Characterization of *Luffa cylindrica* Fibers and the Effect of Water Aging on the Mechanical Properties of Its Composite with Polyester

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ABSTRACT: *Luffa cylindrica* of Turkey's Aegean Region was evaluated in terms of fiber characterization and the mechanical properties of its composite with polyester. Characterization of *Luffa cylindrica* was carried out by Fourier transform infrared spectrophotometer, X-ray photoelectron spectroscopy (XPS), scanning electron microscopy, and thermogravimetric analysis. Cellulose, hemicellulose, and lignin contents of *Luffa cylindirica* were also determined. Deconvolutions of XPS data enable determining the distributions of functional groups on the surface of *Luffa cylindrica*. *Luffa cylindrica*-reinforced polyester composite was subjected to water aging under a steam of seawater containing 5% sodium chloride for 170 h at 50°C. After water aging, tensile strength, flexural strength, interlaminar shear strength, and tensile elongation at break values of the composite decreased ~ 28%, 24%, 45%, and 31%, respectively. However, tensile modulus and flexural modulus did not change significantly. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 123: 2330–2337, 2012

Key words: natural fiber; polymer composite; material characterization; water aging

INTRODUCTION

The glass-reinforced plastics are the most widely accepted composite material in marine field because of the fact that it is the optimum choice in terms of durability, workability, and cost.[1] In the recent years, composite materials have been done by using natural fibers in the place of synthetic fibers as reinforcement. The attractiveness of natural fibers as an alternative reinforcement comes from its relatively high specific strength and specific stiffness and its good eco-friendly performance when compared with traditional fibers such as glass fibers.[2] When natural fibers, derived from annually renewable resources, are used as reinforcement in both thermoplastics and thermoset matrix composites, positive benefits are observed in terms of biodegradability, combustibility, light weight, nontoxicity, decreased environmental pollution, low cost, and recyclability.[3] From this point of view, natural fiber-reinforced polymer composites may be partly alternative to glass fiber-reinforced polymer composites in marine industries.

In this respect, this study has been concentrated on the use of *Luffa cylindrica* fiber that is easily available because of its natural distribution along the Mediterranean coast of Turkey. *Luffa cylindrical*, which is a plant of tropical origin, is widely available in the wet and warm climates of the world.[4] *Luffa cylindrica* are obtained from a subtropical plant of the cucurbitaceous family, which produces a fruit with a fibrous vascular system. Their sizes vary in relation to the areas they grow in ranging from 15 cm to 1 m or even more than 1 m in certain kinds (Fig. 1).

The performance of composite materials under prolonged immersion to water and exposure to wind, rain, and sun is better than other usual construction materials.[5] However, these materials have some drawbacks due to deterioration of structural properties that can lead to unexpected structural failures. These aspects should also be taken into consideration during the design process for a marine structure.[6] Especially, water absorption behavior of natural fiber polymer composites is one of the important characteristics that determine their end-use applications. Water absorption may cause deterioration in some of the properties and needs to be taken into consideration when selecting applications.[7,8]

Some studies claim that water absorption could strongly affect the behavior of *Luffa cylindrica*/polyester

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Figure 1 *Luffa cylindrica.*

composites.[9] The scope of this study was decided to establish the results of the mechanical tests performed to determine the influence of accelerated aging of *Luffa cylindrica* fiber-reinforced polyester composites in seawater environment. In this work, in order to get the values for degradation in seawater, one set of specimens has been conditioned in a 5% solution of NaCl for a period of 170 h at a constant temperature of 50°C. Additionally, the results of X-ray photoelectron spectroscopy (XPS), Fourier transform infrared spectrophotometer (FTIR), thermogravimetric analysis (TGA), and cellulose content of *Luffa cylindrica* fibers were mentioned in detail in order to present scientific data on their characteristic thermal and chemical properties.

