Kenaf Bast Fiber Bundle–Reinforced Unsaturated Polyester Composites. II: Water Resistance and Composite Mechanical Properties Improvement

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Abstract

Water resistance properties of kenaf bast fiber bundle (KBFB)–reinforced composites with either unsaturated polyester (UPE) or vinyl ester (VE) matrices, developed in previous research, were studied. The effects of styrene content in UPE resin on the neat UPE resin tensile properties and molding pressure on composite flexural properties were evaluated. The effect of laser and plasma radiation on fiber-matrix interfacial shear strengths was studied. The composites exhibited high water uptake during short- and long-term water immersion. Encapsulation by surface coating and edge sealing improved composite water resistance. Statistical analyses indicated that molding pressures of 5 to 7 MPa were preferable to achieve the maximum composite mechanical properties, and 37.6 percent (wt/wt) styrene in the UPE resin gave the highest UPE matrix tensile properties in the studied range. Laser and plasma radiation of the KBFBs significantly improved fiber-matrix interfacial bonding.

The fabrication process, tensile properties, reliability, and cost-effectiveness of mechanically retted kenaf bast fiber bundle (KBFB)–reinforced unsaturated polyester (UPE) composites as potential materials for automotive exterior part applications have been reported (Du et al. 2010, submitted for publication a, submitted for publication b). Inexpensive untreated KBFB-reinforced UPE composites possessed higher tensile moduli and tensile strengths close to the specification requirement for glass fiber–reinforced sheet molding compounds (SMCs). These KBFB-reinforced UPE composites were also found to be more cost effective than SMCs for carrying tensile and flexural loads at high fiber loading levels.

The composite water resistance properties at a fiber loading of 65 percent (wt/wt) remains a concern since an automotive component manufacturer requires that the composite weight increase after 24 hours of immersion in water must be less than 0.8 percent (wt/wt). Natural fiber–

reinforced composites generally have high water uptake because of the hydrophilic nature of natural fibers. However, if these materials are designed for automotive exterior component applications, high water resistance is mandatory. Otherwise, composite encapsulation should be used.

Natural fiber–reinforced composite water uptake after 24-hour immersions under different conditions has been summarized. Espert et al. (2004) reported the water resistances of two types of natural fiber–reinforced composites. Sisal-reinforced polypropylene (PP) composites (30% [wt/wt] fiber loading) had a 4.3 percent weight increase after 24 hours at 23°C, and water uptake increased with increasing water temperatures (6.3% at 50°C). A PP and ethylene vinyl acetate copolymer matrix, reinforced by 10, 20, and 30 percent (wt/wt) cellulose fibers, gave weight increases at 50°C of 1.4, 3.4, and 5.6 percent, respectively. Demir et al. (2006) reported a 0.83 percent weight increase for a 10 percent (wt/wt) luffa-reinforced polypropylene composite at 25°C. Silane and maleic anhydride grafted polypropylene (MAPP) chemical treatments of luffa reduced composite water uptake. Dhakal et al. (2007) reported 0.44, 1.20, 1.72, and 2.40 percent water uptake (23°C) for hemp-reinforced UPE composites with fiber volume fractions (vol/vol) of 10, 15, 21, and 26 percent, respectively. Wang et al. (2006) reported a 2.6 percent weight increase for a high-density polyethylene composite reinforced by 40 percent (wt/wt) of rice hull at 23°C. The weight uptake become larger as the fiber loadings increased: 3.6, 3.9, 4.7 and 7.1 percent for composites with rice hull loadings of 50, 55, 60, and 65 percent (wt/wt), respectively. Nosbi et al. (2010) reported the water uptake of 10 to 11.5 percent for a 70 percent (wt/wt) kenaf fiber–reinforced UPE composite at room temperature, and the water uptake was also affected by solution type. The literature suggests that the water resistance of natural fiber–reinforced polymer composite depends on various factors, such as fiber and polymer type, solution type, fiber loading, use of coupling agents, and temperature. Therefore, the water resistance property of the composite generated by the newly developed method (Du et al. 2010) is still unknown.

