# Relation between the Epoxy Resin Curing and the Mechanical Properties of the Composite Reinforced with Aramid/Glass

Avaliação do Comportamento Mecânico e de Cura da Resina Epóxi em Compósito Reforçado com Aramida/Vidro

Evaluación del comportamiento mecánico y de curado de la resina epoxi en compuestos reforzados con aramida/vidrio

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### Abstract

DGEBA has a widespread use in the manufacture of polymer composites to obtain different elements of structural engineering. The mechanical properties can be predicted by the physical-chemical knowledge of the processing conditions of the resin components, and of their storage and handling conditions. This study aimed to characterize the functional groups, as well as the curing behavior of a batch of resin with different times of storage and storage control, using FTIR spectroscopy. The resin was used in the matrix of the composites with textile reinforcement, for which the mechanical behavior was examined through tensile and impact tests. It was noticed that the degradation effect in the polymer structure causes a reduction in the tensile strength and in the impact energy of the composite. **Keywords:** Curing cycle; Epoxy resin; Tensile; Impact; FTIR spectroscopy.

### Resumo

A resina epóxi DGEBA possui uma ampla utilização na fabricação de compósitos poliméricos, para obtenção de diversos elementos da engenharia estrutural. As propriedades mecânicas podem ser previstas pelo conhecimento físico-químico das condições de processamento dos componentes da resina, além de suas condições de acondicionamento e manuseio. Esse estudo teve como objetivo caracterizar os grupos funcionais, como também o comportamento de cura de um lote da resina com diferentes tempos de acondicionamento e controle de armazenamento, utilizando espectroscopia por FTIR. A resina foi utilizada na matriz dos compósitos com reforço têxtil, para os quais se analisou o comportamento mecânico através dos ensaios de impacto e tração. Percebeu-se que o efeito da degradação na estrutura do polímero leva a uma redução da resistência a tração e energia de impacto do compósito.

Palavras-chave: Ciclo de cura; Resina epóxi; Tração; Impacto; Espectroscopia FTIR.

### Resumen

La resina epoxi DGEBA es muy utilizada en la fabricación de composites poliméricos, para obtener diversos elementos de ingeniería estructural. Las propiedades mecánicas se pueden predecir mediante el conocimiento fisicoquímico de las condiciones de procesamiento de los componentes de resina, además de sus condiciones de embalaje y manipulación. Este estudio tuvo como objetivo caracterizar los grupos funcionales, así como el

comportamiento de curado de un lote de resina con diferentes tiempos de acondicionamiento y control de almacenamiento, mediante espectroscopia FTIR. La resina se utilizó en la matriz de composites con refuerzo textil, para lo cual se analizó el comportamiento mecánico mediante pruebas de impacto y tracción. Se observó que el efecto de la degradación sobre la estructura del polímero conduce a una reducción de la resistencia a la tracción y la energía de impacto del material compuesto.

Palabras clave: Ciclo de curado; Resina epóxi; Tracción; Impacto; Espectroscopía FTIR.

## **1. Introduction**

Large and complex structures made from fiber-reinforced polymers are used in numerous industrial applications (Cakić et al., 2012; Kondyurin et al., 2012; Meenakshi et al., 2012; Ramesh et al., 2013; Merad et al., 2007). For engineering structural applications, in which mechanical resistance and elasticity are critical properties, these composites are used because of their excellent physical and mechanical properties, especially their high specific strength, stiffness and density (Hardis et al., 2013). Hybrid composites contain more than one type of fiber in a single matrix. As a rule, several different types of fibers can be incorporated into a hybrid, but it is more likely that a combination of only two kinds of fibers might be more beneficial (Banerjee and Sankar, 2014). It is noteworthy, however, that the processing step of these composite materials is considered as a state-of-the-art technology, due to the characteristics of the products obtained, such as good mechanical properties associated with specific low density.

The epoxy resin is one of the most versatile of the thermorigid materials and it has good mechanical properties, chemical resistance and adhesive strength. Its versatility is due to the greater reactivity of the epoxy ring, since a considerable number of different chemicals may be used in the ring opening and subsequent resin reticulation (Menezes et al., 2013). Moreover, they become a desirable material for matrix in structural composites.

Several factors can affect the curing process and lead to different final properties of the epoxy. The knowledge on cure kinetics and changes in the physical state during curing is crucial for the control and optimization of the final properties of the matrix (Qi Li et al., 2012; Cheng Li et al., 2012). However, the mechanical performance of these epoxy resins depends on the handling and control of its storage environment. The control of these parameters associated with the resin curing process during the manufacture of composite materials will certainly lead to structures with more reliable limits of tensile, flexural, impact and torsion mechanical resistances.

