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Influence of UV Radiation on the Physical-chemical and Mechanical Properties of Banana Fiber

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Surface treatments done in banana fibers (BFs) can generate significant superficial structural changes enabling the production of mechanically stronger composites. In this way, the objective of this study was to evaluate the physicochemical and mechanical properties of banana fibers of Prata specie from inner and outer leaf sheaths, when irradiated with UV light (λ_{max} = 400 nm) during 7 (UV7) and 15 (UV15) days. Structural and microstructural characterizations for non- and irradiated fibers were performed by, FT-IR spectroscopy and Scanning Electron Microscopy (SEM), which showed the influence of UV irradiation on BFs surface and chemical structure. The E_A involved in the thermal degradation process of *In Natura* fiber (188.2 kJ.mol^{-1}) was obtained using Differential Thermal Analysis (DTA/TG). The results obtained from mechanical characterization showed that the UV7 fibers presented significant improvement in tensile strength (89.77 MPa) and elastic modulus (238.94 MPa) as compared to tensile strength (69.99 MPa) and elastic modulus (87.40 MPa) of *In Natura* fibers. Statistical analysis using two-way ANOVA has showed that there were no differences between mechanical properties of BFs from inner to outer leaf sheaths. UV radiation has proved to be a clean method for BF surface treatment, which can improve the mechanical properties of composites based on these fibers.

Keywords: *composites, banana fiber, UV radiation*

1. Introduction

Many authors[1,2] considered the incorporation of banana fiber (BF) as a reinforcing agent in composites, like a way to minimize the accumulation of waste. The BFs similar to other natural fibers are renewable, biodegradable and cheaper than synthetic fibers such as carbon, aramid and glass fibers. Some authors mention that the lignocellulosic fibers have low abrasiveness, which causes less wear on the processing equipment, helping the molding[3].

New approaches in the properties modification of plastics have been observed due to growing concern about the environment and the constant search for the use of fillers in polymeric materials[4]. The study of molecular structure of the lignocellulosic fibers enables the knowledge of their chemical structure, and therefore, justifies a better use as filler in a polymer matrix, giving rise to composites with improved properties[5].

According to Kuruvilla et al.[6], polymers, thermosets and thermoplastics, have different levels of interaction with the natural fibers due to differences in their chemical structures. According to Guimarães et al.[7], the existence of interfacial chemical bonding between fibers and matrix may be considered as the main factor for promoting wetting and compatibility. The UV radiation is a potential energy source able to promote photochemical reactions in the molecular structures of natural fibers changing its mechanical properties, and simultaneously, acts as a clean method to modify surface of natural fibers. However, the study of photochemical reactions in organic materials is an area of intense scientific research[8-11]. Despite its relevance, the degradation mechanisms induced by photons in polymers or natural fibers, which trigger changes in their physical and chemical properties are not yet fully understood[12].

The purpose of this study is to reveal the physico-chemical changes in the molecular structure of BF irradiated by ultraviolet light (UV) and, therefore, changes in their mechanical, structural and microstructural characteristics. In this way, it was also evaluated the changes caused by irradiation on the BF surface, and the way that it may help in the interface interaction between fiber/polymeric matrix by mechanical anchoring.

2. Experimental Procedures

The extraction of BFs was conducted in water at a pressure of 1.2 atm and temperature of 120 ± 5 °C, during 4 h. After, the BFs fed a pair of squeezing roller to remove excess of water and organic residues (or pulp). Further, the BFs were dried at 40 °C for 40 min. The samples have been selected according to the physical arrangement of the fibers in the pseudostem, to investigate possible differences in their mechanical properties. First, leaf sheaths of internal and external regions (inner and outer layers) of an adult pseudostem and ready to fruit gathering were selected (Figure 1). At this stage, the fibers were considered *In Natura*.

BFs were subjected to UV irradiation supported by a metallic frame to keep the fibers aligned during exposure.