Another concern with natural fiber–reinforced composites is their poorer absolute mechanical properties versus those of glass fiber–reinforced composites. Chemical treatment of fibers is the most widely used method for improving composite strengths and moduli. The chemicals used for fiber treatments included alkalis (Valadez-Gonzalez et al. 1999, Agrawal et al. 2000, Aziz and Ansell 2003), acetylation (Sreekala et al. 2000), silanes (Valadez-Gonzalez et al. 1999, Misra et al. 2006, Drzal et al. 2007), maleated coupling agents for thermoplastic matrices (Gassan and Bledzki 1997, Rowell et al. 1999, Feng et al. 2001, Sameni et al. 2003), and isocyanate (Maldas et al. 1989). In general, chemical treatments of natural fibers improve both mechanical properties and water resistance simultaneously. However, chemical treatments increase manufacturing costs and create wastewater discharges and make high-volume production unfeasible. Laser and plasma treatments of fiber have been also reported as green methods to improve fiber-matrix adhesion (Mizoguchi et al. 2000, Morales et al. 2006).

Composite mechanical properties can also be potentially improved via simply adjusting process parameters, such as molding pressure, molding time, and polymer formulation, which require little or no additional capital investment.

Molding pressures were varied during fiber-reinforced composites fabrication in literature reports. The typical molding pressures for fabricating glass fiber–reinforced SMCs ranged from 5 to 10 MPa (European Alliance for SMC 2001). The molding pressures for reported natural fiber–reinforced UPE composites ranged from 0.5 to 3 MPa (Baiardo et al. 2004, Paiva Júnior et al. 2004, Idicula et al. 2005, Misra et al. 2006). de Deus et al. (2005) found that the flexural strength of piassava fiber–reinforced orthophtalic polyester composite (40% [wt/wt] fiber loading) was independent of a molding pressure below 9.2 MPa. The composite flexural strengths in that work decreased when the molding pressures were higher than 9.2 MPa, which could be attributed to fiber crushing. Furthermore, composites with low piassava fiber loadings (20% and 30%, wt/wt) exhibited decreasing flexural strengths as molding pressures increased, which was attributed to fiber misalignment.

According to classical mechanical models (Kelly and Tyson 1965, Halpin 1969), the mechanical properties of fiber-reinforced composites can be improved by improving the matrix mechanical properties. Formulation adjustments of the polymer matrix can affect its mechanical properties, which in turn affect the composite mechanical properties. Sanchez et al. (2000) found that both thermal stability and mechanical properties of a commercial UPE resin (RESAPOL 10-203) were dependent on the resin's styrene content. The UPE resin with 24 percent (wt/wt) styrene had the maximum flexural properties. The relationship between styrene concentrations and UPE mechanical properties was also documented by Mallick (1988). An increase in styrene reduced the modulus of the cured UPE resin since styrene increases the space between cross-links. The tensile strength first increased and then decreased with increasing styrene contents.

The literature review suggested that composite water resistance and mechanical properties can be simply improved through adjusting process and formulation. These process parameters should be studied for producing quality products. The detailed objectives of this study were (1) to obtain water resistance of KBFB-reinforced polymer composites and to investigate the effect of matrix, mat preforming binder, and encapsulation on composite water resistance properties; (2) to investigate the effect of process parameters including styrene content and molding pressure on matrix and composite mechanical properties; and (3) to explore the effect of laser and plasma fiber irradiation on kenaf-polymer matrix shear strengths.

Materials and Methods

Materials

Mechanically retted KBFBs were supplied by Kengro Corporation. The commercial UPE (Aropol Q-6585) and vinyl ester (VE; Derakane 782) were provided by Ashland Chemical Company. The diluent, styrene, and the catalyst, t-butyl perbenzoate (TBP), were purchased from Fisher Scientific Inc. Methyl ethyl ketone peroxide, purchased from Sigma-Aldrich Corporation, and cobalt naphthenate, purchased from Fisher Scientific Inc., were used as another catalyst system. A poly(vinyl acetate) (PVAc) water emulsion (solid content, 46%), provided by Tailored Chemical Products Inc., and the maleic anhydride grafted polypropylene (Epolene G-3015; molecular weight, 47,000; maleic anhydride, <1.0%, wt/wt), provided by Eastman

Chemical Company, were used as mat preforming binders. Polyurethane (Helmsman spar urethane), purchased from Lowe's, and wax (Mainstays candle), purchased from Wal-Mart, and UPE resin (formulation: 100 parts of UPE, 15 parts of styrene, 0.5 part of CoNa, and 1.0 part of MEKP) were used as edge-sealing agents.