The purpose of the current study is to characterize the components and DGEBA with different times and storage control, as well as the curing behavior at room temperature, through the qualitative analysis of absorption spectroscopy experiments in the Fourier Transform Infrared (FTIR) spectroscopy as a function of time. Furthermore, we intend to analyze the effect of the polymer matrix properties on the performance of the hybrid composite by mechanical tensile and impact tests.

Finally, we emphasize the importance of this study at the industrial level since it is necessary to know the resistance of the composite material as a function of storage time of the resin and of the varying control conditions for its use.

## 2. Methodology

#### 2.1 Materials and methods

This work is a laboratory study, according to Pereira (2018). The material used in the experiments was the epoxy resin, consisting of Araldite® GY BR 279 (liquid epoxy resin) and Aradur TM 2963 (hardener), both manufactured by Huntsman.

Structural polymer composites were obtained using hybrid textiles with aramid synthetic fibers (Kevlar 49) and S-glass, in a thermorigid matrix of epoxy resin (DGEBA- Diglycidyl Ether of Bisphenol A).

According to the manufacturer's information, Aradur TM 2963 must be stored in a dry environment, preferably in its original packaging at temperatures between 18-25 °C with a maximum storage time of 2 years. The Araldite GY 279 BR has a

flash point, according to DIN 51858 standard, at 180 °C and it is categorized in Fe IV B level of danger, and it must be stored at 18-25 °C, in a dry environment and, if possible, in the original sealed packaging with maximum storage time of 1 year.

In this work, the Araldite and Aradur reagents were used in and out the expiration date for use, and as a reaction product, obtained epoxy resin R1 and R2, respectively, which were used in the manufacture of composites.

#### 2.2 Reagents used

The epoxy resin (R1) was kept stored at the temperature of 22 °C, relative humidity of 45%, without the presence of light and in the date for use. On the other hand, the resin with a usage time of 12 months (R2) was stored at the temperature of 28 °C, relative humidity of 60%, without the presence of light and out the original manufacturer's date use. The material was processed from this resin in the two storage periods: 2 months and 12 months. The material collection occurred in both periods by withdrawing an amount of material through an appropriate container and immediate storage of the remnants in their respective packaging. The mixture of epoxy resins R1 and R2, was obtained in the ratio of parts by volume and it was mixed in a ratio of Araldite: Aradur (3:2), through the manual stirring process with a glass rod to obtain a homogeneous mixture (DGEBA), in an environment at the temperature of 19 °C and relative humidity of 45%.

#### 2.3 Absorption spectra in the infrared region

Infrared spectra were obtained according to Gonzales (2012) from the pure liquid of Aradur and Araldite with a storage time of 2 and 12 months. The spectrometer experiment was performed by using potassium bromide window - KBr (transparent compound in the vicinity of the ultraviolet with long infrared wavelengths in a range of 0.25-25  $\mu$ m, which does not have significant optical absorption lines in this transmission region, used for optical windows and prisms). On the other hand, the DGEBA R1 and R2 with a storage time of 2 and 12 months, after reaching its Gel point (55 min), was deposited on the surface of the KBr window and the experiment was similar components. From the Gel time, this KBr window with the resin deposited on its surface was left in a curing process within a desiccator. The absorption spectra were performed at cure time intervals of 1 h, 2 h, 3 h, 4 h, 5 h and 24 h. The FTIR spectra were obtained in an environment with a temperature of 19 °C and relative humidity of 45%, using the FTIR Spectrometer (VARIAN 640-IR) with a resolution of 4 cm<sup>-1</sup>.

#### 2.4 Production of the Composite Plate

The composites were produced by compression molding. For the design and manufacture of the reinforcing structural component, three layers of twill-type hybrid textile (Figure 1) were used, consisting of 65% Kevlar 49 (Warp yarn) and 35% S-glass fiber (Weft yarn), produced in handmade manufacturing process by handloom machine. Multifilament yarns of Kevlar 49 (110 Tex) and of S-glass fiber (75 Tex) were used. After fabrication, the textile was cut to size 200 x 150 mm to construct the reinforcing structure.



## Figure 1. Composition of the reinforcing textile.

Source: Authors.

Next, a thin layer of release agent was applied on the molding matrix and a quantity of resin was poured on the mold. In the next step, the three layers of textile and new layers of resin were deposited to, at a later stage, be closed and placed under 5 tons of pressure in a hydraulic press for 24 hours. After the period of compression, the composite plate was demolded in the dimensions of  $200 \times 150 \times 2$  mm (Figure 2).





Source: Authors.

The cutting of specimens for tensile and impact tests were performed after 30 days of the composite plates manufacture. The cut was made by waterjet cutting machine (model GA-Jetstream II from GA Water Jet Manufacturer) to avoid microcracks in the cutting surfaces.