EXPERIMENTAL

Materials

Luffa cylindrica from the Aegean Region of Turkey was supplied from a local shop in İzmir. To separate the inner fiber core from outer mat core, the as-received sponge-gourd was cut carefully. Only the outer mat core of *Luffa* was used in this study. Sodium hydroxide (NaOH) from Merck Corp. was used for the alkaline treatment before fabrication of the composite. It is known that the alkalization treatment removes impurities and increases the fiber surface adhesion characteristic with the resin.[10] The removal of surface impurities on plant fibers is advantageous in fiber-matrix adhesion, as it facilitates both mechanical interlocking and the bonding reaction.[11] *Luffa* fibers were dipped in 2% NaOH solution for about 60 min. Treated *Luffa* fibers were washed with distilled water until a neutral pH was reached and then dried at 60°C for 24 h.

Polipol polyester 383-T (specific gravity: 1.11 g/cm^3, viscosity Brookfield: 950 cP), which is isophthalic acid-type resin, was used as matrix material in fabricating the composite. The resin was mixed before fabricating the composite with the catalyst cobalt octoate (0.35 pph, of a 41% solution in white spirit), the retarder 2.4-pentanedione (0.10 pph), and methylethylketone peroxide (2.2 pph, of a 40% dimethyl phthalate solution) as hardener. The mechanical properties of polyester resin obtained from the supplier (Poliya Polyester) are as follows: tensile strength: 62 MPa, tensile modulus: 3224 MPa, flexural strength: 136 MPa, and flexural modulus: 3734 MPa.

Fiber characterization methods

Cellulose contents of fibers

Cellulose, hemicellulose, and lignin contents were determined by Van Soest Detergent Methods.[12–14] Ash content of *Luffa cylindrica* was determined according to Standard Test Method for Ash in Biomass ASTM E1755-01(2007).

FTIR analysis

A Fourier transform infrared spectrophotometer (FTIR; Perkin Elmer Spectrum BX-II) was used for the determination of functional groups. One milligram of the samples was ground into powder with high-purity infrared-grade KBr powder (100 mg) and pressed into a pellet for measurement. Each spectrum was recorded in the range of 400–4000 cm^{-1} with a resolution of 2 cm^{-1}.

XPS analysis

X-ray photoelectron spectroscopy (XPS) was used to determine the surface elemental compositions of the *Luffa cylindrica* fibers. The XPS spectra were carried out with a Specs ESCA instrument (Germany), equipped with a nonmonochromatic Mg Kα radiation source at a power of 200 W (10 kV and 10 mA) and EA 200 hemispherical electrostatic energy analyzer. The base pressure in the chamber was about 10^{-9} – 10^{-10} torr. The distributions of functional groups in the C1s peak were determined by fitting the curves with Gauss–Lorentz functions.

Thermogravimetric analysis

Thermogravimetric analysis (TGA) was also performed with Perkin-Elmer Diamond TG/DTA Analyzer in Dokuz Eylül University/Turkey. The analyses were carried out in aluminum pans under a dynamic nitrogen atmosphere in the temperature range 25–600°C at a heating rate of 20°C/min.

Scanning electron microscopy

The surface morphologies of *Luffa cylindrica* were observed using a scanning electron microscope (SEM) at an accelerating voltage of 10 kV. SEM photographs were taken at different magnifications (in the range of 25× and 2500×).

Fiber tensile test

To determine the tensile strength of *Luffa cylindrica* fiber, single fiber tensile tests were conducted by using Shimadzu AUTOGRAPH AG-G Series universal testing machine at contact speed 0.1 mm/min. The single fiber was mounted on cardboard end tabs via a quick-setting polyester adhesive. The samples were mounted, such that each specimen had a gauge length of 20 mm.

Composite manufacturing

A mixture of the unsaturated polyester resin and the hardener was applied onto the *Luffa cylindrica* fiber surface by hand lay-up technique (volume fraction: 28%). Two layers were added successively in order to produce a 6.5-mm-thick composite laminate. The layers were compressed in a mold (30 cm × 40 cm) at a pressure of 110 bars, which was applied at room temperature for 90 min.

Water aging

The effect of seawater on the mechanical properties of *Luffa cylindrica*-reinforced polyester composite was studied by exposing it to a steam of 5% sodium chloride (NaCl) solution for a period of 170 h at a constant temperature of 50°C in a conditioner at the mechanical laboratory of Turkish Standards Institution. Specimens were properly placed in the testing machine so as to be exposed to seawater from all directions homogeneously. After the composite samples were treated by seawater steam at a temperature of 50°C for a period of 170 h, they were let to dry at ambient temperature until their humidity leveled back to the initial value.