Experimental

Composite fabrication.—Composites were fabricated as previously described in full detail (Du et al. 2010). As-received mechanically retted KBFBs were ground into short fibers with an average length of 3.3 mm in a Thomas Wiley Mill (Model 4). Short KBFBs were then made into preforms using PVAc or MAPP binders. UPE and VE resins were prepared by using 100 parts of UPE or VE, 15 parts of styrene, and 2 parts of TBP by weight. Mats were preformed using either PVAc or MAPP. Fiver preformed mats were infused with UPE or VE resins to form one prepreg. The resultant prepregs were then compression molded into composites.

Water absorption.—Kenaf/PVAc/UPE composites were cut into 25.4 by 76.2-mm^2 specimens. Twelve replicates of these specimens were made. Kenaf/MAPP/UPE and kenaf/PVAc/VE composite specimens were also prepared. At least two replicates of each of these composites were prepared. To evaluate the effect of encapsulation on composite water resistance properties, 25.4 by 76.2-mm^2 kenaf/PVAc/UPE samples were prepared and were coated with UPE. These surface-coated specimens were individually edge sealed by one of three edge-sealing agents: wax, polyurethane, and UPE, respectively.

Composite water resistance was tested in accordance with ASTM Standard D570-98 (American Society for Testing and Materials [ASTM] 2006b). Specimens were immersed in deionized water at 23°C, and the weight of each specimen was recorded after 2 and 24 hours. Intermediate measurements were recorded for some kenaf/PVAc/UPE composite. A few kenaf/PVAc/UPE and kenaf/PVAc/VE specimens were kept submerged underwater until their moisture contents reached equilibrium.

Styrene content.—The commercial UPE (Aropol Q-6585) resin contains 65 parts UPE and 35 parts styrene by weight. UPE resins with four different styrene content levels were prepared by adding four additional amounts of styrene: 0, 5, 15, and 30 parts by weight, respectively. Table 1 summarizes the amount of each component and the corresponding styrene content for each UPE resin formula-

tion. The mixed UPE resins were cured at ambient temperature for 24 hours and postcured in an oven at 80°C for 3 hours.

The cured resins were shaped to dog-bone tensile specimens using a computer numerical controlled machine and were tested on an Instron 5869 universal testing machine in accordance with ASTM Standard D638-03 (ASTM 2004). Tensile strains were recorded by an Instron 2630-100 series extensometer.

Molding pressure.—The mat prepregs were assembled by the same process for making water resistance property testing samples. The prepregs were then compression molded into the final composites at three pressures: 3, 5, and 7 MPa, respectively. Densities, flexural properties, and impact properties of resultant composites were determined by ASTM Standards D790-03 (ASTM 2003) and D256-06 (ASTM 2006a).

Fiber surface treatment.—Eighteen 50 by 3-mm^2 kenaf strips were cut from kenaf bast fiber ribbons. One side at the end of these strips (3 by 5 mm^2) was smoothed by sandpaper (120 grit). Six strips were dried in an oven at 103°C for 5 hours to remove the residual moisture.

The remaining 12 strips were prepared for investigating the fiber-matrix adhesion improvement via laser and plasma radiation. The pulsed laser beam of 532 nm was generated by an Nd:YAG pumped dye laser with a single pulse energy of 10 to 15 mJ and a repetition rate of 20 Hz. During the treatments, each of the strips was placed on an x-y adjustable platform, and the laser beam was focused to each spot of the strip surface with a spatial resolution of 0.5 mm^2. The averaged exposure time of the laser radiation at each spot was 5 to 10 seconds. The smoothed 3 by 5-mm^2 surfaces of six kenaf strips were universally treated.

