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Polyamide 6/carbon fiber laminated composites

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The work presents a novel simple route to obtain new laminated composites based on carbon fiber fabric layers and polyamide 6 plates, processed via hot melting pressing technique. The paper describes the obtaining process of the composite and the evaluation of its characteristics. Polyamide 6 was used as thermoplastic matrix in form of extruded plates, and it was reinforced with two layers of carbon fiber plain weave fabric. The assembly was pressed at high temperature in order to obtain a laminated structure material. Electronic microscopy and infrared spectroscopy analyses as well as mechanical tests were performed on the final composites as well as on the raw matrix material. The laminated composite was characterized using a relatively new technique consisting of FTIR microscopy. The results of these analyses confirmed the results obtained for the mechanical testing of the tension and flexural strength and modulus. The carbon fabric laminate presented remarkable increase of mechanical characteristics regarding both tensile and flexural strength and excellent improvement of elasticity modulus, with minor increase of density. The characteristics of the new composites based on carbon fiber make them potential candidates for different applications where high mechanical performance and lightweight are crucial elements.

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1. Introduction

In any high-tech structural application, like automotive, marine, aircraft and aerospace, where strength, stiffness, durability and light weight are required, thermoset polymers such as epoxy resins are seen as the standard of performance for the matrix of the composite [1, 2]. Nowadays, there is an increasing interest in using thermoplastics to replace thermosets for laminated composites fabrication due to some advantages that include high toughness, shorter manufacturing cycles, no refrigeration storage required, exhibiting practically infinite shelf life, reprocessing and recycling possibilities [2, 3].

Fiber-reinforced polymers (FRP) are being used more and more intensively in the last decade in civil engineering [4], automotive, aerospace [5], military [6] and sports good industry [7], competing with materials such as steel, aluminium and concrete in cars, aircrafts, buildings, bridges and everyday sports goods [8]. Carbon fiber is used in important structural composites applications, being considered the reinforcement alternative for advanced composites. 30% of all carbon fiber is used in the aerospace industry, being widely used in aircraft components and structures, where its superior strength to weight ratio far exceeds that of any metal [7].

Embedding reinforcing fibers in thermoplastic matrix leads to materials that provide low density, high specific strength and stiffness. The performance of this class of composites depend both on the properties of the fibers due to the manufacturing process [9] and the matrix, but also on the interface between them [5]. The fiber/matrix adhesion issue is one of the main reasons why full potential of carbon fibers as composite materials reinforcement, has not yet been achieved [5]. It is desirable to improve all matrix-dominated properties to enhance the potential of structural applications of composites [10].

Carbon fiber/polyamide are studied as components in automotive applications by Ford Research Laboratory [11], as potential low- cost next generation automotive composites [12, 13], for applications in electromagnetic interference and radio frequency interference evaluating their shielding effectiveness [14], or for military applications [15]. Up to present most literature studies present fiber reinforced polyamide composites in which the reinforcing agent is in form of short carbon fibers and the processing route involves melt mixing [16] or takes place in extruder [15], chopped carbon fibers using microinjection technique [17], long carbon fibers used in anionic polymerization of lactam [18] or different glass fiber reinforced polyamide composites [19, 20]. Very few studies involving 2D fabric reinforced polyamide 6 composites are reported in literature, while none uses hot pressing technique involving polymer plates.

The paper presents the obtaining of high stiffness thermoplastic composites with polyamide 6 matrix in form of extruded plates and carbon fiber fabric reinforcement using a simple hot pressing technique. The achievement process of these laminates using polyamide 6 plates and carbon fabric, using high temperature pressing method, constitutes the novelty of this work; the advantages of the method presented in this paper include reduced complexity of the technology, fast processing time and relatively low cost. The evaluation of mechanical characteristics of fiber reinforced laminate (2D reinforcing agent based composite), compared with PA6 material, highlights the fact that carbon fiber fabric based laminates presented a remarkable performance regarding stiffness and a more than satisfactory improvement of strength. Flexural modulus increased by 580 % and Young's tensile modulus by 400 %, flexural strength was almost doubled by the carbon fabric presence, while tensile strength increased by 20 %. Subsequent to mechanical testing, the laminated materials were characterized using FTIR microscopy, a relatively new technique, with few references in literature studies, and SEM microscopy in order to investigate the matrix fiber- interface.

