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ORIGINAL PAPER

Effect of hydration conditions in thermal and mechanical properties of injection-molded glass fiber/polyamide-6

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Abstract

Engineering polymers are used in a multitude of load-bearing applications where structural integrity and long-term reliability are of the utmost importance. In this study, two different commercial composites (Durethan and Technyl) of polyamide-6 reinforced with 30 wt% of glass fiber were manufactured by injection molding and different hydration conditions were tested. All composites were characterized chemical and mechanically (tensile, flexural, impact and toughness). The fractured surface morphology was analyzed microscopically. According to the infrared spectra, the chemical structures of the composites were not altered by the hydration conditions tested and there were no significant effects on the degree of crystallinity of the samples evaluated. The toughness and impact tests evidenced that the best hydration condition was an immediate immersion, followed by the one performed after 24 h the injection, where for the impact resistance test the Durethan composites was 27.84% higher than Technyl. Wet Pack hydration method, showed similar behavior with samples that were not hydrated, exhibiting low toughness and impact resistance. In general, Durethan composites hydrated by immediate immersion showed the best mechanical performance, being the most suitable to be used in the industry as a stretcher junction component.

Keywords Hydration conditions · Degree of crystallinity · Glass fiber–polyamide-6 · Wet Pack

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Introduction

Polyamide-6 (PA6) is semicrystalline polymers that provide an ideal combination of properties, especially for technical applications. In recent decades, it has been widely used in the field of load-bearing applications, such as mechanical parts often exposed to demanding conditions, combining high load, high temperature regimes and high relative humidity [1]. This polymer combines high mechanical strength and stiffness with good electrical insulation properties, high resistance to heat, wear resistance and the attenuation of noise and vibration [2]. However, the hydrophilic character of PA6 limits its applicability as an engineering plastic and the water sorption mechanism of polyamides is subject of interest for many researchers. [3, 4].

Water is a powerful plasticizer for PA6 that eventually, penetrates into the amorphous regions of the polymer, but also reaches semicrystalline areas. When water comes in contact with PA6, it breaks the H-bonds of the amide group, thereby increasing the mobility of the molecular chain which results in a decrease of the glass transition temperature value [5–7]. The water absorption causes the polymer to swell which causes an increase in the free volume in the matrix resulting in a decrease in the mechanical properties of the composites [8]. Therefore, it is extremely important to analyze the behavior of polyamides during different hydration environments.

During the injection process, the polyamide loses all the remaining water contained in its structure, causing the part to become rigid and fragile. Due to their hygroscopic characteristics, the parts produced will absorb moisture from the environment, changing their mechanical properties and dimensions, until they reach equilibrium with the environment, making them tougher [9]. This is a slow process that depends on the relative humidity, the ambient temperature and thickness of the part. Some methods have been developed to induce parts to equilibrium more quickly; these are known as accelerated adsorption methods and are performed before the parts are used. This is necessary for the parts suffering mechanical bending requests, impact, tensile and torsional because if used without proper moisture content can undergo plastic deformation or fracture compromising the integrity of the part [10].

An attempt to improve the polymer's mechanical behavior, in general, is to incorporate organic and inorganic fillers into the polymer matrix, which results in the formation of composites with highly improved properties even with the inclusion of low amounts of fillers [11–21]. In this sense, glass fiber reinforced PA6 composites are widely used in engineering, as they have improved physical and mechanical properties compared to simple polymers [22]. These composites can be processed by conventional methods, such as injection molding. This technique is widely used to produce polymeric products, and has the advantage of having fast production cycles, suitability for mass production and the versatility of product shapes [23].

Valentin et al. [5] evaluated effects of water absorption on tensile behavior of short glass fiber reinforced polyamide-6.6. Specimens were exposed to various relative humidity levels consisting of 30, 75 and 100%. Bending tests were performed on specimens with different water concentration levels. For water absorption above

1%, tensile strength and modulus were considerably reduced. Silva et al. [24] studied the water absorption and desorption processes of a commercial polyamide. We observed that the humidity level should not exceed the limit values, because if the injection molding process uses wet granules it leads to structural changes in the polymer and generates deterioration of the mechanical properties.

