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doi: 10.5028/jatm.v10.710 ORIGINAL PAPER

An Evaluation of the Dielectric Barrier Discharge Treatment on the Mechanical, Structural and Morphological Properties of Carbon Fibers

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ABSTRACT: Carbon fibers (CFs) have been widely applied in the manufacture of polymeric composites. The mechanical properties of these composites depend on the adhesion in the matrix/CF interface. Since the CFs have poor adhesion properties, a surface treatment of the CFs becomes necessary. The plasma process used in this work was the dielectric barrier discharge (DBD). The DBD treatment of twill weave carbon fibers (2×2) was performed during 2.0; 5.0; 7.5 and 10.0 minutes. CF/polypropylene (PP) composites were produced by hot pressing method. The surface morphology, structure and mechanical properties of CFs were characterized by scanning electron microscopy (SEM), atomic force microscopy (AFM), Raman spectroscopy, interlaminar shear test (ILSS) and dynamical mechanical analysis (DMA), respectively. SEM images show great changes on the CF surface after the treatment, AFM results show that the fiber treated for 2.0 min exhibited the highest roughness and Raman spectra revealed that the carbon fiber crystalline structure did not change after the treatment. The ILSS and DMA results show an increase of the mechanical strength and no major change in the glass temperature of the composites after the DBD treatment, respectively. Some results were compared to those obtained in our previous study, in which the plain weave fiber (1×1) was evaluated.

KEYWORDS: Carbon fiber, Dielectric barrier discharge, Surface treatment, Composites.

INTRODUCTION

Carbon fibers (CFs) have excellent properties such as high mechanical and thermal resistance, high elastic modulus and electrical conductivity, chemical stability, low specific weight, high heat and corrosion resistance (Santos *et al*. 2013; Guo *et al*. 2010; Akonda *et al*. 2012; Zhang *et al*. 2011). Therefore, they have been employed as reinforcement in the manufacturing of composites that are widely applied in some areas such as automotive sector, civil construction, medical prosthesis and, mainly, in aeronautics and aerospace industries, etc. (Liu *et al*. 2015; Santos *et al.* 2013; Li *et al*. 2009; Zhang *et al*. 2013; Kim *et al*. 2011).

In composite materials, the polymeric matrix is responsible for transferring the applied load to the fibers, thus, it is very important to keep a good adhesion between the matrix and the fiber to obtain composites with better mechanical resistance (Brocks

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et al. 2013; Sharma *et al*. 2014; Wu *et al*. 2016). Besides its advantageous properties, the CFs possesses low adhesion to polymeric materials. The low interface adhesion of composites is a drawback, because this can lead to delamination and a decrease of material mechanical resistance. Therefore, a surface modification is required to increase the adhesion on the CF/polymer interface.

The interfacial adhesion in the composites can be originated by mechanical and chemical processes (Brocks *et al.* 2013). Some surface modifications have been used for this purpose, such as 'wet' (chemical), 'dry' (plasma) and 'multiscale' modification (Montes-Morán and Young 2002, Li *et al*. 1997, Guo *et al*. 2010, Zhang *et al*. 2011, Sharma *et al*. 2014). The chemical treatments consist in acid baths that are very pollutant. In consequence, plasma processing has emerged as an alternative approach because it is environmentally friendly. Among different plasma treatments, dielectric barrier discharge (DBD) produces some modifications, such as surface roughness and introduction of polar groups on the surface (Montes-Morán and Young 2002, Sarraf *et al*. 2007).

DBD can produce non-thermal plasma (cold plasma) at atmospheric pressure, thus this process has some advantages comparing to the other plasma processes because it does not require closed chambers, special gases or vacuum equipment. Therefore, it also can be easily implemented in on-line industrial processing (Wang *et al*. 2010).

The use of the CF has continually grown (Akonda *et al*. 2012) since its commercialization in aircraft manufacture (1969), replacing conventional materials such as aluminum and titanium alloys for primary structures (Akonda *et al*. 2012; Soutis 2005, Rezaei *et al.* 2009).

There are many types of CF fabrics and therefore it is important to investigate the CF braiding influence on the mechanical properties of composites. Paiva *et al.* (2005) compared two types of fabrics: plain weave (1×1) and eight harness satin (8HS) and compared two types of resins. It was observed that the different polymeric resins altered the mechanical resistance of the composites, while different fabrics did not change their mechanical properties. On the other hand, some authors (Brocks *et al*. 2013) investigated two different types of reinforcements and noticed that twill fibers had greater adherence. However, as far as we know, the effect of plasma treatment on the shear resistance of different CF fabrics has not been investigated yet.

