Effect of the Atmospheric Plasma Treatment Parameters on Jute Fabric: The Effect on Mechanical Properties of Jute **Fabric/Polyester Composites**

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ABSTRACT: The effect of atmospheric air plasma treatment of jute fabrics on the mechanical properties of jute fabric reinforced polyester composites was investigated. The jute fabrics were subjected to different plasma powers (60, 90, and 120 W) for the exposure times of 1, 3, and 6 min. The effects of plasma powers and exposure times on interlaminar shear strength, tensile strength, and flexural strength of polyester based composites were evaluated. The greatest ILSS increase was about 171% at plasma power of 120 W and exposure time of 6 min. It is inferred that atmospheric air plasma treatment improves the interfacial adhesion between the jute fiber and polyester. This result

INTRODUCTION

Renewable resources in the field of fiber-reinforced materials and the possible subsequent new range of applications represent an important basis in the fulfillment of ecological objectives of creating closed material circuits. Natural fiber-reinforced composites are finding successful application in the automotive, electronics, and engineering sectors.¹

Because of low density, good mechanical performance, unlimited availability, and problem-free disposal properties, natural fibers offer a promising alternative to the other technological reinforcing fibers presently available. Moreover, natural fibers are advantageous when processed because of their lowabrasive properties compared those of to harder inorganic fillers, such as glass fibers or mica.²

The application of natural fibers as reinforcement in composite materials requires a strong adhesion between fiber and the synthetic matrix.³ There are two types of methods used to optimize the fiber surface; physical methods and chemical methods. These

was also confirmed by scanning electron microscopy observations of the fractured surfaces of the composites. The greatest tensile strength and flexural strength values were determined at 120 W for 1 min and at 60 W for 3 min, respectively. Moreover, it can be said that atmospheric air plasma treatment of jute fibers at longer exposure times (6 min) made a detrimental effect on tensile and flexural properties of jute-reinforced polyester composites. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 121: 634-638, 2011

Key words: cold plasma; jute; composite; mechanical properties

modification methods are of different efficiencies for improving the adhesion between matrix and fiber.⁴ Physical treatments change structural and surface properties of the fiber and thereby influence the mechanical bonding in the matrix.⁵ The most important chemical modification involves coupling methods. The coupling agent used contains chemical groups, which can react with the fiber and the polymer. The bonds formed are covalent and hydrogen bonds.⁶

As a type of environmentally friendly physical surface modification technology, plasma treatment is a simple process without any pollution. This technique appears to be original in comparing many chemical treatments as it modifies the surface of the fibers without affecting the bulk properties and the duration times of treatment are also short.³ Cold plasma treatment may improve adhesion between fiber and matrix.⁷

This article includes the effect of atmospheric air plasma treatment of jute fibers on the mechanical properties of polyester-based composite. The tensile, flexure, short-beam shear tests, and scanning electron microscopy (SEM) analysis were performed to analyze the efficiency of the atmospheric air plasma treatment on the mechanical properties of jute fiber reinforced polyester composites.

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EXPERIMENTAL

Materials

Orthophtalic unsaturated polyester resin (C-92 N8) were purchased from Cam Elyaf Corp. Turkey. Methyl ethyl ketone peroxide (MEKP) was used as a catalyst, and cobalt naphthanate was used as an accelerator. The quantities of the accelerator and the catalyst were 0.8 phr and 0.85 phr of the resin, respectively. Jute woven fabric (area density of 300 g/m²) was supplied by Anıl Limited (Turkey).

Alkali treatment and atmospheric plasma treatment of jute fabrics

Jute fabrics were treated with 2% NaOH solution for 1h. After alkali treatment, atmospheric plasma device with four cylindrical electrode pairs was used to treat surface of the jute fabrics. In this study, three different plasma powers (60, 90, and 120 W) and three different exposure times (1, 3 and 6 min) were used for surface treatment of jute fibers. Alkali treatment and atmospheric plasma treatment of the fabric surface were given in details elsewhere.⁸

Composite fabrication

The jute fiber/polyester composite laminates using woven jute fabrics and orthophtalic polyester resin were fabricated with two plies of jute fabrics using hand lay-up process to produce laminates that were about 2 mm thick. After fabrication, the composites were cured at room temperature.

Mechanical testing

All mechanical tests were tested for 6 times to evaluate the repeatability.