The diverse processing conditions used in the industry can affect from part design to mechanical performance. In general, the moisture content in polyamide is a key variable affecting processing (compounding, molding, welding) and end-use performances (mechanical, dimensional, surface appearance) [25]. Different hydration methods are used in the industry, such as Wet Pack and direct immersion in water. Although the effects of hydration on the mechanical properties of glass fiber reinforced polyamide-6 are widely known, an analysis of the literature revealed that there are not many studies that specifically evaluate the effect of hydration techniques used in industries.

In this sense, the SAS Plastic Ltd a Brazilian company produces a mechanical component called stretcher junction, which has the function of joining the tubes that make up the base with casters of a hospital stretcher. These pieces are composed of polyamide-6 with 30 wt% glass fiber and some of them, after a short time of use, started to present mechanical failures. In this context, this work aimed to investigate the mechanical performance of two composites that are used in the articulation of stretchers in hospitals (Technyl and Durethan), under the influence of industrial hydration different conditions.

Materials and methods

Materials

The samples used in this study are commercial in nature and were kindly donated by the Lanxess and Rhodia companies, known as Durethan BKV30 and Technyl C216V30, respectively. Both materials are composed of glass fiber reinforced polyamide-6 (30 wt% of short glass fibers) with a density of 1.36 g cm^{-3} and 1.37 g cm^{-3}, respectively. According to company's information, prior to the preparation of the composites, the fibers were surface treated with an aminosilane coupling agent to ensure their application as reinforcement. The type of glass fiber used was *E*-glass fiber (123D) with a length of 4 mm and a diameter of 10 µm ($L/D = 400$).

Injection molding process

As indicated by both suppliers, the composites were dry at 100 °C for 8 h to minimize air bubbles occurring during the injection molding and to avoid hydrolytic degradation of the composite at high temperatures. After dried process, these materials were injection-molded by using a plastic injection machine (HIMACO, LH150-80 model) at the conditions given in Table 1.

Table 1 Processing parameters used for the injection of the samples test

Parameters	Value
Mold temperature (°C)	80
Nozzle temperature (°C)	260
Injection time (s)	5
Injection pressure (bar)	1304
Injection speed ($cm^3 s^{-1}$)	65
Cooling time (s)	20

Hydration methods

Two hydration methods were tested: direct immersion in water and Wet Pack. Hydration by immersion was performed by completely immersing the specimens in distilled water at 85 °C for 4 h (period in which the samples reached their maximum hydration degree). In this method, newly injected specimens and specimens after 24 h of injection were tested. In the Wet Pack hydration method, specimens were placed in hermetic zip lock packs, with an amount of water equivalent to 15 wt% of the specimens, for 40 h at 23 °C. In this method, only specimens after 24 h of injection were tested. As a control group, Durethan BKV30 and Technyl C216V30 composites without previous hydration were evaluated. In Table 2, we present a summary of the different hydration conditions used in this study.

Attenuated total reflectance coupled to Fourier Transform Infrared spectroscopy (ATR/FTIR)

The Fourier transform infrared spectroscopy (FTIR) in attenuated total reflection (ATR) mode was applied using a Nicolet IS10 spectrometer. Films from each sample were tested using a resolution of 4 cm^{-1} and 32 scans, over a range of 400–4000 cm^{-1}.

Table 2 Information and coding of the samples evaluated in this study

Material	Hydration methods	Code
Technyl C216V30	Without hydration	TDry
Technyl C216V30	Wet Pack after 24 h	TWP24
Technyl C216V30	Hydration after 24 h	TWI24
Technyl C216V30	Immediate hydration	TWI0
Durethan BKV30	Without hydration	DDry
Durethan BKV30	Wet Pack after 24 h	DWP24
Durethan BKV30	Hydration after 24 h	DWI24
Durethan BKV30	Immediate hydration	DWI0