# 3. Tensile and Impact Test

Five specimens were made for each storage time and for the tensile and impact tests. In all cases, the direction of Kevlar 49 fibers was coincident with the direction of the specimens' length, while the direction of the S-glass fibers maintained the width direction in the specimens.

To conduct the tensile test, the specimens were prepared according to ASTM 3039 standard, with the dimensions of 2 x 25 x 150 mm. For the impact test, the specimens were prepared according to ASTM D4812 standard, with the dimensions of 2 x 12.4 x 75 mm (Figures 3 and 4).





Source: Authors.



Figure 4. Specimens for impact test.

Source: Authors.

The tensile test was conducted at room temperature and test speed of 5 mm/min in the universal test machine (INSTRON 3367). The tensile results for mean and standard deviation were analyzed to verify significant differences between the groups with p < 0.05 significance level.

The elastic modulus was performed during the tensile test. Strain gauges (KYOVA KGF-10-120-C1-11) were placed in three tensile specimens obtained in the axial and perpendicular direction of Warp yarn (Kevlar 49). The attachment followed the standard procedure, i.e., the region of the surface of the specimens where the strain gages were attached was adequately prepared by sanding. The strain gage was attached on the central axis of the test specimen with appropriate adhesive (LOCTITE 496). The test procedure consisted of applying a load equivalent to 50% of the yield strength and getting the resulting deformation in a LINX data logger - model ADS 2000. The parameters for acquisition were <sup>1</sup>/<sub>4</sub> of bridge, 120  $\Omega$  and 60 Hz.

The impact test was conducted at room temperature in the impact machine (INSTRON model CEAST 9050). The average and standard deviation results from five specimens per test were used to determine the mechanical behavior of the analyzed materials by means of the energy absorbed due to the 5.5 Joules impact of a falling hammer.

## 4. Results and Discussion

## 4.1 Absorption spectroscopy in the infrared region

A combination of information about the chemical structures presents in organic compounds, and the behavior of functional groups during their polymerization reactions (Okoro, 2021; Lorwanishpaisarn, 2021; Coates, 2000) can be obtained by using the spectroscopy technique in the infrared region.

Figures 5 and 6 show the absorption spectra in the infrared region of Araldite, Aradur, R1 and R2 resins. The analysis of the spectra and their characteristic bands provided information on the attributes of functional groups existing in Aradur, Araldite and DGEBA (R1 and R2), and no marked differences is observed in the spectra.



Figure 5. FTIR spectra of Araldite, Aradur and DGEBA.

Source: Authors.



Figure 6. FTIR spectra of Araldite, Aradur and DGEBA.

Source: Authors.

We observed, in the absorption spectra of DGEBA (Figs. 7 and 8), the characteristic bands of the epoxy group and the decreased intensity of the band in the region near 880 cm<sup>-1</sup>, when the resin was in curing process. The Aradur hardener results from a mixture of epoxy resin and excess of polyamidoamine and it was possible to identify the appearance of the typical bands of the epoxy group in the 807 cm<sup>-1</sup> region. It was also observed, that the DGEBA shows a band at 3388 cm<sup>-1</sup> region attributed to the hydroxyl group, where the hydroxyl is a product derived from the chemical reaction of the amines in epoxy rings.

The Figure 7 shows the spectra of the epoxy resin (R1) at different curing times: 55 minutes (Gel Time), 1 h, 2 h, 3 h, 4 h, 5 h and 24 h. It is observed, by means of the spectra of Figure 7, that the band related to the epoxy ring shifted to a higher wavenumber, as well as the decrease in its intensity with respect to the curing time is probably due to the reactions of these rings with amines during the polymerization process. Figure 8 shows the resin (R2) spectra under the same curing times. The band referring to the epoxy ring has a decrease in its intensity with respect to the curing time, only after 24 hours of curing, showing that the curing process of this resin was slower and that it can influence the mechanical properties when compared with the 2-months-storage resin.

The absorption spectra of the epoxy resin cured after 24 hours with storage periods of 2 and 12 months have similar profiles, but the characteristic bands of the functional groups have different intensities. Probably, the low intensities in the resin spectra with 12 months of storage result from the loss of the unique characteristics and physicochemical properties of these products due to the storage conditions which are not in accordance with the manufacturer's guidelines. There is the possibility of loss of the chemicals' volatile components due to improper storage, which probably influenced the resin polymerization process due to a decrease on its viscosity which makes it difficult for the amines reach the epoxy, and hence the curing process.

After the analyses of the absorption spectra, it can be considered that during the manufacturing process of structural composites, the curing of the polymer matrix is one of the most important aspects to achieve effectiveness of the chemical interactions in the polymerization between the elements that comprise the resin.