The remaining six samples were treated by an atmospheric pressure argon microwave-induced plasma jet that was excited by a 2.45-GHz plasma source. The plasma power was 48 W. The argon flow rate was 0.1 to 0.3 liter/min. The nonthermal plasma gas temperature was close to 450 K. The image of the plasma jet is shown in Figure 1. The plasma plume was chemically reactive because of the presence of plasma radicals and excited neutral species, such as OH, O, NO, O_2^*, N_2^*, etc. The

Table 1.—Formulation of the unsaturated polyester (UPE) resin system.[a]

	Parts (by weight)			
UPE	100	100	100	100
UPE solid	65	65	65	65
Styrene (in UPE)	35	35	35	35
Styrene (additional)	0	5	15	30
MEKP	1	1	1	1
Cobalt naphthenate	0.5	0.5	0.5	0.5
Styrene concentration (%, wt/wt)	34.5	37.6	42.9	49.4

[a] Weight contents of methyl ethyl ketone peroxide (MEKP), cobalt naphthenate, UPE, and styrene. Styrene present in the final compositions was the sum of the styrene present in the UPE resin and the additional styrene added to the formulation.
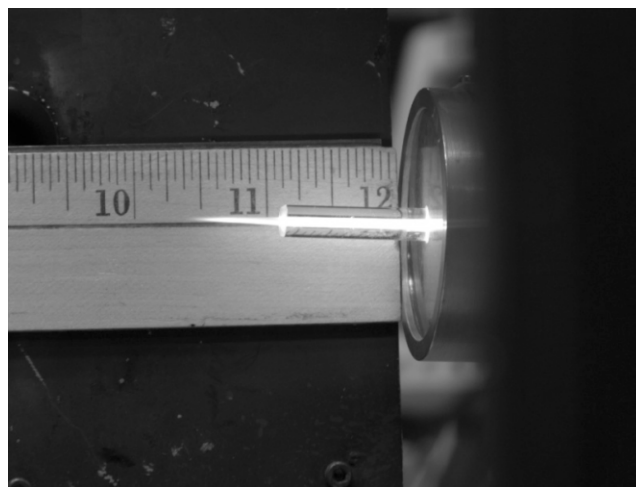

Figure 1.—An image of the atmospheric argon microwave plasma jet for treating kenaf strip surfaces.

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smoothed 3 by 5-mm^2 surface of each of the kenaf strips was exposed to the plasma jet tip for 1 second. In this case, the surfaces were treated with a combination of low-temperature nonthermal heating and chemical bath resulted from reactive species, predominantly OH, O, and O$_2$* (Fridman 2008).

Shear specimens were prepared by pasting all 18 control or treated kenaf strips onto 13 by 13 by 6.4-mm^3 UPE blocks using the UPE resin as the adhesive. These specimens were cured at ambient temperature for 24 hours and postcured at 80°C for 3 hours. Shear forces were determined on an Instron 5566 universal testing machine and shear strengths were calculated by the following equation:

$$\text{Strength} = F_f/(L \cdot W)$$

where F_f, L, and W are the failure force, overlap length, and width, respectively.

Results and Discussion

The experimental data means were compared using a general linear model procedure. The main effects of encapsulation, sealing agent, styrene content, molding pressure, and laser or plasma treatment on composite water resistance, matrix, composite, and fiber-matrix shear properties, respectively, were studied.

Water absorption

The weight increases of kenaf/PVAc/UPE, kenaf/MAPP/UPE, and kenaf/PVAc/VE composites during the water immersion test are illustrated in Figure 2. The average weight increase of kenaf/PVAc/UPE composites after 24 hours of water immersion was 22.1 percent (wt/wt). The average weight increases of the kenaf/MAPP/UPE and kenaf/PVAc/VE composites were 10.2 to 11.8 percent (wt/wt), respectively, both significantly lower than that of kenaf/PVAc/UPE ($P = 0.009$ and 0.018). The greater water uptake for the kenaf/PVAc/UPE composite is attributed to the hydrophilicity of the kenaf fibers and the PVAc adhesive as well as imperfect fiber-matrix bonding.

The lower water uptake by kenaf/PVAc/VE composites may be due to the better adhesion between KBFBs and the VE resin. VE molecules contain hydrogen bonding hydroxyl groups along the molecular chain length that can promote adhesion to the hydroxyls of the lignocellulosic fibers (Mallick 1988). The lower water uptake by the kenaf/MAPP/UPE composites is the result of MAPP reaction on the fiber surfaces, which improves compatibility between KBFBs and UPE.