Differential scanning calorimetry

Differential scanning calorimetry (DSC) measurements were performed using a DSC 6000, Perkin Elmer. 10 mg of samples were subjected to two heating cycles from 25 °C to 280 °C and a cooling cycle from 280 °C to 25 °C, at a heating rate of 10 °C min^{-1} under nitrogen flow (50 mL min^{-1}) to prevent sample oxidation. The crystal weight fraction or crystallinity was calculated according to the following Eq. 1:

$$X_c = \frac{\Delta H_f}{w\Delta H_f^{\circ}} \times 100 \tag{1}$$

where ΔH_f is the measured enthalpy of the sample's melting, ΔH_f° is the enthalpy of a 100% pure crystalline sample's melting; 188 J g^{-1} is a commonly accepted average value for PA6 and w is the mass fraction for PA6 in the composites [26].

Mechanical testing

The tensile, flexural and Izod impact tests were performed according to ASTM D638 (speed of 5 mm min^{-1}), ASTM D790 (speed of 1.5 mm min^{-1}) and ASTM D256, respectively. For the tensile and flexural tests was used the Universal testing machine EMIC; and CEAST equipment with a 2.75 J hammer was employed for the Izod impact tests. The specimens' dimensions in the tensile and flexion tests were: 165 × 13 × 3.2 mm and 127 × 12.7 × 3.2 mm, respectively. In the Izod impact test, the specimens had a square section with 10.2 mm of side and 75 mm in length, with a notch at 28 mm with *V*-shaped. Toughness was calculated by integrating the area under the curve of the stress versus strain graph. Seven specimens were tested for each condition assessed.

Scanning electron microscopy

After tensile test, the fractured surface was analyzed by Field Emission Gun Scanning Electron Microscopy (FEG-SEM Model Tescan—Mira 3) with an operating voltage of 15 kV. To improve the samples conductivity and avoid electrical charging, before analysis, all samples were coated with a thin gold layer for 10 min.

Statistical analyses

The mechanical data were analyzed using descriptive statistics in the Statistica software. The results were expressed as mean ± standard deviation. Specimens were assessed for each condition with six degrees of freedom. Multiple comparison Fisher test was used to determine the significant differences between group means in an Analysis of Variance setting (ANOVA) between samples, with 99% confidence interval.

Results and discussion

FTIR spectroscopy

FTIR spectroscopy was performed in all samples with different hydrations conditions to evaluate eventual changes in the frequencies and shapes of the OH^- absorptions of water. Figure 1 presents the spectra of the eight samples, all spectra showing the characteristic peaks of polyamide-6. There are no significant changes in the samples as no supplementary peak appears (Fig. 1a, b). Table 3 summarizes the assignment of FTIR bands.

The sharp and very strong peak (*) at 3291 cm^{-1} (Fig. 1c, d), is assigned to the N–H stretching of the hydrogen-bonded N–H in the crystalline portion and in the ordered amorphous portion [27]. The swelling at (⌂) 3468 cm^{-1} attributed to the OH stretching absorption of the sorbed water, appears in all spectra except in the TDry sample (black line). For Lim et al. [28], the shoulder in approximately 3490 cm^{-1} is assigned to free N–H stretching mode and it increases when the