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Thus, the metallic support ensures that irradiation acts homogeneously onto the surface of the fibers, as shown in Figure 2a. Quotas of the fibers were irradiated in a photobleaching chamber (Figure 2b) for 7 (UV7) and 15 days (UV15). The photobleaching chamber used in this study was build based on ASTM D5208-09[13].

Sample preparation and testing procedures used in the tensile test followed the parameters described in ASTM C1557-03[14]. The tensile test was conducted using an EMIC DL200 using a 500 kgf load cell and test speed of 0.5 mm/min at room temperature. The measurement of average diameter of samples, necessary for the calculation of the cross sectional area, was conducted in an optical microscope Zeiss Jenavert.

Two-way ANOVA analysis[15] was applied in order to verify the existence of significant differences among the data obtained from tensile test according to internal and external regions of pseudostem, with and without UV treatment, and the interaction of these two factors. Statistical analyzes were performed using the Minitab software tools.

To accomplish FT-IR analysis, *In Natura* and UV irradiated BFs were grinded and evaluated by spectrophotometer Perkin-Elmer Spectrum 100 in the wavenumber range of 650-4000 cm^{-1}.

The BFs samples were subjected to thermogravimetry analysis (TG) and differential thermal analysis (DTA) (Netzsch - STA449F3) to reveal temperatures and maximum rates of degradation of the material. The TG/DTA test was conducted using a heating rate of 5 °C.min^{-1} from 25°C to 800°C in air atmosphere. To study the activation energy (E_a) involved in the degradation process, the test was repeated changing the rate of heating to 10 °C.min^{-1} and 15 °C.min^{-1}.

The microstructural characterization of the BFs surface was evaluated by scanning electron microscope (SEM) (Zeiss EVO15MA).

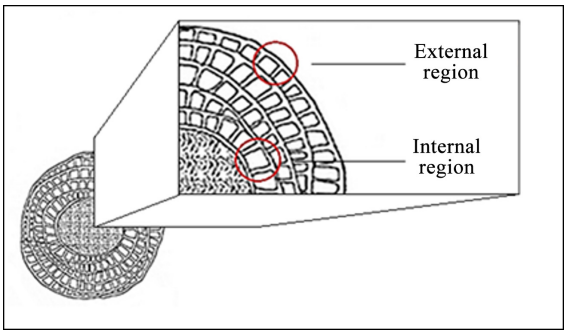

Figure 1. Leaf sheaths of internal and external regions (inner and outer layers) of an adult pseudostem.

3. Results and Discussions

3.1. Mechanical test

Table 1 summarizes results for the basic statistics relating to stress, elongation and E distributions values obtained from the tensile test. The table contains each average, number of samples, standard deviation, variance, p-value, type of distribution and the confidence interval for the mean, representing a reliability estimated in 95% of cases of probable values, and the test is again applied to a new sample[16].

Tensile strength and elastic modulus obtained to *In Natura* were 69.99 MPa and 87.40 MPa, respectively. Mukhopadhyay et al.[17] have reported values about 150 MPa, similar to those reported in this work. According to the statistical evaluation, the UV7 radiation showed an increase in average tensile (89.77 MPa) and E values (238.94 MPa) as compared to FBs untreated (*In Natura*). In contrast, the UV15 BFs showed loss of mechanical properties with reduced tensile strength. Similar observations have been made by Rahman & Khan[18] in their research with coir fiber irradiated by UV. Increase of tensile properties with increasing radiation doses could be due to the inter-crosslinking between the neighboring cellulose molecules occur under UV radiation and the decrease for longer periods of radiation could be due to the photo-degradation of cellulose backbone at higher UV doses. During photo-degradation there will be loss in strength due to primary bond breakage in the cellulose constituent. Thus, UV radiation was detrimental to BFs treated for long periods of exposure, but at manageable levels, it was able to increase the mechanical properties.