Figure 3 shows the water uptake of UPE-coated kenaf/PVAc/UPE composites after 24 hours. The control specimens (edges were not sealed) had an average water uptake of 8.3 percent versus the water uptake of 4.3, 3.3, and 1.6 percent (wt/wt), respectively, for the UPE-coated kenaf/PVAc/UPE composites, which were edge sealed by wax, polyurethane, and UPE. Both the surface coating and the edge sealing significantly improved composite water resistance properties. However, none of these absorbed less than 0.8 percent (wt/wt) water, which is the specification requirement for glass fiber/UPE composites from an automotive parts manufacturer.

After 24 hours of immersion in water, the control specimens of the UPE-coated KPU composites swelled along all four edges. In contrast, the UPE-coated composites edge sealed by wax, polyurethane, and UPEs absorbed water only through sporadic distributed pores located along the four edges. These pores formed passageways through which water can infiltrate. Natural fibers are hydrophilic and hygroscopic, readily absorbing moisture. Water was absorbed into the composites in two forms: free water by capillary action (through cell cavities) and bound water by diffusion (passageways that connect adjacent cells). Fiber swelling caused high local stresses and expanded the gaps between fiber and matrix, forming additional passages.

The water uptake of kenaf/PVAc/UPE and kenaf/PVAc/VE composites versus time during a long-term water immersion test is plotted in Figure 4. After 650 hours, the composite moisture contents have almost approached equilibrium. Approximately half the water was absorbed in the first 24 hours.
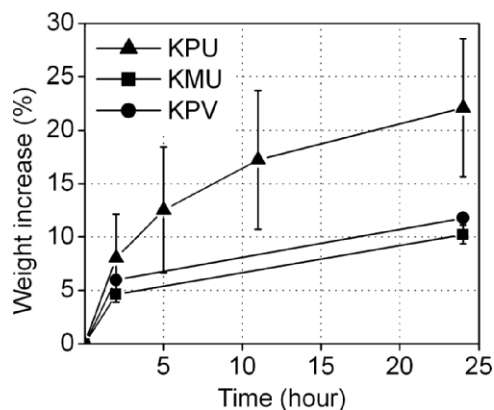

Figure 2.—Weight increases of kenaf/PVAc/unsaturated polyester (KPU), kenaf/MAPP/unsaturated polyester (KMU), and kenaf/PVAc/vinyl ester (KPV) composites versus time during a 24-hour water immersion test.
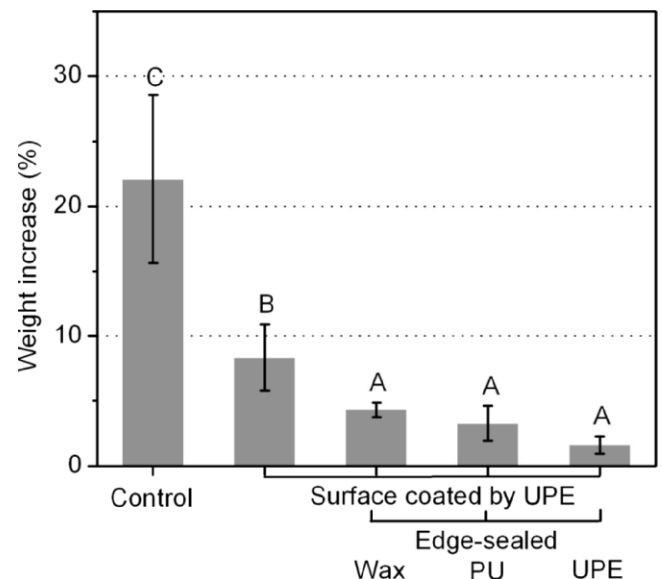