The curing cycle used together with other process parameters such as time, temperature, pressure, and the sequence of combinations of these variables can certainly control the quality of the final product to be obtained. Therefore, for the curing process to occur, it is necessary that the resin epoxy groups react with stoichiometric quantities of curing agents (Mousa and Ghaemi, 2008; Block et al., 2013; Cheng Li et al., 2012; Tudorachi, 2020).

Figure 7. FTIR spectra of DGEBA, the epoxy resin (R1) at different curing times: 55 min (Gel Time), 1, 2, 3, 4, 5 and 24 h.



Source: Authors.





Source: Authors.

From the analysis of the methodology established for the investigation of DGEBA curing reactions using absorption spectroscopy in the infrared region, it is possible to say that this technique is effective because it provides direct information about the functional groups present in the precursors and in the cured epoxy resin; specially, because it has the capacity of monitoring the curing process of the reactions and because it is an effective method for the *in-situ* analysis of such material.

#### 4.2 Tensile Test

Table 1 shows the values of yield strength, tensile strength and elastic modulus of the composites that were made with the epoxy resin in both storage times of 2 and 12 months.

Composite	Yield Strength (MPa)	Tensile Strength (MPa)	Elastic Modulus (GPa)	Impact Energy (kJ/m <sup>2</sup> )
Epoxy Resin 2 months	$(68.80 \pm 6.36)$	$(112.90 \pm 3.47)$	$(9.45\pm0.25)$	$(91.78 \pm 3.15)$
Epoxy Resin 12 months	$(51.34 \pm 1.76)$	$(87.09 \pm 2.53)$	$(10.95 \pm 0.12)$	$(49.45 \pm 4.33)$
Source: Authors.				

Table 1. Values obtained in tensile and impact tests. The standard deviation values are shown in brackets.

It is observed that the composite which used the epoxy resin with a storage time of 12 months showed lower yield strength and tensile strength compared to the value of the 2-month-storage resin. The elastic modulus of the two composites was, respectively, 9.45 GPa and 10.95 GPa for the resin with 2- and 12-months storage. Although the values are closer, the resin R1 with 2 months shows lower elastic modulus, consequently, has lower stiffness.

The yield strength of the composites made from the resin with a storage time of 2 and 12 months was, respectively, 68.80 MPa and 51.34 MPa. The reduction in the yield strength of the composite with 12 months of storage was probably due to the loss of the physico-chemical characteristics of the precursors and to differences in the polymerization reactions in the curing process. This reduction in strength can be regarded with the shortening of the cross-links in the structure of the composite during the curing process of the resin with 12 months of storage; resulting in a polymeric structure with less ductile characteristic.

The deformation behavior of polymers is governed by their molecular structure, in which this behavior is quite complex, since it involves different phenomena related to different molecular mechanisms such as: viscous flow, elasticity, viscoelasticity and Hookean elasticity (Baig, 2020). As the direction of the fibers was identical in the tensile test of the resins composite specimens with a storage time of 2 and 12 months, these results suggest a correlation between the toughness of the epoxy matrix and the reinforcement, resulting in an elongation when the fibers are tensioned (Rezende et al., 2011; Neto and Pardini, 2006; Oliveira, 2020). The obtained data suggest that the 2-months-storage epoxy resin used in the composite matrix with reinforcement of Kevlar and glass performed well, improving the tensile mechanical proprieties.

#### 4.3 Impact Test

Table 1 also shows the values of impact of the composites made with both the epoxy resin with storage time of 2 and 12 months. An increase in the percentage of crystallinity and reduction of the average molar mass of the epoxy resin provides a reduction in impact strength (Cozza, 2020), as noted in Table 1.

The impact energy had a mean value respectively of 49.45 kJ/m<sup>2</sup> and 91.78 kJ/m<sup>2</sup> for 2 and 12 months of resin storage. This reduction of the impact energy with longer storage time of the resin can be associated with the shortening of the cross-links in the structure of the composite during the resin curing process, generating a polymeric structure with more brittle

characteristics, and therefore exhibiting a lower toughness in the polymeric matrix of the composite made with the 12-monthsstorage resin.

## 5. Conclusion

The absorption spectroscopy in the infrared region was an effective technique to compare the two storage times of the resin, because it provided direct information on the functional groups existing in the precursors and in the cured epoxy resin. The analyses indicate that, during the manufacturing process of the structural composites, the polymer matrix curing is one of the most important aspects to obtain effectiveness of chemical interactions in the polymerization reactions among the elements that constitute the resin.

The resin composite with shorter storage time showed the highest values of yield strength, tensile strength, and impact, which emphasizes the importance of the matrix properties with respect to the properties of the composite.

The results of this study emphasize the importance of the adequate handling and storage of compounds used to obtain epoxy resin, which makes it possible to obtain better mechanical properties of these composites with the purpose of industrial applications.

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