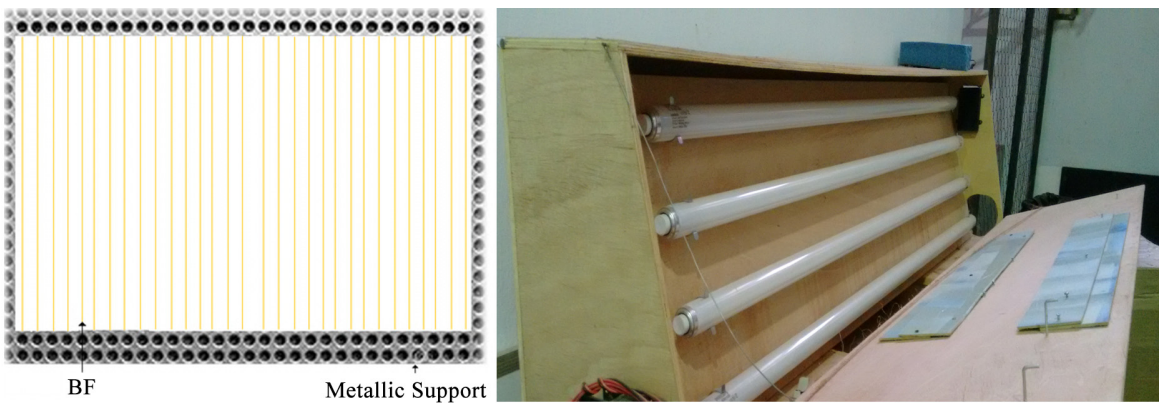

Figure 2. Apparatus used to apply UV radiation on BFs. (a) Representation of the metallic frame to keep the fibers aligned during exposure. (b) Photobleaching chamber build based on ASTM D5208-09[13]. Bibliographic source: Author.

Table 1. Basic statistics of the data distribution of tensile strength, deformation and E of the BFs.

Region	Treatment	Measures	Variable		
			Tensile strength (MPa)	Elongation (mm)	E (MPa)
External Fibers	*In Natura*	*Number of Samples*	30	30	30
		Average	69.99	0.83	87.40
		Standard Deviation	24.20	0.26	24.54
		Variance	585.57	0.06	602.25
		P-value	0.286	0.666	0.127
		Distribution Type	Normal	Normal	Normal
		Confidence Interval for the Mean (95%)	60.95-79.02	0.73-0.92	78.23-96.56
	UV7	*Number of Samples*	30	30	30
		Average	89.77	0.47	238.94
		Standard Deviation	36.14	0.28	108.22
		Variance	1306.42	0.08	11711.14
		P-value	0.731	0.154	0.036
		Distribution Type	Normal	Normal	Not Normal
		Confidence Interval for the Mean (95%)	76.27-103.26	0.37-0.58	198.53-279.35
	UV15	*Number of Samples*	30	30	30
		Average	44.29	0.32	162.02
		Standard Deviation	23.30	0.14	84.30
		Variance	542.983	0.02123	7106.59
		P-value	0.005	0.15	0.005
		Distribution Type	Not Normal	Normal	Normal
		Confidence Interval for the Mean (95%)	35.59-52.99	0.27-0.38	130.55-193.50
Internal Fibers	*In Natura*	*Number of Samples*	30	30	30
		Average	69.68	0.98	69.17
		Standard Deviation	20.80	0.23	20.02
		Variance	432.82	0.05	400.933
		P-value	0.901	0.074	0.508
		Distribution Type	Normal	Normal	Normal
		Confidence Interval for the Mean (95%)	61.91-77.45	0.89-1.06	61.70-76.65
	UV7	*Number of Samples*	30	30	30
		Average	89.04	0.61	121.01
		Standard Deviation	25.19	0.24	66.50
		Variance	634.825	0.055	4421,600
		P-value	0.289	0.244	0.005
		Distribution Type	Normal	Normal	Not Normal
		Confidence Interval for the Mean (95%)	79.63-98.45	0.52-0.69	96.18-145.84
	UV15	*Number of Samples*	30	30	30
		Average	44.47	0.37	122.66
		Standard Deviation	25.82	0.15	63.23
		Variance	666.45	0.02	3998.17
		P-value	0.005	0.024	0.005
		Distribution Type	Not Normal	Not Normal	Not Normal
		Confidence Interval for the Mean (95%)	34.87-54.12	0.32-0.43	99.05-146.28

Bibliographic source: Author.