2. Experimental

2.1 Materials

The matrix used was polyamide 6 (PA6), obtained from S.C.ICEFS Săvineşti, and it was delivered in pellets form and the 2-dimensional (2D) reinforcing agent consisted of carbon fiber plain weave fabric CARP/T193, produced by Chemie Craft, France, having 3000 filaments (3K warp), 193 g/m^2 weight per unit area. In order to perform a comparison with the obtained laminates samples, simple PA6 samples were developed and tested.

2.2 PA6 samples obtaining process

The polymer samples were obtained by direct melt extruding of the PA6 pellets in a twin-screw extruder with gradual temperature increase on the ten heating areas of the screw, in a temperature range from 195°C- 240 °C, at a 220 rpm screw rotation speed, followed by cooling in water bath, chopping and drying of the filaments. Subsequently to the extrusion process, the pellets were injection moulded to obtain test specimens with specific shape for the tensile mechanical tests (dumbbell) and flexural tests respectively (rectangular), as well as PA6 plates.

2.3 Laminate obtaining process

The laminated composites consisted of two carbon fiber fabric plies interposed between three plates of PA6. The laminates were obtained by stacking the layers alternately and pressing the structure at high temperature in a hydraulic CARVER hot platens press. The laminate was pressed with 30 kg/cm^2 increasing the temperature value from 25 °C to 240 °C, with a 5 minutes dwell time (time in which the temperature is kept at the same value) at 230 °C and 235 °C and 10 minutes at 240 °C, followed by cooling to room temperature while maintaining the same pressure. The obtained laminated plates had 3 mm thickness having 11.4 % by weight carbon fiber equivalent to 8 % by volume of the composite. Dumbbell and rectangular specimens for mechanical tests were cropped from the laminated composite plates.

2.4 Characterization techniques

Mechanical characterization. To evaluate their mechanical strength, the materials were subjected to mechanical tests at room temperature, using INSTRON 5982 machine. Tensile tests were performed according to SR EN ISO 527, using minimum 5 specimens of each sample, setting a 50 mm/min tensile rate for the PA6 polymer and 5 mm/min tensile rate for the laminate, as the standard suggests for single polymer and fiber reinforced polymers respectively, given the different values for the elongation at break of the carbon fiber and polymer.

3-point flexural tests were performed according to SR EN ISO 178/SR EN ISO 14125, on minimum 5 specimens of each sample, using a 2 mm/min speed of test for both polyamide sample and laminate, as the standard indicates, conventional deflection (1.5 x specimen thickness) and nominal span length (16 x specimen thickness).

Thermal stability under load. Thermal stability under load represented by the value of deflection temperature was evaluated using Qualitest HDT1 Tester, according to SR EN ISO 75, using 2 °C/min heating rate, 0.34 mm standard deflection. Rectangular specimens were subjected to 3-point bending test at 1.8 MPa flexural stress, in a siliconic oil medium.

Water absorption. Water absorption measurements were performed according to ASTM D 570 – 98. The specimens were dried at 50 °C and then allowed to cool to room temperature in a desiccator before weighing them to the nearest 0.1 mg. Water absorption tests were conducted by immersing the specimens in a deionized water bath at 25°C for 24 h, 48 h and 72 h. After each period of time, the specimens were taken out from the water, wiped with a dry cloth to remove surface water and then kept at room temperature for 1 h before reweighting them to the nearest 0.1 mg.