Mechanical testing

Tensile tests were carried out according to ASTM D-3039 standard using a Shimadzu Autograph AG-IS Series universal testing machine equipped with a 5 kN load cell. The cross-head speed was held constant at 2 mm/min. Flexural strength and modulus of the composite were determined by a three-point bending test according to ASTM D-790. Preferred specimen span-to-depth ratio was 16 : 1 while the cross-head was moved at a constant speed of 1.3 mm/min. Short beam shear tests were performed following ASTM D-2344 to determine the apparent interlaminar shear strength (ILSS) of the composite. The test fixture was mounted in a 5 kN capacity, screw-driven load frame. Shimadzu Autograph AG-IS Series universal testing machine was operated at a constant cross-head speed of 1.3 mm/min. Support span/sample thickness ratio of 5 : 1 was maintained throughout the tests. The mechanical tests were carried out at room temperature and relative humidity of 50%.

TABLE I
Component of *Luffa cylindirica*

Component	Content (%)
Cellulose	63.0
Hemicellulose	14.4
Lignin	1.6
Ash	0.9
Others	20.1

Statistical analysis

The data regarding mechanical tests were expressed as mean ± standard deviation ($n = 6$) and analyzed using Student's t-test for the calculation of significance level of the data. Differences were taken into consideration statistically significant at $P \leq 0.05$.[15]

SEM observations

The fracture surfaces of tensile-tested specimens were examined using the scanning electron microscope (JEOL JSM 6060) in the secondary electron mode. The samples were coated with a thin layer of metallic gold in an automatic sputter coater (Polaran SC7620) before examination by SEM.

RESULTS AND DISCUSSION

Fiber characterization methods

Cellulose contents of fibers

Cellulose, hemicellulose, and lignin contents of *Luffa cylindrica* were presented in Table I. Tanobe et al.[16] examined *Luffa cylindirica*, which was collected from Southeast region of Brazil. They obtained that the contents of holocellulose, α-cellulose, hemicelluloses, and lignin were 82.4%, 63%, 19.4%, and 11.2%,

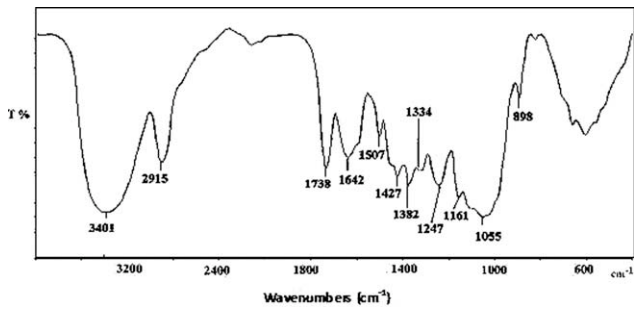

Figure 2 Fourier transform infrared spectrum for *Luffa cylindrica* fibers.

respectively. In this study, the contents of holocellulose, α-cellulose, hemicelluloses, and lignin were obtained to be 77.4%, 63.0%, 14.4%, and 1.6%, respectively. There is a considerable difference between the lignin contents of two different *Luffa cylindrica*.[16] However, the contents of α-cellulose are the same in this study and in the study of Tanobe et al.[16]

FTIR analysis

Fourier transform infrared spectrophotometer (FTIR) spectrum of *Luffa cylindrica* was presented in Figure 2. Peak positions of untreated *Luffa cylindrica* were summarized in Table II. Except for small shifts in the functional groups, the obtained functional groups on the *Luffa cylindrica* in this study are similar to the findings in the study of Tanobe.[16]

XPS analysis

Atomic concentrations on the surface of *Luffa cylindrica* were obtained to be C: 59.6, O: 36.1, and N: 4.3. O/C ratio for *Luffa cylindrica* is 0.61. To determine the amount of functional groups, deconvolution of **C1s** peak was conducted. **C1s** core level spectra were deconvoluted into three distinct components, as presented in Figure 3. The binding energies (eV)