Thus, in this work, the effect of DBD treatment on the mechanical, structural and morphological properties of twill weave fabric (2×2) was investigated for PP/CF composites production. The CFs were characterized by SEM and AFM to analyze their surface morphology and Raman spectroscopy to analyze the crystalline structure. In addition, the adhesion on the composite interface was characterized by interlaminar shear test (ILSS) and the glass transition temperature by dynamical mechanical analysis (DMA). The results were compared with our previous work (Santos *et al*. 2013), in which the plain weave fabric (1×1) was treated by DBD plasma.

EXPERIMENTAL

MATERIALS

Carbon fiber (CF)

The as-received CFs in the form of twill weave braiding (2×2) were acquired from Texiglass Company. Each carbon fiber tow contains 3000 monofilaments with sizing and its density ranges between 1.6 and 2.0 g/cm^3.

Polypropylene (PP)

A 0.08 mm-thick isostatic polypropylene (PP) film was used, with density of 0.95 g/cm^3 and a melting temperature of 170 °C. This film was provided by the Polibrasil Resinas industry, the same film employed in Santos *et al*. 2013.

DIELECTRIC BARRIER DISCHARGE TREATMENT

The DBD reactor operates at atmospheric pressure. It has two stainless steel electrodes (ϴ11.5 cm). The inferior electrode was turned on high voltage source and other was grounded. As dielectric barrier was a flat glass with a 2 mm-thickness, attached to both electrodes. The gap between two dielectrics was set to 4 mm.

The voltage magnitude was 35 kV peak to peak at a frequency of 60 Hz. A schematic of the DBD system was detailed in a previous work (Santos *et al*. 2013). CF samples (7 cm × 7 cm) were treated at 2.0, 5.0, 7.5 and 10.0 minutes.

HOT COMPRESSION MOLDING

The composites were produced by hot compression molding process, with treated and untreated fibers, with nominal volume proportion of 60/40 (CF/PP). It was employed a vacuum bag during the production. A Solab, model SL-11 press was used. The processing parameters were: 180 °C for 60 min and 2 MPa. From this procedure, 10 laminates with thickness around 3 mm were produced.

CARBON FIBER CHARACTERIZATION

Scanning electron microscopy (SEM) analysis was performed using a JEOL microscope, model JSM 5310. Carbon fiber roughness was obtained by atomic force microscopy (AFM) analysis using a Veeco Microscope, Digital Instruments, model Nanoscope V, the microscope operated in intermittent contact (tapping mode). For each sample, five areas of 16 μm^2 were scanned and the topography was characterized by quadratic mean roughness (Rq), automatically calculated by the software provided with the equipment. Raman spectra were obtained by the MicroRaman Renishaw System 2000 with Ar^+ laser (λ = 514.5 nm) as an excitation source. The radiation penetration depth is estimated to be approximately 5.0 μm.

COMPOSITES CHARACTERIZATION

Interlaminar shear strength analysis

The ILSS analyses were carried out using ten samples of each experimental condition. Their dimensions were approximately 18 mm × 6 mm × 3 mm, according to the ASTM D 2344/D2344M-00 (Standard ASTM 2007). This test determines the shear resistance of the composites. The samples were cut in a band saw machine, Universal RMF 400-S model and tested in a Shimadzu universal testing machine, Autograph AG-X model.

Dynamic mechanical analysis (DMA)

The DMA analysis was performed using a DMA equipment, model 6100, SII Nanotechnology Inc. The parameters were the following: temperature range: –100 to 100 °C; heat rate: 3 °C/min; amplitude: 10 μm; force: 2000 mN; atmosphere: N_2; gas flow: 100 mL/min; frequency: 1 Hz; dual cantilever. According to the ASTM D7028-07E1, the specimen dimensions were 10 mm × 50 mm × 3 mm.

RESULTS AND DISCUSSIONS

SCANNING ELECTRON MICROSCOPY (SEM)

This analysis aims to evaluate the influence of the DBD treatment on the CF surface morphology. Figure 1 shows SEM images of the untreated and treated carbon fibers.

