavior of the material as a function of the sorption curve. The problem is that at the same time that water is getting absorbed, low molecular weight segments leave the material making it difficult to correlate the mechanical property changes taking place in the material and the sorption curve.

Further work should be done studying the sorption behavior of fully post-cured material and the corresponding mechanical behavior.

3.4. Fracture Analysis

In case of longitudinal specimens, fracture of composite materials took place by delaminations of unidectional fibers. In case of transverse specimens, fracture occurred along the fiber's length in a linear form. For a better comprehension Figure 8 shows fracture regions of unaged and aged longitudinal specimens observed using the stereo microscope. It was observed that aging treatment cause no change in the fracture process of the material. Aged specimens exhibited similar fracture surface aspect to as unaged specimens.

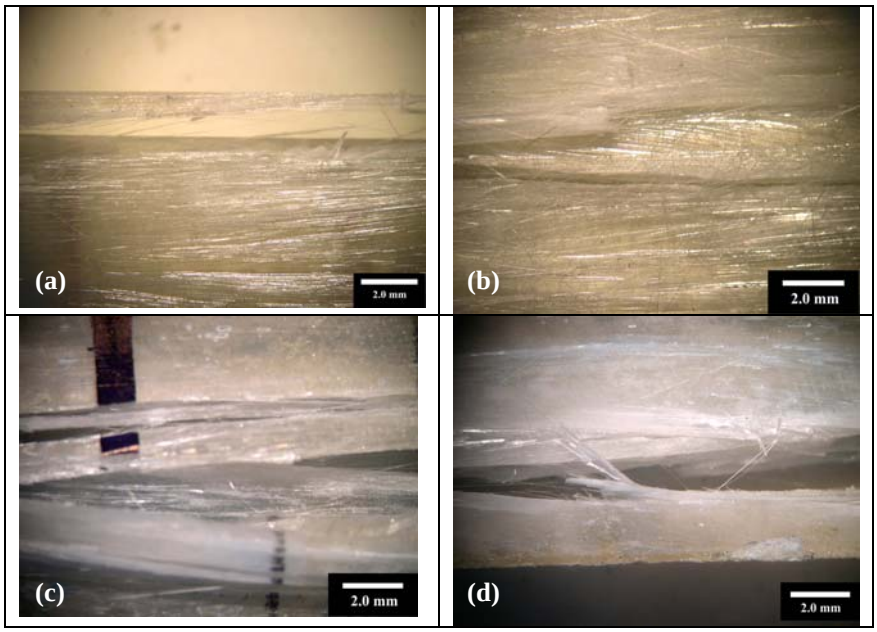

Figure 8. Fracture surface of longitudinal samples for: (a) and (b) unaged specimens; (c) and (d) aged specimens.

4. Conclusions

Unidirectional composite material developed show good mechanical properties in terms of tensile strength, tensile strain, modulus of elasticity and toughness. However , when submitted to hygrothermal treatment presented a marked fall in tensile strength, tensile strain and toughness and a considerable increase in modulus of elasticity due to effect of post-cure of the resin.

The absorption behavior of samples immersed in distilled water at 60ºC confirms Fickian behavior.

Optical images show that the microstructure of the material was not affected by aging process.

Images obtained by stereo microscopy shows no difference in the fracture surface aspect after the aging process.

5. Acknowledgements

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6. References

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