Figure 3.—Weight increases from water uptake after 24 hours of control and encapsulated kenaf/PVAc/unsaturated polyester (KPU) composite (values with the same capital letter are not statistically different at the 5% significance level).
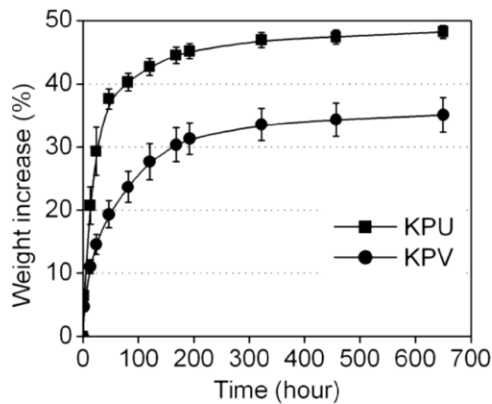
Figure 4.—Weight increases due to water uptake of kenaf/PVAc/unsaturated polyester (KPU) and kenaf/PVAc/vinyl ester (KPV) composites versus time during a long-term water immersion test.

Figure 6.—Composite properties versus compression molding pressures: (a) bulk densities (oven dried), (b) impact strengths, (c) flexural strengths (MOR), and (d) flexural moduli (MOE) (values with the same capital letter are not statistically different at the 5% significance level).

Styrene content

Tensile moduli and strengths of the cured UPE resin (no fibers) versus styrene content are plotted in Figure 5. As the styrene content increased, tensile strengths and moduli initially increased and then dropped. The maximum tensile modulus and strength appeared at a styrene content of 37.6 percent (wt/wt). Thus, 5 parts styrene per 100 parts of the commercial UPE resin was the optimized blending ratio in the studied range for styrene and the UPE resin in order to achieve the maximum matrix tensile properties. The cured UPE failed in cleavage fracture modes. This leads to larger scatter in tensile strengths than moduli.

Molding pressure

Figure 6 illustrates the relationship between densities (bulk density, oven dried), impact strengths, and flexural properties of the kenaf/PVAc/UPE composites fabricated
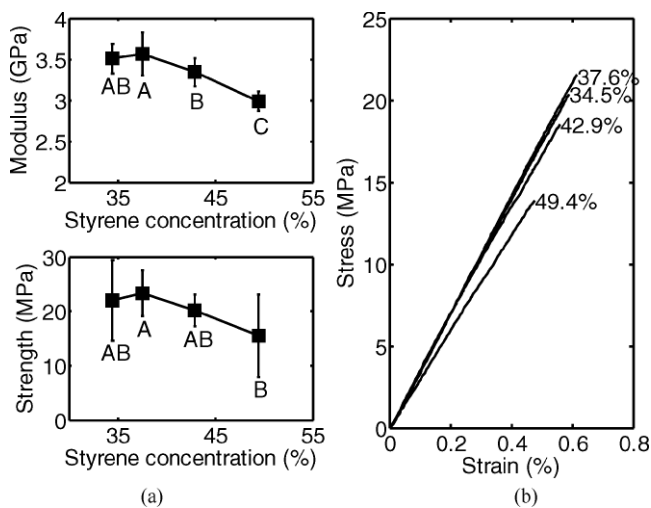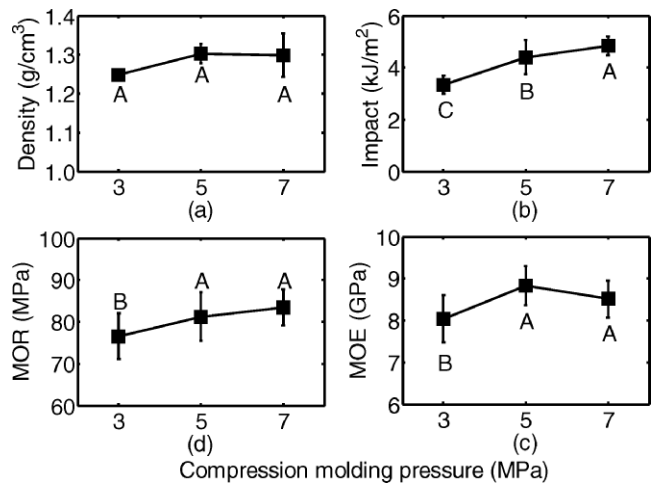

Figure 5.—Tensile properties of neat cured unsaturated polyester (UPE) versus its styrene contents by weight: (a) tensile properties versus styrene contents (values with the same capital letter are not statistically different at the 5% significance level) and (b) typical stress-strain plots of UPE specimens at four styrene levels.

under molding pressures from 3 to 7 MPa. High molding pressures create better physical contact between the matrix and the fibers. However, if the pressure is too high, fiber crushing might occur, lowering composite mechanical properties (de Deus et al. 2005).