Density measurement. The density of the materials was measured to evaluate the weight change generated by the presence of the 2 layers of reinforcing agent. The density was calculated as the ratio between mass and volume; measurements were performed using rectangular shape samples with accurate dimensions. The mass of the samples was measured using an accurate analytical balance, while volume was measured using displacement method.

Physico-chemical and morphological analysis. The obtained materials were characterized in terms of the physico-chemical and morphological properties. Physico-chemical and morphological analysis were registered in the cross- section (break area of the specimen subjected to mechanical testing). The polymer was subjected to spectroscopy analysis using FTIR Nicolet 6700 FTIR (Thermo Nicolet, Madison, WI) spectrometer, and scanning electronic microscopy (SEM) using HITACHI S2600N. SEM and FTIR microscopy (iN10 MX Mid

Infrared FTIR Microscope) was performed on the laminate samples to evaluate the matrix/fiber interface.

3. Results and discussion

3.1 FTIR Spectroscopy

The IR analysis (Fig. 1.) presents the spectra of PA6 samples developed as control sample and also the spectra of pure PA6 pellet, in order to examine if the polymer suffered any thermal degradation during processing by injection. The spectra present the characteristic peaks for the material, showing the absorption peak from 1634 cm^{-1} corresponding to the stretching of the C=O bond from PA6 and the band from 1537 cm^{-1} corresponding to the deformation vibration of the N-H group (amide II) [21]. The band from 3294 corresponds to the N-H stretching vibration, and the one from 3084 cm^{-1} corresponds to the CH stretching vibration [22]. Bands from 2922 and 2853 cm^{-1} are assigned to CH_2 antisymmetric and symmetric stretching [23], while NH deformation and CH_2 scissoring determine the peaks from 1417 and 1461 cm^{-1} [22]. The bands from 1377 and 1201 correspond to CH_2 wagging vibration, and the one from 1244 appears due to N-H bending and C-N stretching [22]. CO-NH skeletal motion determines the appearance of 1168 cm^{-1} peak [24], C-C bending vibrations determine the bands at 970 and 576 cm^{-1} [22] and out of plane bending of NH appear at 683 cm^{-1} [22, 23]. Comparing with the PA6 pellet spectrum, it can confirm that no degradation of PA6 occurs during the processing, as no supplementary band appears.

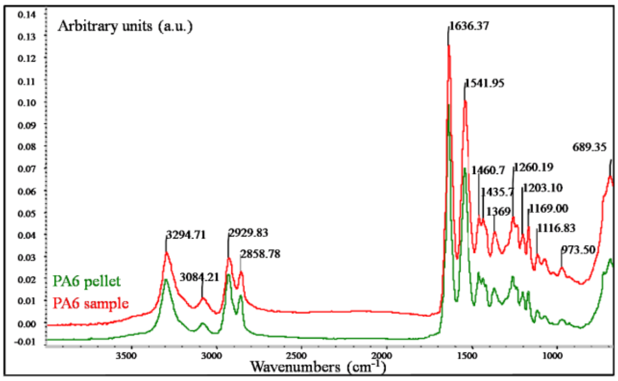

Fig. 1. FTIR spectra of the pure PA6 pellet and PA6 sample

3.2 SEM Analysis

All SEM images were registered in the fracture area of the mechanically tested samples.