Figure 3 illustrates the mechanical behavior of BFs according to pseudostem region (internal and external) and treatments applied (UV7 and UV15).

Table 2 refers to two-way ANOVA statistical analysis. Considering the p-value it should be accepted that there is sufficient evidence to ensure that the mean stress values differ between treatments, but did not differ directly in function of the pseudostem region.

3.2. Thermal characterization

Figure 4 shows that *In Natura* BFs exhibits three characteristic peaks of weight loss, which are associated, respectively, to the moisture loss, the degradation of cellulose (plus hemicellulose) and lignin loss[19]. According to Figures 5 and 6, the UV irradiation caused a change in thermal

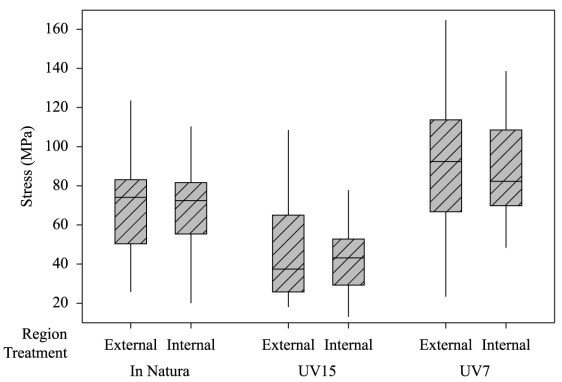

Figure 3. Boxplot of BFs tensile strength. Bibliographic source: Author.

Table 2. ANOVA statistical analysis for mechanical behavior of BFs.

Source	Degree of freedom	Sum of Squares	Mean Square	F	P-value
Region	1	1963	1962.7	1.84	0.177
Treatment	3	153241	51080.4	47.8	0.000
Interaction	3	6497	2165.6	2.03	0.111
Error	232	247897	1068.5		
Total	239	409597			

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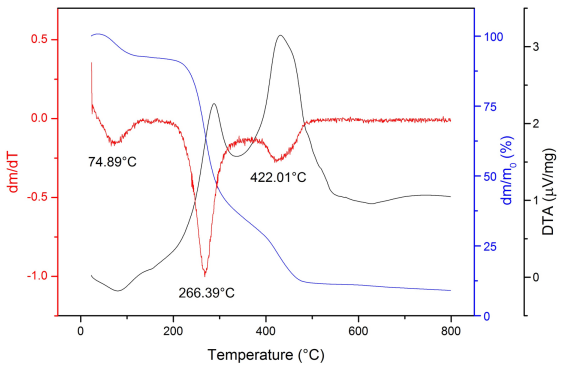

Figure 4. TG/DTG/DTA curves of *In Natura* BF. Bibliographic source: Author.

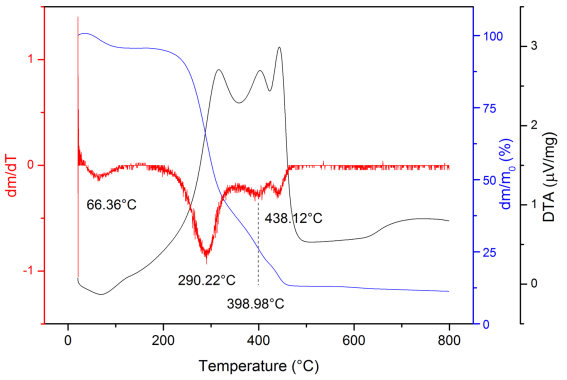

Figure 5. TG/DTG/DTA curves of UV7 BF. Bibliographic source: Author.