Many axially oriented grooves on the untreated CF surface (Fig. 1a) were observed. This feature is due to the manufacturing process of carbon fiber (wet spinning) and subsequent application of polymeric coating (sizing) (Dai *et al*. 2011). It is also noted that after DBD treatments, there was agglomeration of particles on the CFs surface (Fig. 1b-e). This finding can be explained as a redeposition of sizing originally removed from the CF by etching during the DBD process. These results are similar to the ones in our previous work (Santos *et al*. 2013), where it was showed that the plain weave carbon fiber surface was modified after the DBD treatment due to plasma etching.

ATOMIC FORCE MICROSCOPY (AFM)

The AFM analysis was conducted to analyze the influence of DBD treatment on the CF roughness. Figure 2 shows the topography of the untreated and treated fibers.

AFM image of untreated fiber (Fig. 2a) showed an irregular surface (due to CF manufacturing), which corroborates the observations obtained by SEM analysis. After the DBD treatment, a modification in the CF surface morphology was observed (Fig. 2b-d).

Figure 3 shows the quadratic mean roughness (Rq, average from five different areas of the CFs) for each condition of treatment.

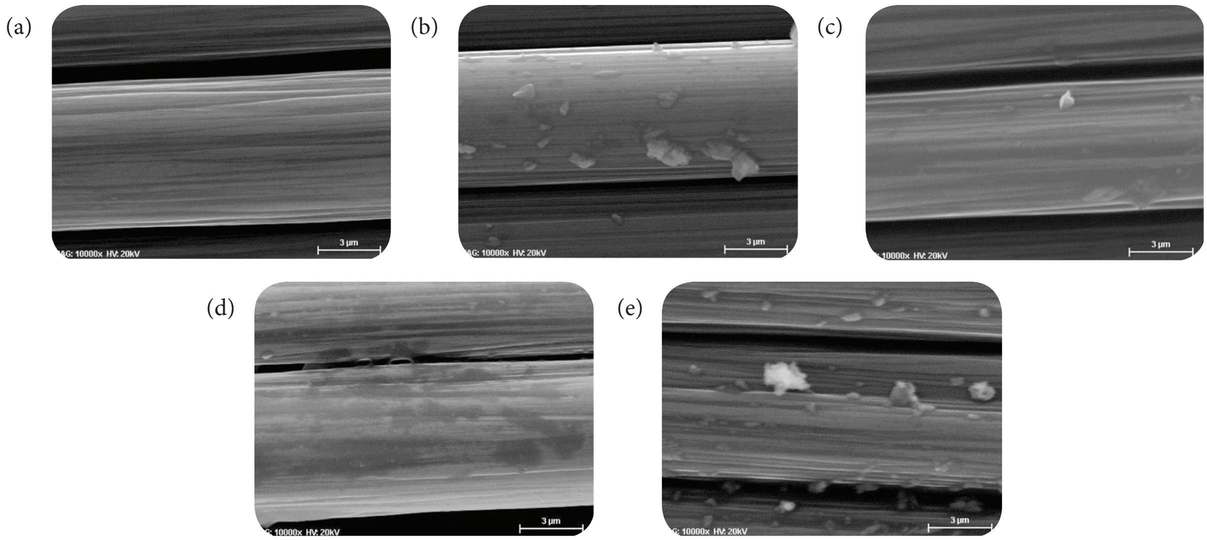

Figure 1. SEM images. Untreated CF (a) and treated CFs at: (b) 2.0 min; (c) 5.0 min; (d) 7.5 min; and (e) 10.0 min (10000x).