Short-beam shear test

Interlaminar shear strength (ILSS) was measured to estimate the interfacial adhesion strength of the composite. The short-beam shear test (interlaminar shear test) was performed according to ASTM D-2344. The samples were tested at a crosshead speed of 1.3 mm/min. The support span/sample thickness ratio was 5 : 1.

Tensile test

Tensile tests were performed by using a computercontrolled Shimadzu Autograph AG-G Series universal testing machine, a 5-kN load cell. The tensile tests of the composites were carried out according to the ASTM D-3039 standard, and the crosshead speed

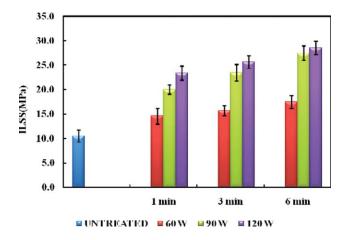


Figure 1 Interlaminar shear strength values for air treated jute/polyester composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

was 2 mm/min. System control and data analysis were preformed using Trapezium software.

Flexure test

The flexural strength and modulus of the composites were measured using the three-point bending test according to the ASTM D-790 standard. A threepoint flexural test was conducted with the universal testing machine using a crosshead speed of 1.3 mm/ min and a span length of 80 mm.

SEM analysis

The fracture surfaces of the composite samples were observed using a JSM 6060 scanning electron microscope (SEM), manufactured by JEOL (Tokyo, Japan). An acceleration voltage of 5–20 kV was used for SEM investigation. The fracture surface was coated with a thin gold layer before SEM analysis to reduce the extent of sample arcing.

RESULTS AND DISCUSSION

Short-beam shear test results

It is well known that good mechanical properties of composites are largely controlled by the interface, which is usually required to be strong in polymermatrix composites, thus transferring load from the matrix to the fibers efficiently.^{9–11} The mechanical properties of the interface are characterized especially by the ILSS. Figure 1 shows the ILSS of jute fiber-reinforced polyester composite as a function of plasma power and exposure time. The ILSS of untreated jute/polyester composite was 10.5 MPa. All the ILSS values of the atmospheric air plasmatreated composites are improved generously. As can

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TABLE I Tensile Properties of Fabricated Composites

Power/time		Tensile strength (MPa)	Tensile modulus (GPa)	Elongation at break (%)
Untreate 60 W 90 W 120 W	d 1 min 3 min 6 min 1 min 3 min 6 min 1 min 3 min 6 min	$56.8 \pm 2.4 \\71.5 \pm 4.3 \\86.6 \pm 6.3 \\79.6 \pm 5.8 \\78.4 \pm 5.6 \\87.3 \pm 5.7 \\82.56 \pm 3.5 \\91.2 \pm 4.6 \\84.1 \pm 5.2 \\72.3 \pm 3.4$	$5.2 \pm 0.7 \\ 5.6 \pm 0.9 \\ 5.9 \pm 0.7 \\ 5.7 \pm 0.4 \\ 5.6 \pm 0.9 \\ 5.9 \pm 0.7 \\ 5.6 \pm 0.5 \\ 6.0 \pm 0.5 \\ 5.8 \pm 0.2 \\ 6.1 \pm 0.4 \\ 0.4$	$\begin{array}{c} 1.19 \pm 0.06 \\ 0.97 \pm 0.04 \\ 1.09 \pm 0.06 \\ 0.85 \pm 0.02 \\ 0.97 \pm 0.04 \\ 1.12 \pm 0.08 \\ 0.79 \pm 0.05 \\ 0.95 \pm 0.06 \\ 1.13 \pm 0.03 \\ 0.76 \pm 0.01 \end{array}$

be seen from Figure 1, the greatest ILSS values for each exposure time were obtained at 120 W. The ILSS values increase to 23.3, 25.6, and 28.5 MPa with the increment of about 122, 144, and 171% after atmospheric air plasma treatment for 1, 3, and 6 min at 120 W, respectively.

The ILSS results indicate that atmospheric air plasma treatment improves the interfacial adhesion properties between the fiber and the matrix. As was emphasized in Ref. 8 (Part 1 of this study), surface friction coefficient values were increasing with the increasing of treatment time and plasma power because of the etching effect of the atmospheric plasma. It is known that surface friction values indicate the roughness characteristic of the fabric surface. Therefore, the ILSS increasing can be explained by the increase of surface roughness of jute fiber, which causes a better mechanical interlocking.