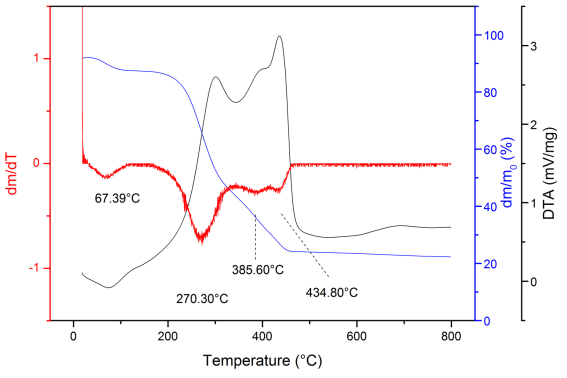

Figure 6. TG/DTG/DTA curves of UV15 BF. Bibliographic source: Author.

Table 3. Characteristic temperature of weight loss.

Samples 1.1.	Temperature (°C)			
	Endothermic peak	*Exothermic peak*		
In Natura	74.89	266.39	422.01	-
UV7	66.36	290.22	393.98	438.12
UV15	67.39	270.30	385.60	434.80

Bibliographic source: Author.

behavior of the BFs, as evidenced by the formation of a new exothermic peak at ~400 °C. The Table 3 summarizes the temperatures related to weight loss and the character of the endo- or exothermic reaction degradation rate of 5 °C/min.

The E_a involved in the thermal degradation process was obtained using Flynn-Wall-Ozawa method (FWO)[20] that TG/DTA analyzes were undertaken using heating rates of 5, 10 and 15 °C.min^{-1}. Figure 7 refers to the test results. The Table 4 summarizes the data necessary to calculate E_a. The Table contains, for each heating rate adopted, the temperature ranges in which the weight loss rate is maximum (or volatilization, according Flynn), the ratio $\frac{d\alpha}{dt}(or\,k)$ and inverse temperature $\left(\frac{1}{T}\right)$. Linear regression of the curve $\ln k$ vs $\left(\frac{1}{T}\right)$, as shown in Figure 8, provides the fit between the discrete points and the line generated by linearization provides the slope, which equals the ratio $\left(\frac{-Ea}{R}\right)$. The value of the gas constant (R) is 8.3144621 $\frac{J}{mol.K}$ [21]. The E_a of the thermal process involved in *In Natura* BFs showed a value of 188.2 kJ.mol^{-1}. This value is similar to the activation energy obtained by other researchers studying for bagasse ranged from 87 to 225 kJ/mol[22], whereas it ranged between 149 and 210 kJ/mol for banana fibers[23]. In general, the decomposition of plant fibers occurs in two to four stages of weight loss at 25 °C to 800 °C, depending on the types and sources of natural fibers[24]. Sathasivam & Haris[23] reported the effect of temperature on chemical modification of banana pseudostem fibers. They found that the banana fiber started to degrade at 271 °C with maximum degradation at 320 °C.

3.3. FT-IR spectroscopy

Figure 9 shows FT-IR spectra of of In Natura, UV7 and UV15 BFs. It was noted similarity to all spectra of the samples are nearly the same transmittance in the range of bands between 2000 and 4000 cm^{-1} (data not shown). According to Kang et al.[25], the hydrocarbons have functional groups

Table 4. Determination of variables to calculate the E_a.

Heating Rate (°C/min)	Temperature Range (°C)			*Maximum fraction weight loss*	ln k	1/T (K^{-1})
	On Set	*Maximum weight loss*	*End*			
5	250	266.39	270	0.86	–0.15	0.0018416
10	260	275.8	290	0.93	–0.07	0.0017762
15	280	280.93	290	0.96	–0.04	0.0017762

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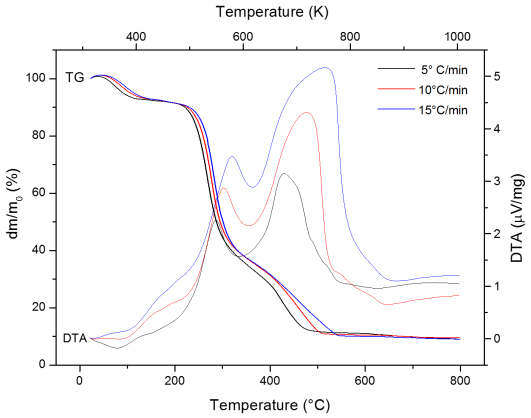

Figure 7. TG/DTA curves of *In Natura* BF at different heating rates. Bibliographic source: Author.