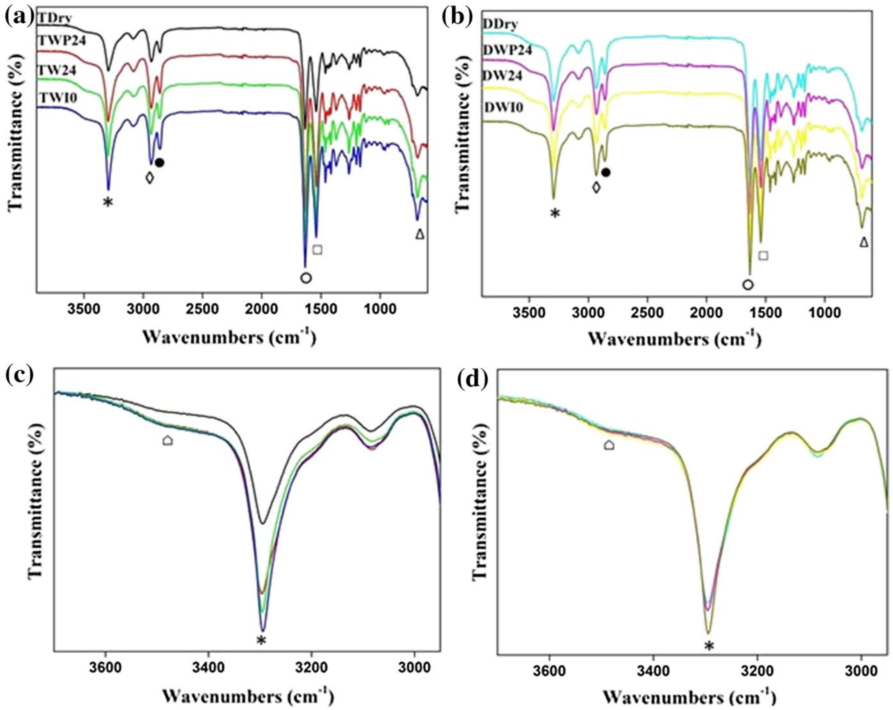

Fig. 1 FTIR spectra **a**, **b** of the samples evaluated in this study **c**, **d** from 3700 to 3100 cm^{-1}

Table 3 Assignment of FTIR peaks of the samples evaluated in this study

FTIR peak (cm^{-1})	Assignment
(*) 3291	Hydrogen-bonded N–H stretch in the crystalline phase
(⌂) 3468	Free N–H stretch
(◇) 2932	Asymmetric CH_2 stretch
(●) 2858	Symmetric CH_2 stretch
(○) 1633	Amide I, C=O, C–N stretch and C–C–N deformation
(□) 1538	Amide II; N–H in plane bending, C–N, C–C stretch
(Δ) 684	Amide V

immersion time in distilled water increases, overlapping the N–H stretching bands. Iwamoto and Murase [29] studied the interactions of nylon-6 with water by following the FTIR spectra and determined that the sorbed water could have some influence on the intensity of the 3291 cm^{-1} peak.

Depending on how aggressive, or not, the hydration conditions used may be, the changes in the polyamide chemical structure caused by the water sorption mechanism will be more noticeable or not. In many cases, these changes are imperceptible, the FTIR technique was unable to detect them, and so our study cannot affirm that any chemical change in polyamide structure has taken place. In this sense, some authors use the XPS technique to investigate the effect of water on chemical bonds.

Differential scanning calorimetry

A quantitative determination of the crystallization of a semicrystalline polymer after processing the thermomechanical is mandatory to understand its properties. The thermal properties obtained from the DSC heating curves (Fig. 2) were summarized in Table 4, which includes melting peak temperatures (T_{m1}, T_{m2}), enthalpy

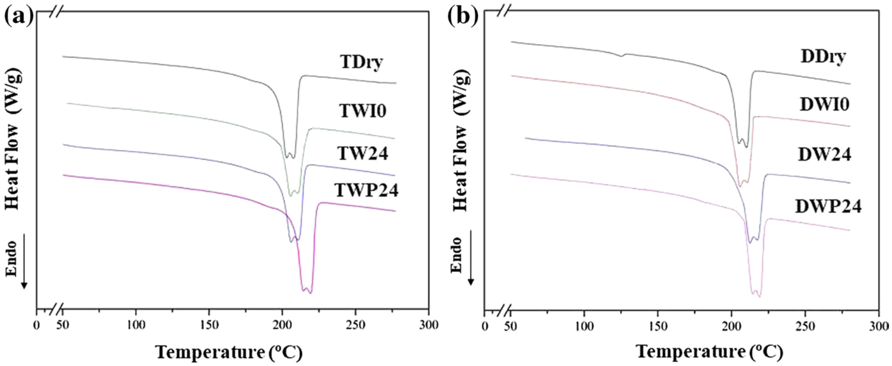

Fig. 2 DSC curves corresponding to the second heating **a** Technyl and **b** Durethan samples

Table 4 DSC thermal properties of composite at various hydrations conditions (second heating)

Samples	T_{m1} (°C)	T_{m2} (°C)	ΔH_m (J g^{-1})	X_C (%)
TDry	205	210	50.6	26.9
TWI0	205	210	53.6	28.5
TW24	205	210	58.0	30.8
TWP24	215	220	61.7	32.8
DDry	205	210	56.1	29.8
DWI0	205	210	57.3	30.5
DW24	210	215	58.4	31.0
DWP24	210	215	60.9	32.4

of melting (ΔH_m), and degree of crystallinity (X_c). The X_c of the composites can also be calculated after the melt-crystallization process using a reference heat of fusion for the fully crystalline PA6 that equals to 188 J g^{-1} (Eq. 1).