Tensile testing

Table I shows tensile properties of fabricated composites such as tensile strength, modulus, and elongation at break values.

Tensile modulus of untreated jute/polyester composite was 5.2 GPa. When jute fibers were atmospheric air plasma treated, tensile modulus of jute/ polyester composites increases for each plasma power and exposure time. As can be seen from Table I, the greatest tensile strength was obtained at 120 W for 1 min. The lowest tensile strength belongs to untreated jute-reinforced polyester composites. Tensile strength values of plasma treated jute fibers were presented in a previous study.⁸ In that study, the lowest fiber tensile strength value was obtained at plasma power of 120 W for exposure time of 6 min. However, the highest fiber tensile strength value belongs to jute fibers, which are not plasma treated. It is inferred that the adhesion between jute fiber and polyester is more important here. As mentioned earlier and Part 1, surface friction values indicate the roughness characteristic of the fabric surface.⁸ After plasma treatment, there was an increase in the fabric surface roughness in comparison with the alkali-treated jute fabric. The surface friction coefficient values also increased with the increasing of treatment time and plasma power. For the coating deposited by the conventional plasma, it is believed that the mechanical interlocking effect is the main bonding mechanism at the interface of substratecoating rather than the physical bonding.

Moon and Jang¹² indicate that the mechanical interlocking through the micropitting is known to play a key role in improving the interfacial adhesion of ultra high-modulus polyethylene fiber/vinyl ester composites by the argon plasma treatment. Surface roughness generally aids in adhesive bonding by providing a mechanical interlocking effect.¹³ At higher plasma powers and exposure times (such as 120 W and 6 min, respectively, in this study), decreasing of fiber strength was more dominant for the determination of tensile strength of composite. Under these conditions, the most suitable plasma power and exposure time can be considered as 120 W and 1 min, respectively. As a result of plasma treatment of jute fibers at 120 W for 1 min, tensile strength of jute fibers increased by 61%. On the other hand, the detrimental effect of atmospheric air plasma treatment on the mechanical strength of composite occurred at 120 W for 6 min.

Flexural testing

Flexural properties of jute/polyester composites are presented in Table II. It is obviously seen that, in most of the cases, flexural strength of the composites changed marginally when compared with that of the untreated jute/polyester.

A significant increase was obtained only at power of 60 W for 3 min, which corresponds to an increase of 22% approximately. Namely, it seems possible to

TABLE II					
Flexural Properties of Jute/Polyester Composites	s				

	1	5	1
Power/time		Flexural strength (MPa)	Flexural modulus (GPa)
Untreated		76.5 ± 3.2	5.12 ± 0.54
60 W	1 min	83.1 ± 6.4	6.34 ± 0.97
	3 min	93.6 ± 7.5	6.67 ± 0.76
	6 min	89.7 ± 7.8	6.56 ± 0.45
90 W	1 min	81.5 ± 8.6	6.78 ± 1.23
	3 min	86.6 ± 5.7	7.94 ± 0.96
	6 min	83.7 ± 7.3	6.72 ± 0.67
120 W	1 min	88.1 ± 5.7	6.27 ± 0.45
	3 min	81.2 ± 7.3	7.03 ± 0.35
	6 min	72.1 ± 4.5	6.87 ± 0.31