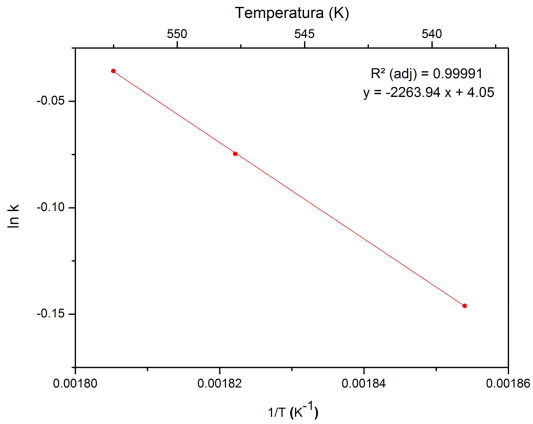

Figure 8. Arrhenius plot of ln k *vs* 1/T for estimation of thermodynamic parameters. Bibliographic source: Author.

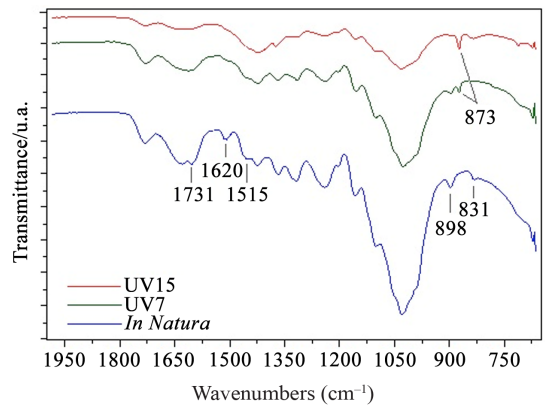

Figure 9. FT-IR spectra of *In Natura*, UV7 and UV15 BFs. Bibliographic source: Author.

–OH (3500-3300 cm^{-1}) as well as the type of aliphatic bond C-H (3000-2800 cm^{-1}), the latter has no aromatic rings[26]. The bands with the wavenumber 3342 cm^{-1} and 2915 cm^{-1} were identified in all treatments and correspond to the functional groups mentioned earlier. Therefore, the band of 3342 cm^{-1} is characterized by vibrational stretching –OH group present in the cellulose structure and is commonly found in lignocellulosic fibers. The other band of 2915 cm^{-1} is attributed to the stretching of the C-H bond of methyl group (CH_2) and methylene (CH_3)[27]. With reference to the vibrational energy spectrum of the *In Natura* fibers, the main structural changes were identified in the peaks at wavenumbers below 2000 cm^{-1}.

The FT-IR spectrum of the *In Natura* fibers showed bands at 1731 cm^{-1}, 1620 cm^{-1} and 1515 cm^{-1} that were not detected in the spectra of UV irradiated fibers. The band at 1731 cm^{-1} is assigned to the stretching vibration of C=O unconjugated groups of hemicellulose (vibration of aliphatic carboxylic acids and ketones)[7]. According to Sinha & Rout[28], the disappearance of this peak is an indication of partial removal of hemicellulose.

The band at 1620 cm^{-1} is associated with the conjugated C=O groups present in the structure of lignin[7]. Some authors also mention that the 1620 and 1515 cm^{-1} band are associated with C-C bond[29]. The peak at 898 cm^{-1} and 831 cm^{-1} are observed in the spectrum of the *In Natura* fibers. They are assigned to the stretching of C-O-C groups of the glycosidic bonds (β-1→4) and are designated as "amorphous" transmittance bands[30].