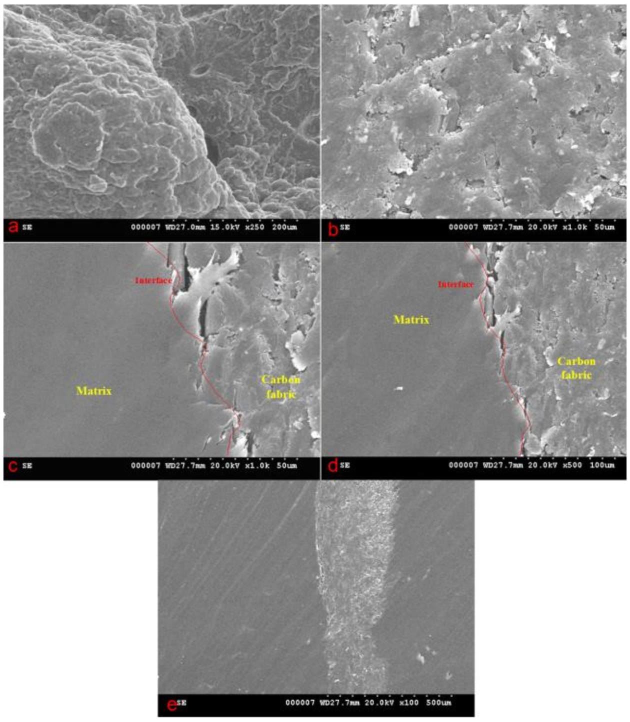

Fig. 2. SEM images of: PA6 polymer (a- x250), carbon fiber fabric (b- x1.0 k), laminated composite with PA6 matrix and carbon fabric reinforcement (c-x1.0 k, d- x500, e- x100)

SEM analysis was performed on the PA6 sample (Fig. 2- a), in order to visualize the morphology of the material, which is characteristic to a polymer and on the laminated composite, with the aim of visualizing the interface between the two phases. The SEM images of the fracture surface of PA6 reinforced with carbon fiber fabric demonstrate the good interface between the fabric and the thermoplastic matrix. The positive reinforcing of the matrix with the 2 carbon fabric layers was also confirmed by the results of the mechanical tests. At different magnifications, SEM images allow the visualization of layered structure of the material (Fig. 2- e) at low magnification, the carbon fiber/PA6 interface (Fig. 2- c, d) as well as the carbonaceous region after mechanical testing (Fig. 2- b). Based on these images, it can be seen that the carbon fiber layers are irregular, with thickness ranging between ~100 and 280 um. The carbon fiber/PA6 interface is very important to be analyzed and could offer valuable results which can explain some of the mechanical properties. In the case of PA6/ carbon fiber laminate the mechanical testing induced a strong stress which leads to PA6 detachment from the carbon fiber fabric in some areas, as it can be seen from the SEM images (Fig. 2- c), an issue that could be the reason for obtaining lower increment values for tensile and flexural strength properties compared to modulus increment values.

3.3 FTIR Microscopy

FTIR microscopy highlights the laminated structure of the PA6/carbon fiber fabric composite, and it is used to evaluate how homogeneous was the high temperature pressing lamination and to observe the specimen in detail in the cross section region. Based on the FTIR spectrum of the laminated material (Fig. 1.), the distribution of the components was monitored at ~1650 cm^{-1}.

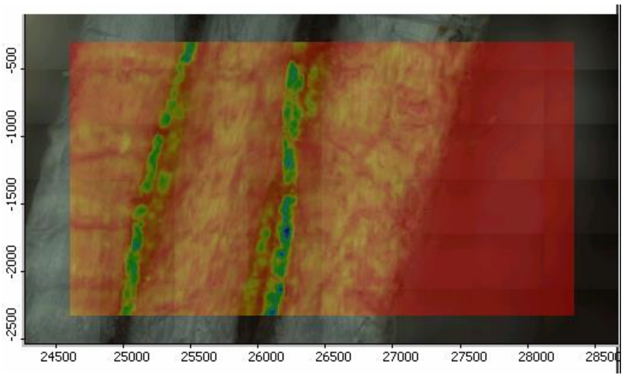

Fig. 3. FTIR image overlapped on video image of the PA6/carbon fiber fabric laminate composite

By overlapping the video image and the FTIR image, the polyamide and carbon fiber fabric layers can be highlighted. The FTIR image was recorded in the 715 – 4000 cm^{-1} domain, and for the overlapping from the wavelength intensities corresponding to the ~1660 cm^{-1} wavelength corresponding to the maximum intensity band of the amide 6 ([25]). The carbon fiber fabric can be observed in both regions with minimum absorbance at 1660 cm^{-1}, while polyamide 6 can be observed in the 3 regions with high absorbance. It is important to mention that the carbon fibers that form the carbon fabric are visible, especially at 26 000 µm. The distance between the carbon fabric layers is approximately 1000 µm (1 mm), the polyamide layers having approximately 1000 µm.