Polyamides are able to crystallize because of strong intermolecular hydrogen bonds through the amide groups, and because of Van der Waals forces between the methylene chains. The second heating process of PA6, reveals the existence of two main crystalline phases: the stable monoclinic α form and the unstable monoclinic γ form [30]. The two melting peaks (T_{m1} and T_{m2}) were associated with a difference in melting temperatures between the two phases present. The melting peak around 215 °C could be attributed to the melting of the α-crystalline form. The one around 210 °C probably indicates the melting of the thermodynamically unstable γ-crystalline form. The enthalpy of melting increases from dry to wet conditions, which can be associated with a possible process of recrystallization of the polyamide, which strengthens the molecular arrangement, needing greater energy for the melting of the crystals. Consequently, the degree of crystallinity was slightly higher in dry conditions than in wet conditions. This difference is not significant (is within the experimental error of the measurements, which is in the order of 10%) therefore, the effect of crystallinity on the mechanical properties of the samples can be considered negligible. Glass fiber reinforced-polyamide-6,6 specimens subjected to dry, 50% relative humidity, and wet conditions, revealed similar results, with the absorption of water, there were negligible changes in the melting temperature, degree of crystallinity and enthalpy of melting [31].

Mechanical properties

Table 5 illustrates the average values of the tensile and flexural modulus, toughness and impact obtained for all specimens evaluated. Comparing the specimens in the different processing conditions and using the ANOVA-Fisher multiple comparison tests with a significance of 1%, we noted that the tensile modulus of the TDry sample was 9.35% higher than the DDry sample, and these were higher than the other samples, indicating that the samples in the dry condition have characteristics

Table 5 Summary of mechanical properties (standard deviations) of composites under different conditions

Samples	Tensile modulus (MPa)	Flexural modulus (MPa)	Toughness (J m^{-3})	Impact (J m)
TDry	7607 ± 220	6119 ± 129	275 ± 17	135 ± 26
TWI0	6304 ± 218	3907 ± 152	524 ± 18	197 ± 6
TW24	5920 ± 225	5266 ± 143	404 ± 36	213 ± 10
TWP24	5433 ± 230	5222 ± 71	277 ± 62	149 ± 12
DDry	7113 ± 228	6360 ± 159	281 ± 25	124 ± 9
DWI0	6026 ± 246	3962 ± 35	519 ± 28	273 ± 7
DW24	5732 ± 235	4316 ± 34	406 ± 42	238 ± 10
DWP24	5491 ± 227	4522 ± 147	307 ± 33	162 ± 9