TABLE II
Peak Positions of Untreated *Luffa cylindrica*

Wave number (cm^{-1})	Assignment
3401	OH stretching
2915	C—H stretching
1738	C=O stretching of acetyl or carboxylic acid
1642	Absorbed H_2O
1507	Aromatic bending C—H (ring)
1427	CH_3 and CH_2 sym. Bending pyran ring
1382	C—H bending
1161	Antisym. bridge C—OR—C stretching (cellulose)
1055	C—OR stretching (cellulose)
898	Antisym., out of phase ring stretching

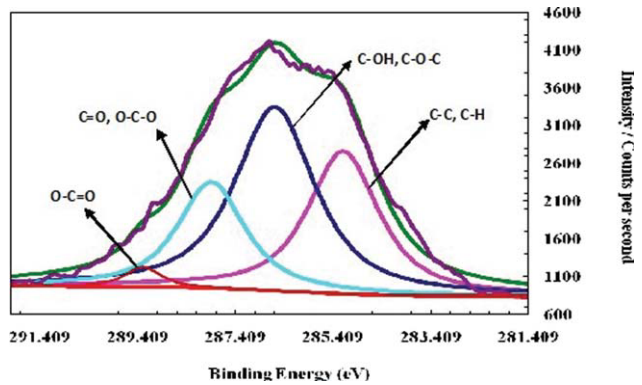

Figure 3 High resolution XPS spectra showing the deconvoluted **C1s** envelope. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

and distributions (%) of functional groups were given in Table III.

Tanobe et al.[16] determined the O/C as 0.57 for *Luffa cylindrica* collected from Southeast region of Brazil. It is revealed by Tanobe that the O/C (0.61) ratio is an indication that there still is lignin on the surface, when this ratio reaches 0.83 the sample is said to be pure cellulose, whereas when it is in the range 0.31–0.40, it is said to be pure lignin.[17]

Thermogravimetric analysis

It is known that natural fibers contain cellulose, hemicellulose, and lignin, and these components contribute the thermal stability of natural fibers. Therefore, the contents of these components affect the thermal stability of fibers. The thermal degradation of natural fibers is a two-stage process, one in the temperature range 220–280°C and another in the range 280–300°C. The low-temperature degradation process is associated with degradation of hemicellulose, whereas the high-temperature process is due to lignin.[18,19] TG and derivative thermogravimetric analysis (DTGA) graphs of *Luffa cylindrica* were presented in Figure 4. Weight loss values in the ranges 25–125 and 125–600°C are obtained to be about 8 and 71%, respectively. The weight loss versus temperature curve exhibits a first drop from 25 to 125°C, which could be due to the evaporation of water retained by the fibers. Weight loss values in the range 25–125°C and 125–600°C are 7.8 and 70.5%, respectively. Total

TABLE III
Contributions of Functional Groups on the Surface of *Luffa cylindrical*

	Binding energy	%
C—C, C—H	285	31.9
C—OH, C—O—C	286.6	43.4
C=O, O—C—O	287.9	22.5
O—C=O	289.3	2.2

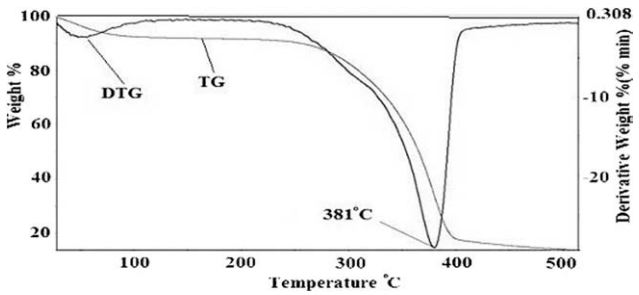

Figure 4 TGA analysis of *Luffa cylindrica*.

weight loss value in the range 25–600°C is 78.3%. The maximum degradation temperature of *Luffa cylindrica* is obtained to be 381°C.

Scanning electron microscopy

SEM micrographs of *Luffa cylindrica* were presented in Figure 5 at ×25, ×500, ×1000, and ×2500 magnifications. The shapes of fiber resemble long strips and have not a flat surface. There are grooves or channels parallelly distributed along with the longitudinal direction of fibers and small particles penetrate into channels. These small particles seem to be available on the surface as well.

Fiber tensile test

Tensile strength of *Luffa cylindrica* was conducted, and a comparison was given in the Table IV. As can be seen from Table IV, tensile strength of *Luffa cylindrica* was relatively lower than most of the fibers presented in Table IV.