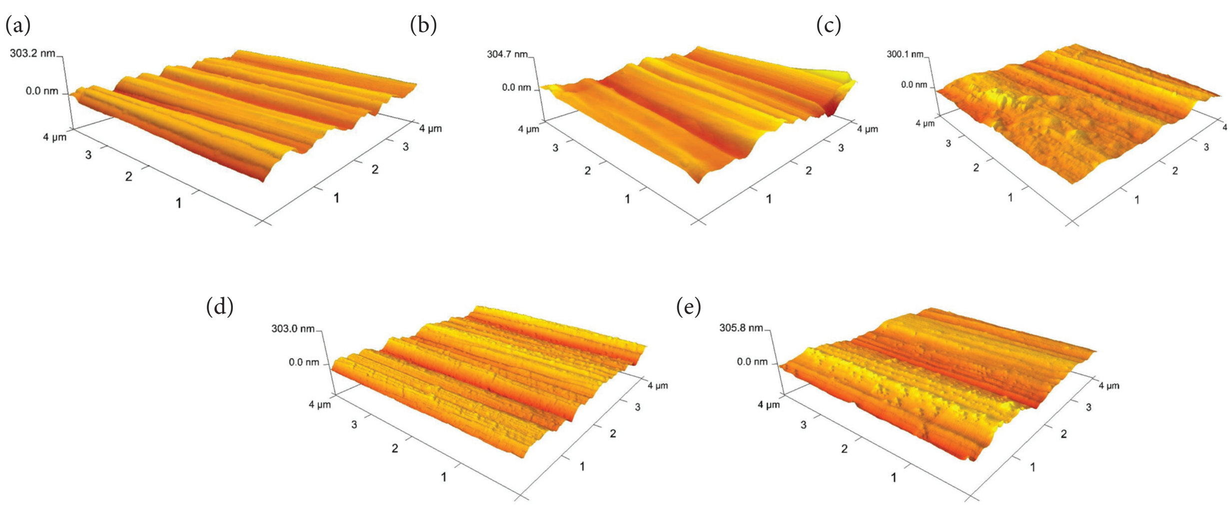

Figure 2. AFM images of the fibers. Untreated (a) and treated at: (b) 2.0 min; (c) 5.0 min; (d) 7.5 min; and (e) 10.0 min.

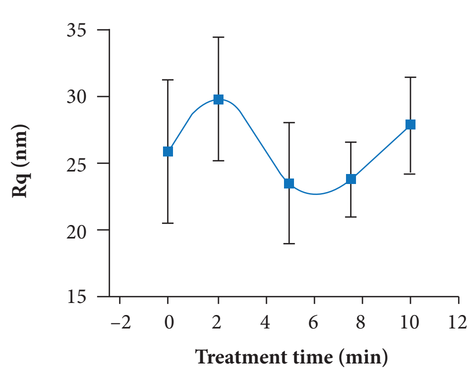

Figure 3. CF roughness as a function of treatment time.

It was observed that the roughness did not vary monotonically with the treatment time. The highest roughness was achieved for the sample treated for 2 min (~16% higher compared to the untreated CF (~25 nm). Variation of the surface morphology after DBD treatment was also noted in Santos *et al.* (2013) investigation for plain weave (1×1) braiding. Borooj *et al.* (2016) also inferred that the plasma treatment modified the surface roughness. Besides the introduction of polar groups on the sample surface, DBD treatment can also promote surface etching. These two processes happen simultaneously and are reported in previous studies (Montes-Morán and Young 2002; Sarraf *et al.* 2007). Therefore, the increase of CF roughness in the beginning is caused by etching on the initially smooth fiber surface. However, as the DBD treatment time was further increased the plasma attack preferentially occurs at the CF surface irregularities (peaks) and consequently the CFs roughness is decreased.

The roughness increase is favorable for better mechanical anchorage between CFs and polymeric matrix as shown in other studies (Santos *et al.* 2013; Li *et al.* 2009). Therefore, an increase of the adhesion in the composites interface is expected and interlaminar shear tests results will be presented for confirmation.

DYNAMICAL MECHANICAL ANALYSIS

Figure 4 shows the storage modulus for all composites (untreated and treated); this modulus refers to the stiffness of a viscoelastic material (Ashori *et al.* 2016). It can be noticed that the modulus tend to decrease for the treated samples, indicating that the treatment reduced the stiffness of the material. However, this decrease is very small, showing that the plasma treatment does not affect significantly this feature of the material.

Figure 5 shows the glass temperature as a function of treatment time. It can be seen that the glass transition temperature (Tg) did not modify significantly. In other words, the treatment did not affect the operating temperature of the material.

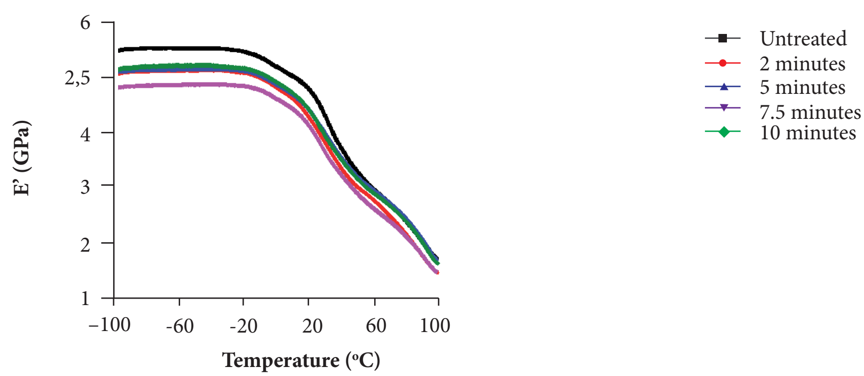

Figure 4. Storage modulus for all composites.