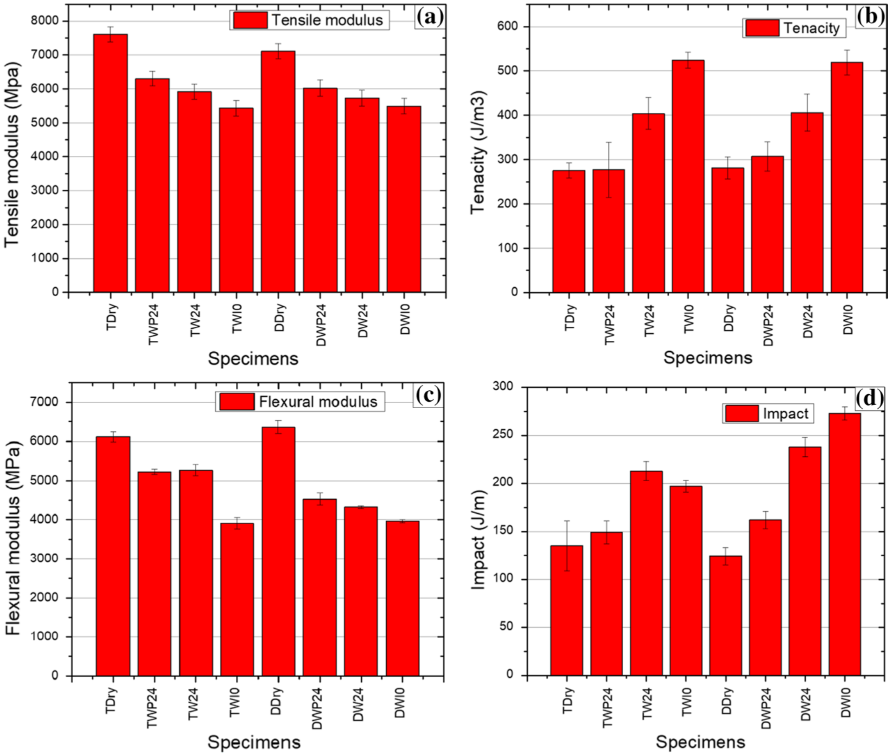

Fig. 3 Tensile modulus **a**, toughness **b**, the flexural modulus **c** and impact test **d** of composites subjected under different hydrations conditions. Confidence intervals used for the averages were 99%

of greater rigidity. We observed that for all hydration environments evaluated, there was no significant difference between the tensile strength values (Fig. 3a).

According Huang and Sun [32] the tensile strength decreased when your glass/polyester composites were immersed in water. The amount of water in the composites caused swelling of the matrix, deteriorate the interphase and, therefore, weaken the composites fiber–matrix interfacial shear strength. Some studies suggest that the water absorption process is a physical phenomenon and can be recovered with water desorption [33]. There are different reports in the literature about the recovery of the water absorption effect on materials properties. Nonetheless, for composite materials, the loss of mechanical properties is not only related to the plasticization effect of water but also to the degradation of the fiber-matrix interface. Xu [34] studied the mechanical properties of cellulose fibers to reinforce nylon 6 and nylon 66. The result indicated that the change in properties (and fiber size) determines the final properties of composites.

Variation of toughness defined as the area under the stress–strain curve is shown in Fig. 3b. Comparing statistically the toughness of both specimen (Technyl and Durethan), the highest values were obtained for the immediate hydration condition (TWI0 and DWI0), with no significant differences concerning the manufacturers' values (Fig. 3b). PA6 material is commonly used in applications where high toughness is required, and water shows beneficial effect on this property. This is due to the plasticizing effect of water on polyamides, as these molecules are lodged between macromolecular chains, reducing the attraction forces between them and increasing the polymer molecular mobility, thus resulting in increased toughness.

However, significant differences were observed in relation to the toughness of the immediately hydrated samples (TWI0 and DWI0) and the hydrated samples after 24 h (TW24 and DW24) for both suppliers. Also we observe (Fig. 3b) that the Wet Pack conditions (WP24) and dry conditions did not show any difference between each other; the toughness values of these composites were the lowest, possibly due to the low hydration of the specimens. Low hydration reduces the mobility of the molecular chain, increasing intermolecular forces, and making the plasticization process more difficult, demonstrating the hydration via Wet Pack to be inefficient to reduce the stiffness under the experimental conditions used.

From Fig. 3c, we observed that for dry conditions the flexural modulus reached the maximum values of 6119 MPa and 6360 MPa, respectively. The samples treated with immediate hydration showed the lowest values of flexural modulus, with no significant differences between the statistical means of both manufacturers. Normally, the increase in the moisture content in the PA6 polymer matrix generates a decrease in flexural strength, originating from the hydrogen bonds between water molecules and the PA6 matrix. However, some authors [35] report the existence of microvoids that allow moisture to fill these spaces and enhance the bending behavior.