Composite properties

TGA analysis of *Luffa cylindrica*-polyester composite

TGA and DTG curves of *Luffa cylindrica*/polyester composite were presented in Figure 6. As can be seen from TGA curve, thermal degradation of *Luffa cylindrica* increased after fabricating *Luffa*/polyester composites. Degradation *Luffa*/polyester takes place almost in one step with a peak maximum (T_{max}) at 398°C. After *Luffa cylindrica* was reinforced with unsaturated polyester matrix, its maximum degradation temperature increased from 381 to 398°C. Also, it can be added that while degradation for *Luffa cylindrica* starts at about 200°C, degradation for *Luffa*/polyester composite starts at about 270°C.

Weight loss until 200°C is about 4%. It can be noted that in the temperature range 25–600°C, most of the mass (95%) was lost.

SEM observation of *Luffa cylindrica*-polyester composite

Figure 7 shows the SEM micrograph of the composite fracture surface obtained from tensile tests. Although pull-outs cannot be clearly seen from SEM image, cavities or gaps, which have different sizes and shapes, occur in the matrix. It indicates a weak

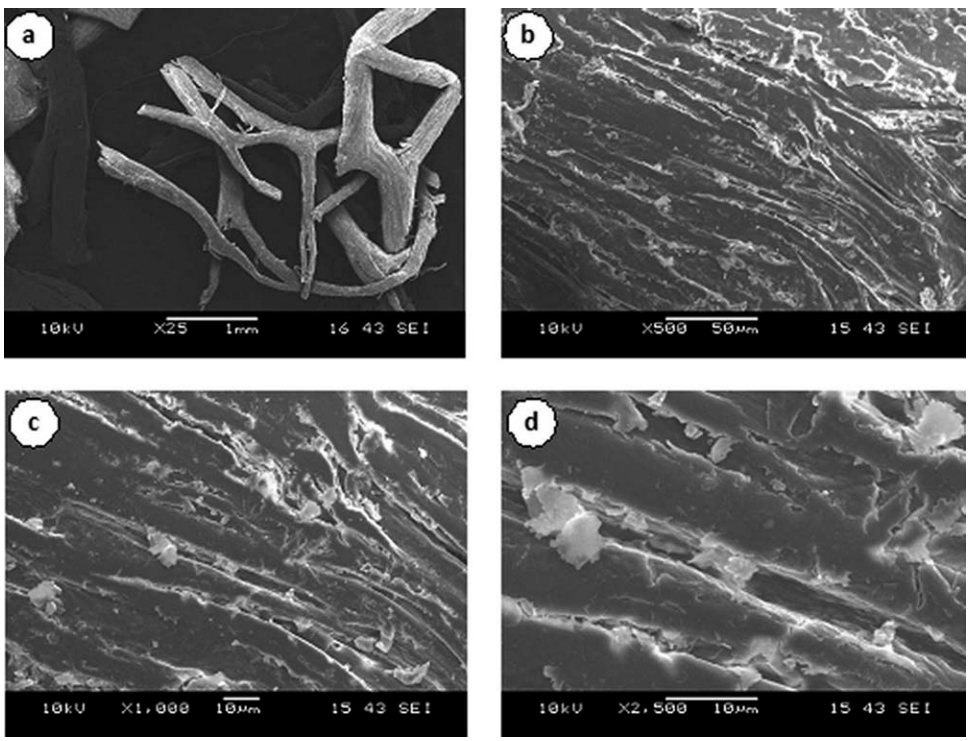

Figure 5 SEM micrographs of *Luffa cylindrica* at different magnifications (a) ×25; (b) ×500; (c) ×1000; (d) ×2500.

TABLE IV
Properties of Selected Natural Fibers

Fiber	Tensile strength (MPa)	Tensile modulus (GPa)	Elongation at break (%)	Reference
Jute	400–773	10–30	1.5–1.8	20, 21, 22
Hemp	690	70	2.0–4.0	23
Kenaf	930	53	1.6	23
Ramie	400–938	61.4–128	3.6–3.8	23, 24
Cotton	200–400	5.5–12.6	6.0–8.0	21, 22
Coir	95–174	2.5–4.5	13.7–41	20–27
Sisal	511–635	9.4–22	2.0–2.5	22, 29
Banana	700–800	27–32	2.5–3.7	20, 21, 29
Pineapple	362–748	25–36	2.0–2.8	21, 30, 31
Flax	500–1500	27.6	2.7–3.2	23
Luffa cylindrica	385 ± 10.52	12.2 ± 1.02	2.65 ± 0.05	

interface between *Luffa cylindrica* and polyester matrix.