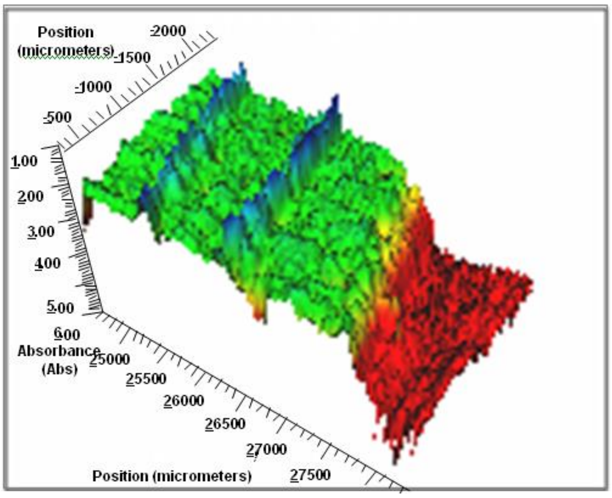

Fig. 4. FTIR signal intensity based 3D image of the PA6/carbon fiber laminate

Also, based on the FTIR results, the laminated structure is visualized in the 3D image (Fig. 4), especially for the distance between layers. The region over 27000 µm, colored in red (at the right side), represents a void owned to the level difference between the sample and the mount.

3.4 Mechanical Testing

The mechanical evaluation of carbon fiber laminated composite performance consisted of tensile and flexural testing of the specimens. Tensile tests focused on the modification of tensile strength and Young's modulus of elasticity in order to compare with the results obtained for the simple PA6 matrix

The specimens' geometry for tensile tests was 1B type (dumbbell), as the one presented in Fig. 5.

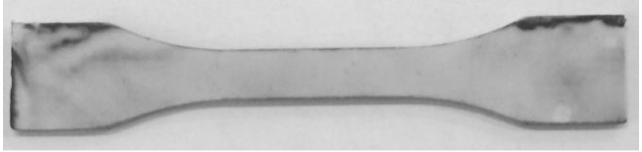

Fig. 5. PA6/carbon fiber laminate specimen for tensile test

Fig. 6 presents the tensile curves for the polymer sample and for the laminated composite. Tensile strength of a material is the maximum amount of tensile stress that it can be subjected to before failure.

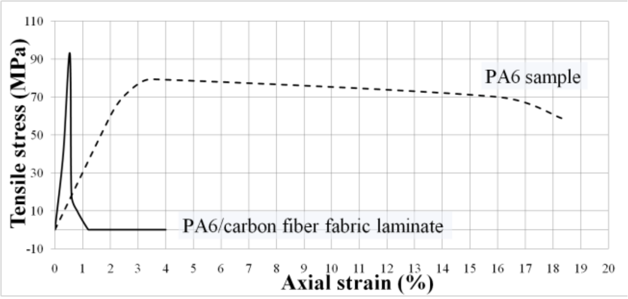

Fig. 6. The stress- strain curves for the tensile testing of the PA6 sample and PA6/carbon fiber laminate

Some specimens presented fracture in the middle of the narrow area, others near to the broad area of the dumbbell shape, and one specimen presented failure of the broad area, near the grips. Except this one, all the other occurred failure modes are considered valid and used to calculate the tensile strength and modulus of the tested specimens. Fig. 7 shows one of the specimens tested in tensile, which presented valid failure mode.