The behavior of the impact strength of the composites is presented in Fig. 3d. A similar trend to the toughness test was obtained in the results of impact resistance. The hydration condition that provided the highest impact resistance was obtained for the DWIO samples, and the impact resistance values of the Durethan composites were 27.84% higher than the Technyl materials. Similarly in immersion method after 24 h the injection (WI24), the Durethan composites were 11.5% higher than the Technyl samples. However, statistically in Wet Pack (WP24) and dry conditions, there were no significant differences between conditions and neither among manufacturers, showing the inefficiency of hydration in the Wet Pack condition. In summary, the ANOVA-Fisher multiple comparison tests showed that in the experimental conditions studied, the mechanical properties of the Durethan composites were statistically more significant than the Technyl composites, for a confidence interval of 99%. The diffusion of water molecules through the material generates separation of the polymeric chains, increasing the mobility of the chains and consequently the toughness and resistance to impact, essential properties in components such as the junction of the stretcher that is molded by plastic injection.

FEG-SEM analysis

In order to explain the mechanical behavior obtained, we decided to explore the water effect on fiber/matrix interface, for this, SEM micrographs of the samples' fracture surface in different hydration conditions are shown in Fig. 4a–f. In the dry condition (DDry and TDry, Fig. 4a, e, respectively), a fragile fracture is observed, where the fibers were extracted from the polymer leaving well-defined holes in the rupture section. It is also possible to observe exposed fibers with a smooth surface and without polymer attached to them. In the Wet Pack condition (Fig. 4b, f), the fracture region has characteristics very similar to the dry condition, a nearly clean fiber with small retention of the matrix was observed. The holes where the fibers were extracted show small deformations indicating low hydration and plasticity of the polymer. This result explains the similarity in the values of impact resistance and toughness in the dry and wet pack conditions. The fracture region in the immersion condition after 24 h the injection (Fig. 4c, g) evidenced fibers totally impregnated with the polymer matrix, and deformed holes, showing plasticity and hydration in the fracture region. Finally in the DWI0 and TWI0 samples, an excellent bond between fiber and matrix was observed (Fig. 4d, h), as well as continuous cover of fiber by matrix attesting significant plastic deformation [36]. This fact was reflected in the superior performance of these samples in the impact and toughness tests concerning the other conditions.

Water increases the mobility of the molecular chain making it an important plasticizing agent. The different hydration conditions did not significantly affect the crystallinity of the samples, but we observed that the three different hydration forms that the industry performs, via immediate immersion or up to 24 h after the injection process, were effective in relation to hydration. In the Wet Pack system, where injected components were subjected to hydration in a package with water 15 wt%, hydration was inefficient and therefore showing low plasticity in the evaluated specimens.

Conclusions

The influence of different industrial hydration conditions on the mechanical properties of two commercial composites (Durethan and Technyl) made with glass fiber reinforced PA6 was investigated. After the injection of the composites, the specimens were submitted to different hydration environments: Wet Pack and two immersion conditions: immediate and hydration after 24 h the injection. The FTIR