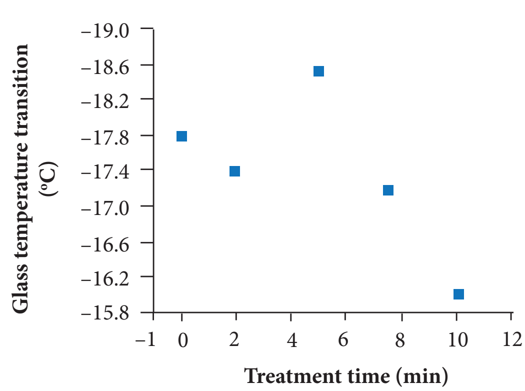

Figure 5. Glass temperature *versus* treatment time.

RAMAN SPECTROSCOPY

Raman spectroscopy analysis has great application to detect modifications in the crystalline structure of carbon materials (Montes-Morán and Young 2002; Ren *et al.* 2015). Raman spectroscopy was performed to provide useful information about the presence of phases and changes in the CF structure after the plasma treatment.

Figure 6 shows the Raman spectra of the untreated and treated fibers during 2.0, 5.0, 7.5 and 10.0 minutes. All spectra exhibited the characteristic D (disordered) and G (ordered or graphitic) bands of carbonaceous materials. For all samples the D band was located at approximately 1369 cm^{-1} and the G band at 1598 cm^{-1}. It can be also observed that there was no change in the CF structure after treatment. To support this statement, the I_D/I_G ratios were calculated. The D and G peaks from Fig. 5 were fitted by Lorentzian curves and the I_D/I_G ratios were calculated using the area under D and G bands and the results are presented in Fig.7.

Each point of the curve in Fig. 7 is an average of five ratios for each condition of treatment. It can be noticed in Fig. 7 that (within the measurements uncertainty) there was no significant change in the I_D/I_G ratios after the treatment. Thus, it can be concluded that the DBD treatment does not alter the CF structure, as inferred by other authors in (Li *et al.* 2009).

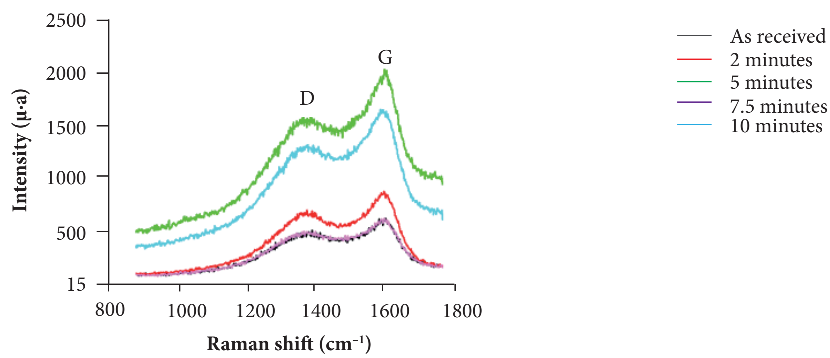

Figure 6. Raman spectra of the untreated and treated CFs.

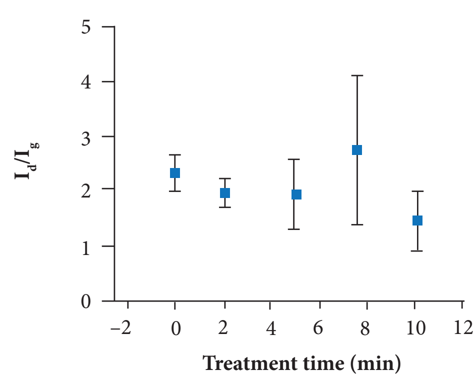

Figure 7. ID/IG ratios as a function of DBD treatment times.

INTERLAMINAR SHEAR TEST (ILSS)

This test was performed in order to examine the CF/PP composite shear strength. Figure 8 shows the CF/PP composite shear strength values as a function of the treatment time.