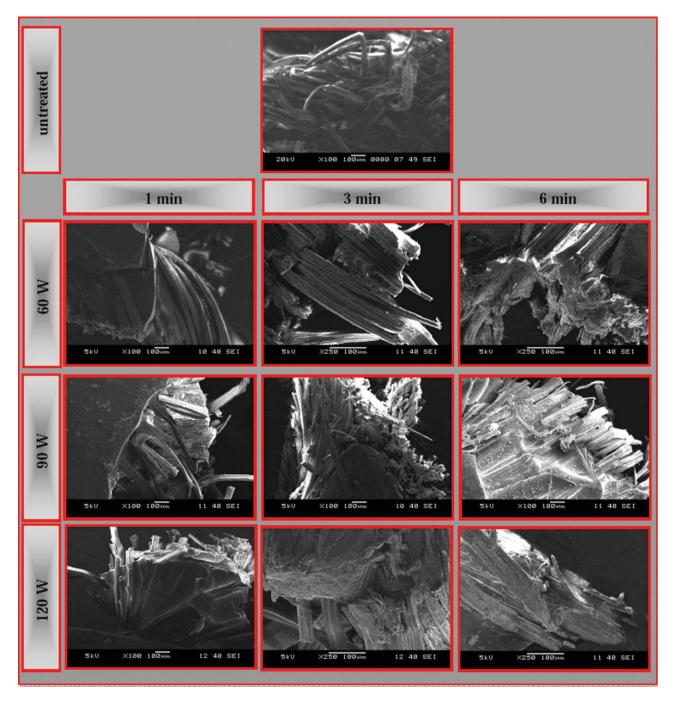


Figure 2 SEM images of the breakage region for jute fabric laminates that were tensile tested. (at different magnification levels). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

modify the surfaces of jute fibers without degrading them by exposing to a plasma power of 60 W for 3 min. It is also worth noting that treatments at 120 W for 6 min caused slight decrease in composite flexural strength in comparison with that of untreated jute-reinforced polyester composite.

Flexural modulus of the composites does not follow the same regime like the flexural strength. When the test results are evaluated, it can be seen that flexural modulus of the composites changed only marginally for 1 min at all plasma power levels when compared with that of the untreated jute/ polyester. Air plasma treatment for 3 min at 90 and 120 W yielded a significant increase in flexural modulus of the composites.

The main purpose of the plasma surface treatment of the fibers used as reinforcement in composite materials is to modify the chemical and physical structures of their surface layer to tailor fiber/matrix bonding strength, without influencing fiber bulk mechanical properties.¹⁴ High-plasma powers may etch the bulk of the fiber and weaken the fiber strength.

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As was shown in Ref. 8 (Part 1), longer exposure to plasma may also cause a loss of fiber strength, which is the case for 6-min long treatments where flexural strength of the composites almost remained unchanged and slight decreases in their flexural modulus were recorded. Yuan et al.¹⁵ reported that because cellulose and hemicellulose are more reactive to plasma, they are more easily etched away by plasma treatments, leaving more nonpolar lignin on the fiber surface, which contributes to the improvement of interfacial adhesion. To optimize the plasma treatment time and power, application of a plasma power of 60 W for 3 min may be considered as the most suitable one in terms of flexural strength for the surface modification of fibers while fabricating air plasma-treated jute reinforced polyester.

SEM observation

Scanning electron microscope (SEM) observation of the fractured surfaces of the composites was presented in Figure 2. As can be seen from Figure 2, it was found that the surfaces of untreated jute fibers were clean, and the fibers were pulled out from polyester matrix. The clean fiber surfaces and fiber pull-outs show that the interfacial adhesion between untreated jute fiber and polyester matrix is poor. Fracture morphology of plasma-treated composites shows that jute fibers were located in polyester matrix and well oriented along the composite, as seen from Figure 2. When the jute fibers are treated with atmospheric air plasma, the fibers are generally held together by unsaturated polyester resin and stick firmly to the jute fiber. Namely, greater amount of unsaturated polyester resin among the jute fibers exist due to better adhesion. It may be said that SEM micrographs for the composites are also consistent with ILSS values.

CONCLUSIONS

Atmospheric air plasma treatments of jute fabrics were conducted at plasma powers of 60, 90, and 120 W and exposure times of 1, 3, and 6 min. After fabricating of plasma-treated jute fiber-reinforced polyester composites, ILSS, tensile, and flexural properties of the composites were assigned. The greatest ILSS values obtained at 120 W. The ILSS values increased about 122, 144, and 171% after atmospheric air plasma treatment of jute fibers for 1, 3, and 6 min at 120 W, respectively. The whole ILSS values of the atmospheric air plasma-treated composites are greater than that of untreated jute/polyester composite. The ILSS results imply that atmospheric air plasma treatment of jute fibers improves the interfacial adhesion between jute fiber and polyester composites. The greatest tensile strength was obtained at 120 W for 1 min, which corresponds to 61% increase. The greatest increase in flexural strength was obtained at power of 60 W for 3 min, which corresponds to an increase of 22%. From SEM observations, it was seen that jute fibers, which are atmospheric air plasma treated, are held together by matrix material. As a result of this study, it is concluded that greater plasma power and exposure times caused a better adhesion, but longer exposure times (6 min) at any plasma power made a negative effect on tensile and flexural properties of jute-reinforced polyester composites.

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