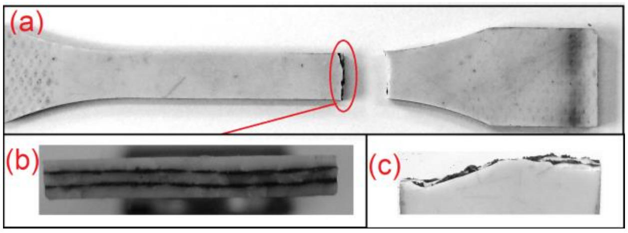

Fig. 7. Specimen after tensile testing (a); Top view of the fracture area (cross-section) (b); Lateral view of the fracture area (c)

The mainly failure mode for all cases is fiber breakage and matrix cracking; fiber breakage and matrix cracking occurred synchronously. This failure mode confirms the good interface between the two phases, showed by SEM images, that is owned to the matrix penetrating through the fibers that compose the carbon fabric.

Fig. 8 presents the load- extension evolution observed during flexural testing of the materials. The test was carried out until conventional deflection was reached, and as the specimen did not break until this value, the flexural strength is represented by flexural stress (at conventional deflection), calculated using the flexural load at the value of conventional deflection.

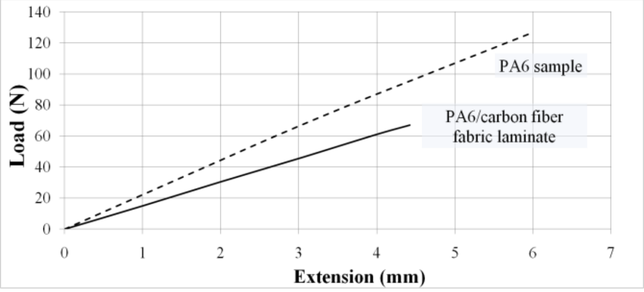

Fig. 8. The load-extension curves for flexural testing of the PA6 sample and PA6/carbon fiber laminate

For each sample tested, an average of optimum specimen replicas was calculated. Comparing the tensile and flexural strength and modulus results (Table 1) it is observed the positive trend in reinforcing effect of carbon fiber (2D fabric) in the PA6 matrix composites. The results show a substantial increase in mechanical strength and an excellent improvement of the elasticity modulus in both tensile and flexural tests, while maintaining a low density advantage. Tensile strength of the laminate increased by 17.4 %, from 79.4 MPa for the simple PA6 to 93.2 MPa for the fiber reinforced PA6, while the Young's modulus increased by 400 % compared to the single PA6 (3.18 to 15.83 GPa). In terms of flexural characteristics of the laminate, the improvements were even higher compared to the simple polyamide sample, flexural strength presented an increase by 48 %, while flexural modulus increased by approximately 580 %.

These remarkable mechanical improvements were obtained at a fiber content of only 8 % by volume of the composite and just a minor increase in density from 1.11 g/cm^3 for PA6 to 1.15 g/cm^3 for the laminate (less than 4 % increase). The **density measurement** was performed in order to evaluate the weight change generated by the presence of the 2 layers of reinforcing agent, and to assess the possibility of using this kind of materials in applications where light weight is an advantage.

The carbon fabric laminate superior mechanical characteristics are owned also to the polymer penetration through the fibers that constitute the fabric, leading to a composite structure similar to that of reinforced concrete, without the development of any supplementary polymer-fabric interaction. The good bonding between the phases of the laminated composite is also confirmed by the axial strain at break values (Fig. 6), as polyamide 6 material presented an extension of 18.3 %, while the PA6/carbon fiber laminate strain at break was approximately 1.2 %. This result is due to the fiber effect in the laminate, as carbon fiber exhibits values for elongation at break between 1-1.5 % [26, 27]. Carbon fiber determines the decrease in elongation property for the composite, leading to high tensile strength and modulus laminates, results obtained due to the good fiber-matrix adhesion.