The UV irradiated fibers spectrum, there is a characteristic peak at 873 cm^{-1}, which has not previously seen in the spectrum of the *In Natura* fibers. The band at 873 cm^{-1} indicating the presence of out of plane aromatic rings C-H of bending type vibration and indicate that the aromatization occurred[31]. This assertion is confirmed by Schimidt[32], which mentions the C-H deformations of the benzene rings are identified in peaks near to 874 cm^{-1}.

3.4. SEM

Figure 10 presents the micrographs of the *In Natura* and irradiated fibers. It can be seen that the samples irradiated with UV reveals the occurrence of severe surface degradation, not present in *In Natura* fibers (a) and (b). The surface of the fibers showed wide longitudinal cracks and flaking of the microfibrils, providing a rough relief. Thus, the effective surface area was expanded as a result of the degradation caused by the photochemical process.

The micrograph of the fibers UV7 reveals defibrillation across the surface of the microfibrils in the contours identified as seen in Figure 10c, d. At some points, defibrillation has

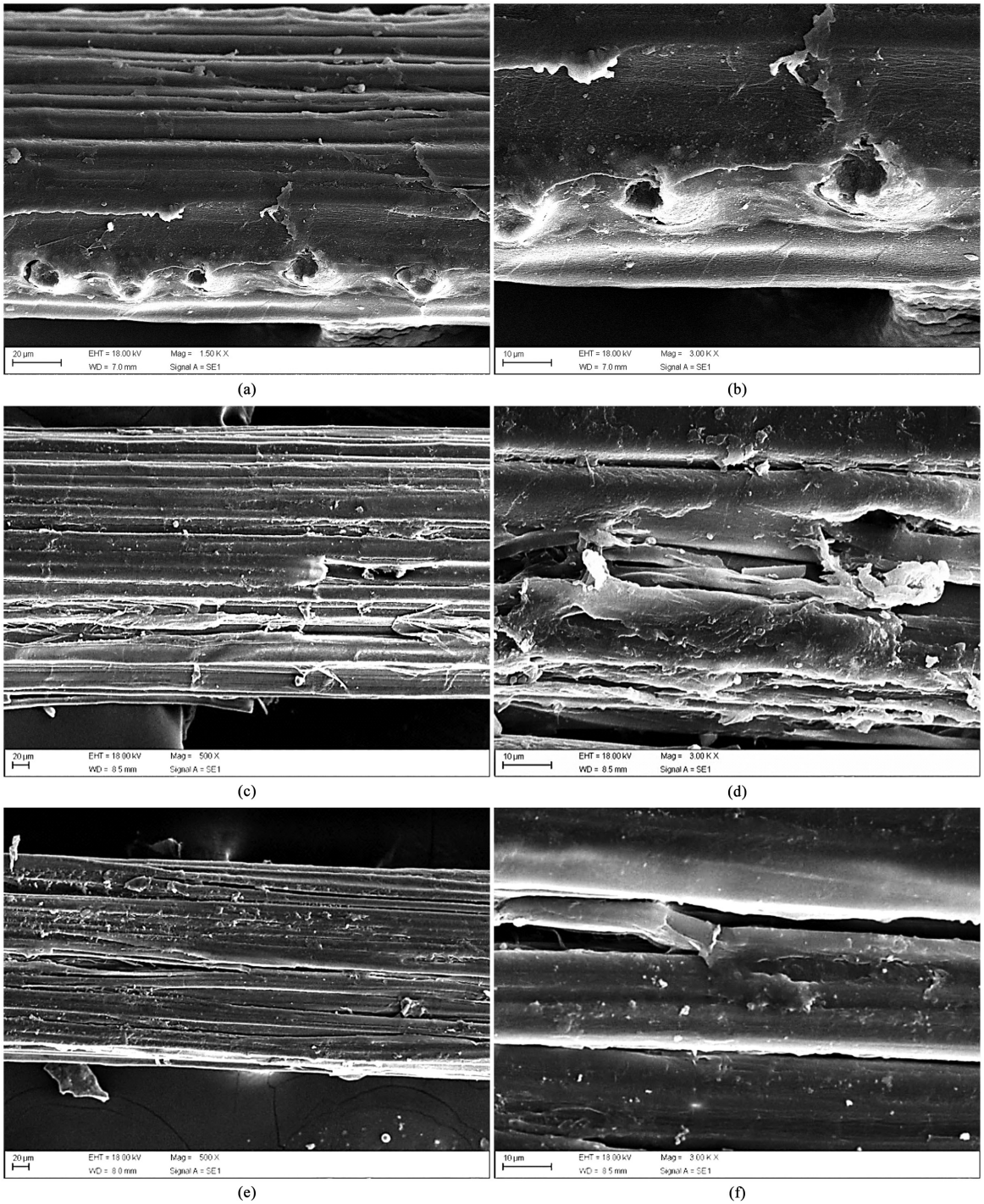

Figure 10. SEM images of BFs. (a) *In Natura*, (b) *In Natura* surface detail, (c) UV7, (d) UV7 surface detail, (e) UV15, (f) UV15 surface detail. Bibliographic source: Author.

occurred in depth reaching the inner parts of the fiber. The SEM analysis of UV15 fibers revealed a similar result found in UV7 fibers, but with more intense levels of degradation as shown in Figure 10e, f. Cracks in UV15 BFs assumed greater size than UV7 BFs and separated the most peripheral microfibrils. The micrograph shows the existence of longitudinal cracks in the entire length of the UV15 fibers and cracks in depth.

The incidence of UV irradiation on the BFs was able to modify its surface, making it rougher and leading processes defibrillation. The separation of the microfibrils allows creating spaces that could be filled by polymeric matrix providing better intrusion. The fiber morphology can help the mechanical and chemical anchoring adhesion between the fiber and the matrix, by increasing the effective surface

area of the interfibrillar regions, and the structural changes resulting from the active photochemical process.