Composite densities increased significantly as molding pressures increased from 3 to 5 MPa (Fig. 6a). High pressures reduced void volume and improved the fiber-matrix interface in the composites, thereby increasing composite densities. Densities leveled off when the molding pressure increased from 5 to 7 MPa. Therefore, the composites were not further compressed.

Composite impact and flexural strengths increased as the molding pressure increased from 3 to 7 MPa (Figs. 6b and 6c). Statistical analyses showed that, at the 5 percent significance level, increasing the molding pressures from 3 to 5 MPa significantly improved composite flexural ($P = 0.020$) and impact strengths ($P < 0.001$). A further increase in molding pressures from 5 to 7 MPa caused an insignificant increase in composite flexural strengths ($P = 0.320$) and an additional significant increase in impact strengths ($P = 0.049$). The increase in impact and flexural strengths is attributed to fewer voids and better fiber-matrix contact and bonding, achieved when higher pressure is used.

Composite flexural moduli increased significantly as molding pressures increased from 3 to 5 MPa ($P < 0.001$; Fig. 6d) and then slightly decreased as molding pressures were increased to 7 MPa. However, this decrease was not statistically significant ($P = 0.131$). The results indicate that a 5-MPa molding pressure was the optimum to achieve the highest flexural moduli and that a 7-MPa molding pressure was the best for flexural and impact strengths.

Laser and plasma treatments

The fiber-matrix shear strengths of both control and laser-treated or argon plasma–exposed kenaf strips with the UPE matrix are illustrated in Figure 7. It is easily seen that the laser- and plasma-treated kenaf strips have greatly improved fiber-matrix shear strengths by 91.6 and 83.3 percent,
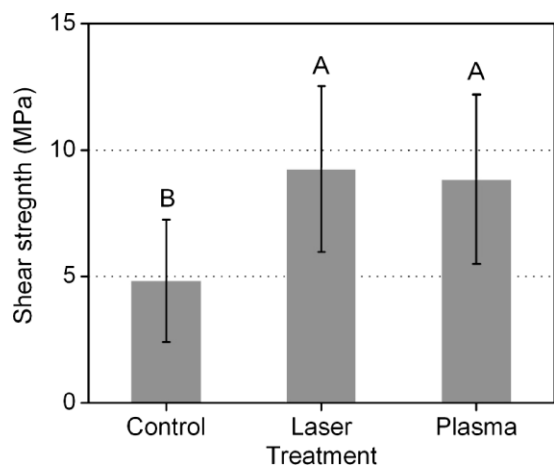
Figure 7.—The interfacial shear strengths of untreated and laser-exposed and plasma-treated kenaf strips bonded to the cured unsaturated polyester resin (values with the same capital letter are not statistically different at the 5% significance level).

respectively. Statistical analyses suggested that these laser and plasma treatments led to significant ($P = 0.033$ and 0.049, respectively) fiber-matrix shear strength increases. These experimental results also support similar conclusions made by Morales et al. (2006). These results suggest that the laser and low-temperature plasma exposures, widely used in material processing, are also a new environmentally friendly method for enhancing interface compatibility in natural fiber–reinforced composites.

Conclusions

The kenaf/PVAc/UPE composites had low water resistance properties. Encapsulation improved composite water resistance efficiently. The use of a VE matrix or MAPP as a mat preforming binder significantly improved composite water resistance properties.

The commercial UPE used in this study exhibited the highest tensile properties when the styrene content was 37 to 38 percent (wt/wt) for the given styrene content range. The optimized molding pressures for fabricating kenaf/UPE composites were 5 to 7 MPa to achieve the best mechanical properties. Laser and plasma radiation of kenaf fibers improved fiber/matrix shear strengths. These parameters studied can be referenced for manufacturing process optimization.

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