Table 1 Mechanical and thermo-mechanical characteristics of PA6 based materials

Measured property	PA6	PA6/carbon fiber fabric laminate
Density, g/cm^3	1.11	1.15
Tensile stress, MPa	79.4	93.2
Young's Modulus, GPa	3,18	15,83
Tensile elongation, %	18.3	1.2
Flexure stress, MPa	78.6	116.6
Modulus (Young's Flexure stress), GPa	2.31	15.71
HDT, °C	70	77.1

3.5 Thermal stability under load

Thermal stability under load measurements (Table 1) show an increase in the heat deflection temperature (HDT) of the PA6/carbon fiber laminate up to 77.1 °C compared to 70 °C for the PA6 matrix. In comparison to the mechanical results improvement, the increment is not highly significant. This could be explained by the fact that the carbon fiber layers, located in the inner region of the cross section, near the symmetry axis have a reduced influence on the inertia moment of the specimen and consequently on the deflection in the center of the specimen. Also, the matrix intrinsic properties are not modified when used in the laminated composite, so its thermal behavior under load is mainly the same as in the simple polymer. Considering that there is a good physical interface between the fabric and the polymer, carbon fabric bends along with the matrix that "softens" as temperature increases.

3.6 Water absorption

The water absorption of the PA6 matrix and carbon fiber laminated was measured after 24 h, 48 h and 72 h immersion in deionized water at room temperature. Percentage of absorbed water is calculated using the equation:

$$M_t(\%) = \frac{M_w - M_d}{M_d} \cdot 100 \qquad (1)$$

where: M_t- increase in weight (%), M_w- weight of the wet sample (g), M_d- weight of the conditioned (dried) sample (g)

Water absorbtion evaluation was performed in order to verify the bonding of the polyamide to the fabric at the edge of the laminate plate in aqueous environment. It can be noticed that both PA6 and PA6/carbon fiber laminate absorbed approximately the same percent of water after 24, 48 and 72 h immersion (Table 2). This result demonstrates that the surface of the edge section of the laminate, where the layered struture is exposed to water, is sufficiently uniform, given by the good physical bonding between the fabric and the polymer. The water does not penetrate the layered structure itself, but is only absorbed by the matrix.

Table 2 Water absorption at different immersion times

Immersion time (h)	Water absorption (%)	
	PA6	PA6/carbon fiber fabric laminate
24	0.97	0.97
48	1.41	1.37
72	1.81	1.85

4. Conclusions

Essentially, the results can be summarized in a few conclusions. The study presents the analysis of laminated materials consisting of carbon fiber and polyamide 6 matrix, obtained through a simple and reduced complexity process that uses high temperature pressing of the two types of materials layers into a laminated structure composite. The advantages of this method include the reduced complexity of the technology, fast processing time and relatively low cost.

The microscopic characterization of the laminated materials included SEM microscopy as well as FTIR microscopy, the latter being a relatively new technique with few literature references. Images obtained using both techniques showed a good interface between the matrix and the carbon fiber fabric, observation that was also confirmed by the results of the mechanical tests, and also by the water absorption test.

Polymer/fabric laminate composites presented remarkable improvement of mechanical characteristics, compared to the simple polymer both regarding strength (tensile strength increased by approximately 20 %, flexural strength by 48 %) and especially elasticity modulus (Young's modulus increased by 400 %, while flexural modulus by 580 %) with an insignificant density increase compared to PA6. These facts may support the idea that this type of laminated materials are suited for advanced applications in transport industry (automotive, aeronautics) where lightweight materials that exhibit high mechanical performances are essential elements. Future studies will take into consideration to develop laminated composites with different design (using different numbers of carbon fiber fabric layers) and also a modification of the fabric in order to enforce the fiber- matrix adhesion by chemical interactions as well, in addition to the polymer penetration through the fibers that constitute the fabric.

Acknowledgments

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