For all treated samples, an increase in the shear strength of about 70% compared to the untreated fiber (2.9 MPa) was observed. This increase can be explained by better adhesion in the CF/PP interface. This fact can be attributed to the increase of the CF surface roughness that provides an improvement of the mechanical anchorage in the composites interface. Furthermore, a better chemical interaction between the treated CF (containing oxygen polar groups on its surface) and the polymeric matrix can occur. This observation was inferred in our previous work (Santos *et al.* 2013) and in other studies (Guo *et al.* 2010; Zhang *et al.* 2011; Li *et al.* 2009). It is well known that the DBD treatment introduces polar species (containing oxygen) on the surface of various materials, such as polymers (Oliveira *et al.* 2014), aramid fiber (Xi *et al.*, 2008), aluminum (Zhang *et al.* 2016), and several studies have been found with carbon fiber (Luo *et al.,* 2014). Some authors (Oliveira *et al.*, 2014) treated the polymer PA66 by DBD processing and used the XPS and FTIR techniques to analyze the samples. The XPS results showed an increase of oxygen and nitrogen concentration for treated polymer about 74% and 17%, respectively, compared to the untreated sample. The O/C and N/C ratios were also increased by approximately 113% and 115%, respectively. In addition, Zhang *et al.* (2016) treated aluminum by DBD plasma and observed that the material became more hydrophilic due to a high percentage of polar groups detected by XPS analysis.

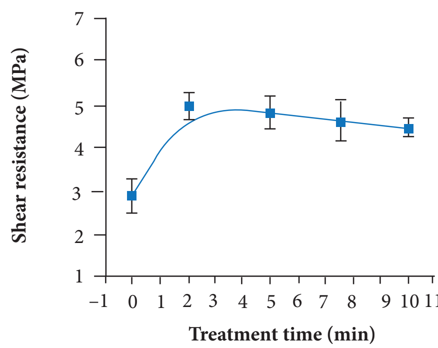

Figure 8. Shear strength as a function of the treatment time.

In Xi *et al.* (2008), an addition of polar groups on the aramid fibers by DBD treatment was noticed and Luo *et al.* (2014) used the DBD to treat carbon fibers. The XPS results presented an insertion of O–C=O groups on the carbon fiber surface after the treatments. Therefore, it can be concluded that DBD processes is quite efficient for introducing active species in various materials and more specifically on carbon fiber.

It was also noted that the shear resistance slightly decreased with the treatment time due to the plasma etching on the CFs surface. This behavior is in agreement with the one observed in Santos *et al.* (2013), where the shear resistance enhancement reached a maximum of 67% for the CFs treated during 5 minutes. In this work, the shear resistance improvement was around 70% for all treated samples. Therefore, different CFs braiding does not affect the adhesion in the composites interface. This fact was also inferred by Paiva *et al.* (2005).

CONCLUSIONS

Twill weave fabric CFs were treated by air-DBD plasma. After this, the carbon fibers, as well as the produced composites from them, were characterized and the results were compared with those obtained for the plain weave fabric. SEM and AFM images presented some surface morphological modifications for both CF fabrics. AFM results showed an increase of the carbon fibers roughness for shorter treatment times (2.0-5.0 min). Raman spectra showed no significant changes in the crystalline structure of both fabrics, indicating that the treatment effect is limited to the carbon fiber surface. Finally, the ILSS results evidenced that different braiding of CFs does not affect the adhesion in the composites interface. The enhancement of shear strength of composites was approximately 67-70% higher than the one obtained with the untreated carbon fibers. Moreover, the DMA results show that treatment did not change the working temperature of the material. DBD treatment of CFs arises as an efficient method for enhancement of the adhesion in the composites interface and, consequently, composites with higher shear strength can be obtained.

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AUTHOR'S CONTRIBUTION

Conceptualization, Silva LLG; Methodology, Vidal DCSM, Santos AL, Silva LLG and Kostov KG; Investigation, Vidal DCSM, Santos AL, Silva LLG and Kostov KG; Writing – Original Draft, Vidal DCSM and Santos AL; Writing – Review & Editing, Vidal DCSM, Santos AL, Silva LLG and Kostov KG; Funding Acquisition, Silva LLG and Kostov KG; Resources, Silva LLG and Kostov KG; Supervision, Silva LLG.

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