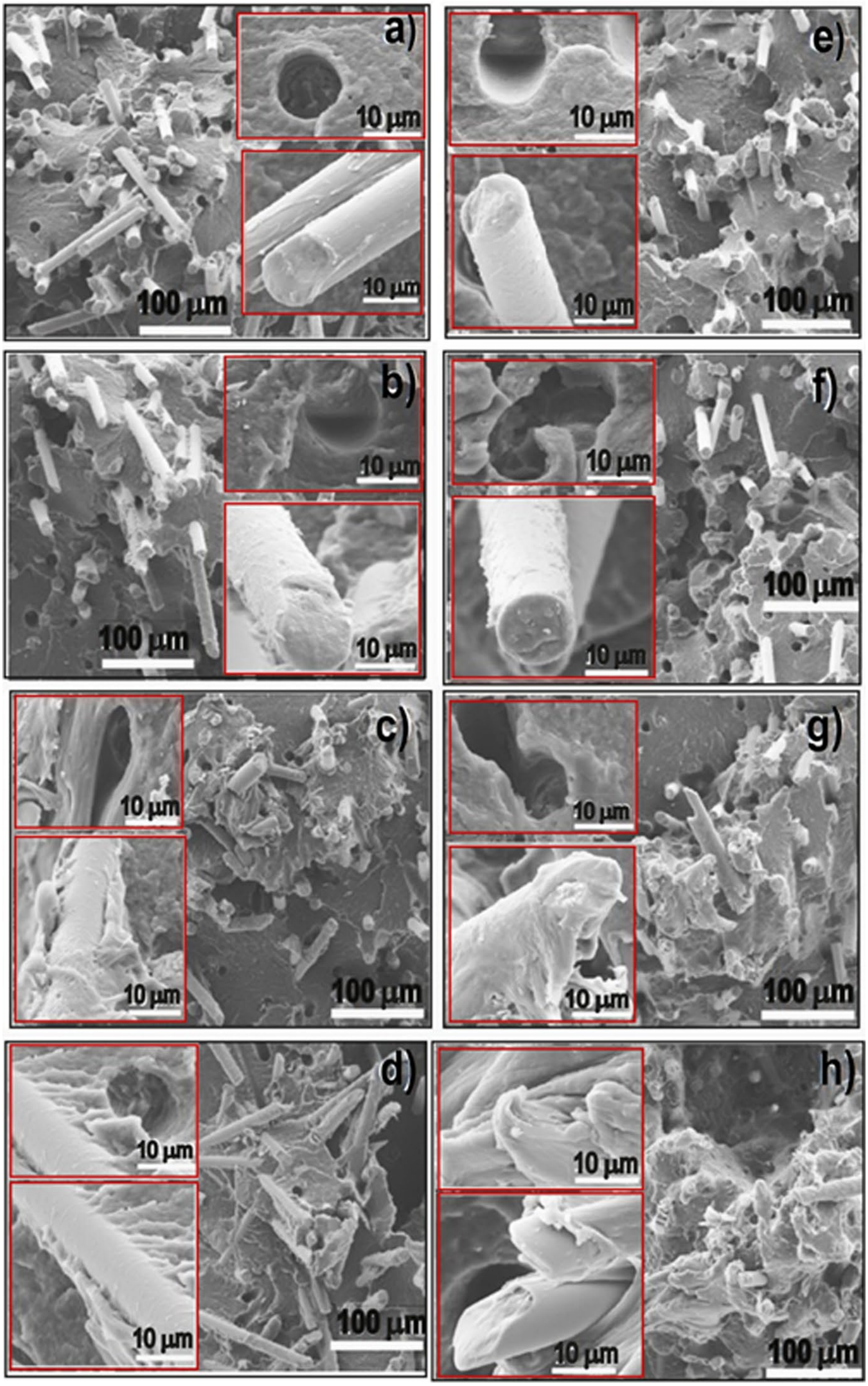

Fig. 4 SEM micrographs of the fracture surface after tensile tests of **a** TDry, **b** TWP24, **c** TWI24, **d** TWI0, **e** DDry, **f** DWP24, **g** DWI24 **h** DWI0 samples

characterization of the specimens did not show the existence of new peaks. The different hydration conditions did not significantly affect the crystallinity of the evaluated samples. With the aid of the ANOVA-Fisher multiple comparison tests, we concluded that the immediate immersion was statistically more significant than the Wet Pack method, where the Durethan composites presented an impact resistance 27.84% higher than the Technyl composites. Samples hydrated by the Wet Pack method, showed behavior to the samples without hydration, showing low toughness and impact resistance. There were significant differences in the comparison between the hydration methods, the highest toughness and impact resistance were obtained for samples with immediate immersion, followed by immersion after 24 h of injection. Durethan composites hydrated by immediate immersion showed the best mechanical performance, being the most suitable to be used as a stretcher junction component.

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Author contributions Based on the experimental data, N.B. Guerra analyzed the results and wrote the article. E. Ferreira carried out the preparation of the injection molds, the mechanical tests and the microscopic characterization of the samples. J. Duarte and M. Zeni performed and analyzed the Differential Scanning Calorimetry tests. J.S. Weber was in charge of placing and supervising the samples under the different conditions of guaranteed hydration. C.A. Figueroa and A.F. Michels planned, supervised and revised the final document.

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