Mechanical properties of *Luffa cylindrica*-polyester composite

Before seawater steam exposure, tensile strength, flexural strength, and ILSS values of *Luffa cylindrica*/polyester composite are 49.66, 81.13, and 20.35 MPa, respectively. After seawater exposure, tensile strength, flexural strength, and ILSS values decreased significantly to 35.98 ($P = 0.009$), 61.74 ($P = 0.0002$), and 11.27 ($P = 0.000013$) MPa, respectively. The decreases in the mechanical properties can be visualized in Figure 8. The decrements are 28%, 24%, and 45% for tensile strength, flexural strength, and ILSS, respectively. Tensile strength of the composite, which subjected to seawater steam, is more affected than flexural strength under the same conditions. However, the greatest decrease was observed in ILSS value. As expected, there is a decrease in the mechanical properties after aging due to the harmful effect of the absorbed water. It is seen that aging of *Luffa*-reinforced polyester composites in seawater affects the adhesion between polyester matrix and *Luffa cylindrica* significantly. This case may be explained by the swelling effect. After water exposure, fiber swells and becomes more porous and less stiff.[32–34] The swelling can promote interfacial debonding due to dimensional instability, and more water is absorbed through the voids formed in the interface. The voids that exist between fibers and matrix material enhance seawater diffusion through the defects, pores, and microcracks formed along the fiber/resin interface.[35,36] The effect of water diffusion through the material is a matrix plasticization, which reduces the mechanical properties of the composite. The presence of water also causes delamination at layers interface.[37]

The tensile modulus ($P = 0.16$) and flexural modulus ($P = 0.22$) do not decrease significantly. Moduli of composite material are less sensitive to aging effects than its tensile strength. According to Ahmed and Jones, the interfacial adhesion between fiber and matrix does not have strong influence on modulus, inasmuch as the forces acting on the interface are not exceeded by the applied stress.[38] After seawater steam exposure, elongation at break value also decreased by 31%. As conclusion, mechanical properties of *Luffa Cylindrica* /polyester composite change after seawater absorption.

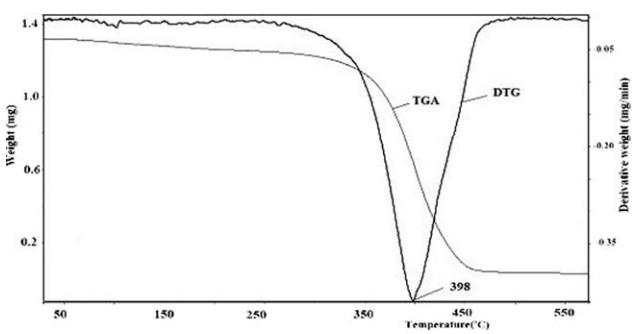

Figure 6 TGA analysis of *Luffa cylindrica*/polyester composite.

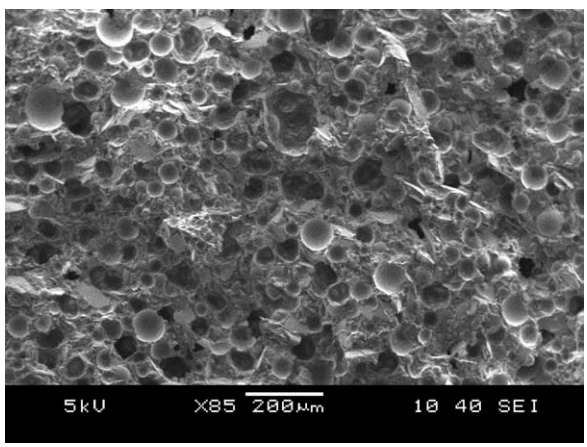

Figure 7 SEM micrographs of *Luffa cylindrica*/polyester composite.