5. Conclusions

The UV radiation caused changes in the chemical structure and mechanical properties of BFs. Statistical analysis using two-way ANOVA of tensile test, showed that the UV radiation was able to significantly increase the tensile strength of the fibers, when applied in admissible levels. The FB irradiated for 7 days showed higher tensile strength (89.77 MPa) and E values (238.94 MPa) than FB *In Natura* that presented about 69.99 MPa to tensile strength and 87.40 MPa to E. However, this significant improvement in the mechanical properties of FB irradiated for 15 days was not observed and FB UV15 presented lower values to tensile strength (about 44 MPa). Also, it was noted the reduction in elastic deformation to both irradiated fibers, UV7 and UV15. The tensile test indicated no significant difference between the mechanical behavior of fibers from inner to outer leaf sheaths, increasing the number of fibers that can be extracted.

The results of thermal analysis tests for *In Natura* and UV BFs presented different outcomes, but the E_a involved in the thermal degradation process of In Natura fiber (188.2 kJ.mol^{-1}) was similar to literature reports. Fibers irradiated by UV presented an atypical peak in DTA, which represent a new exothermic reaction, which could be associated to a reorganization of the BF chemical structure after UV radiation. It was confirmed by FT-IR analysis, which indicated that the photochemical reactions caused by the incidence of photons in BFs led to changes in the characteristic bands of transmittance of the structure of BFs. These changes are associated with the formation of aromatics structures by cyclization caused by the breaking of the weakest bond in the structure of BF and that may assist the chemical anchoring with the polymeric matrix.

UV radiation has proved to be a clean and cost effective method for the mechanical improvement of BFs, aiming at the production of composites with better mechanical and thermal properties. The study may also help scientific production based on photochemical processes and phenomena in lignocellulosic fibers.

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