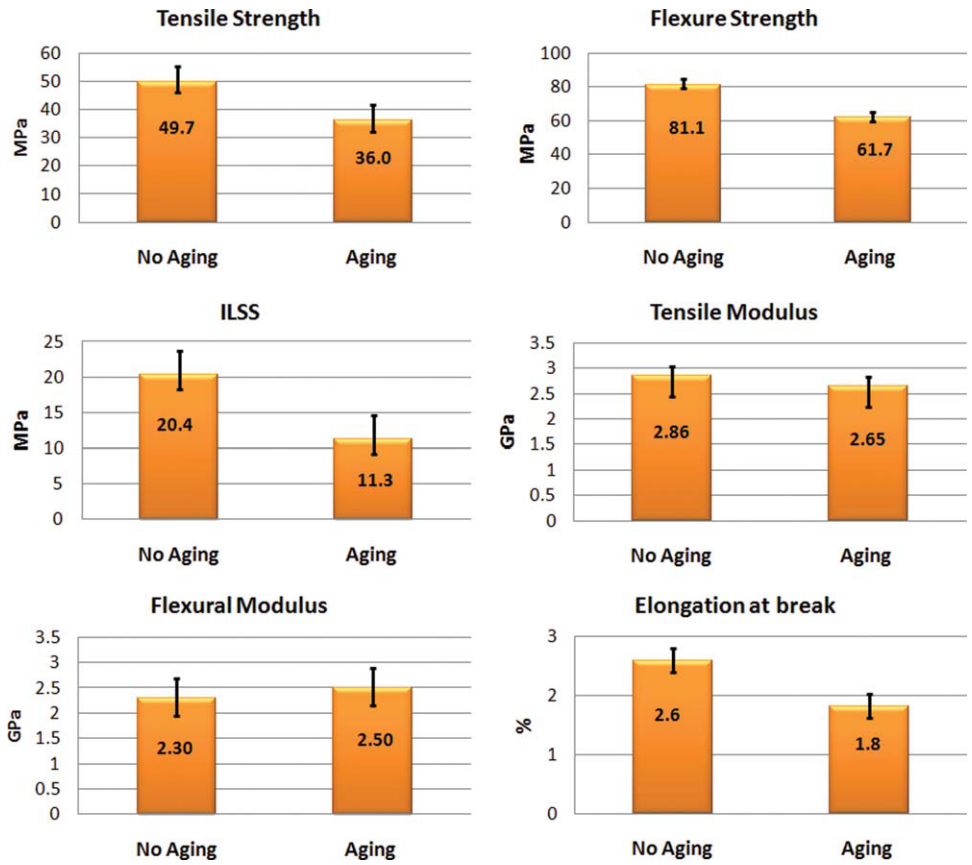

Figure 8 The mechanical properties of *Luffa cylindrica*/polyester composite. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

CONCLUSION

In this study, first, *Luffa cylindrica* of Turkey's Aegean Region was characterized by using Fourier transform infrared spectrophotometer, XPS, scanning electron microscopy, TGA, and fiber tensile test. The contents of cellulose, hemicellulose, and lignin were also obtained. Major findings were as follows: degradation temperature was 381°C, O/C ratio was 0.61, and the distributions of surface functional groups were C—C, C—H (31.9%), C—OH, C—O—C (43.4%), C=O, O—C—O (22.5%), and O—C=O (2.2%). Cellulose, hemicellulose, and lignin contents were 63.0%, 14.4%, and 1.6%, respectively. Tensile strength and tensile modulus of *Luffa cylindrica* were determined to be 385 MPa and 12.2 GPa, respectively.

Second, aging of *Luffa cylindirica*-reinforced polyester composite under a steam of seawater containing 5% NaCl for 170 h at 50°C was conducted. Tensile strength, tensile modulus, and elongation at break of *Luffa cylindrica* were 385.0 MPa, 12.02 GPa, and 2.65%, respectively. After water aging, tensile, flexural, ILSSs, and elongation at break values of the composite decreased ~ 28%, 24%, 45%, and 31%, respectively. Namely, aging of *Luffa cylindrica*-reinforced polyester composites under a steam of seawater-containing 5% NaCl for 170 h at 50°C decreased the mechanical properties of the composite, significantly.

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