Kenaf Bast Fiber Bundle–Reinforced Unsaturated Polyester Composites. I: Processing Techniques for High Kenaf Fiber Loading

Yicheng Du Jilei Zhang Hossein Toghiani Thomas E. Lacy, Jr.
Yibin Xue Mark F. Horstemeyer Charles U. Pittman, Jr.

Abstract

The fabrication of kenaf bast fiber bundle/unsaturated polyester composites with high (60% to 67%, wt/wt) fiber contents was explored in this study. Mechanically ground kenaf bast fiber bundles were preformed into mats with a polyvinyl acetate emulsion adhesive. The preformed mats are easy to handle during subsequent processing with the unsaturated polyester resin and laminate compression molding. Fiber loadings as high as 65 percent (wt/wt) were achieved. The generated composites possessed high elastic moduli, and their tensile strengths were close to specification requirements for glass fiber–reinforced sheet molding compounds. These composites also exhibited higher specific tensile moduli and strengths than glass fiber–reinforced sheet molding compounds' specific modulus and strength lower bounds and those calculated from specification requirements for glass fiber–reinforced sheet molding compounds. If reduced void contents and enhanced interfacial binding can be achieved through improved processing, then natural fiber composites similar to those developed in this work have the potential to possess mechanical properties competing against those of currently used automotive sheet molding compounds.

Renewable and biodegradable natural fibers have the potential to replace petroleum-based or glass fibers in automotive structural part applications. These natural fibers possess many desirable characteristics, including low density, low cost, and high specific strength and stiffness. Automotive components made from lightweight natural fiber–reinforced polymer composites can reduce overall vehicle weight, which results in improved fuel economy and lower exhaust emissions.

Kenaf bast fiber bundles (KBFBs) are one type of commonly used natural bast fiber. Others include hemp, flax, and jute (Holbery and Houston 2006). Kenaf (*Hibiscus cannabinus* L.) is an annual herbaceous plant originally from Africa. It has a growing period of 90 to 150 days and may grow to 2.4 to 6 m in height. Its single, straight stem consists of an outer fibrous bark and an inner woody core that yield two distinct fibers, bast and core, respectively. The bast constitutes about 26 to 35 percent (by dry weight) of its stem, yet genetic strains have been developed that yield 35 percent or greater bast portions (Lloyd and Seber 1996). The harvested kenaf stems are usually first decorticated to separate the bark from the core, producing ribbons of kenaf

The authors are, respectively, Graduate Student and Professor, Forest Products Lab. (yd33@msstate.edu, jZhang@cfr.msstate.edu), Associate Professor, Dept. of Chemical Engineering (hossein@che.msstate.edu), Associate Professor, Dept. of Aerospace Engineering (lacy@ae.msstate.edu), Mississippi State Univ., Mississippi State; Assistant Professor, Dept. of Mechanical and Aerospace Engineering, Utah State Univ., Logan (anna.xue@usu.edu); Professor, Dept. of Mechanical Engineering (mfhorst@cavs.msstate.edu), and Professor, Dept. of Chemistry (CPittman@chemistry.msstate.edu), Mississippi State Univ., Mississippi State. The use of trade names is for the convenience of the reader. Such use does not constitute endorsement by Mississippi State University over other products equally suitable. Neither the US Government nor any agency thereof, nor any of their employees, makes any warranty, expressed or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the US Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the US Government or any agency thereof. Approved for publication as Journal Article no. FP547 of the Forest and Wildlife Research Center, Mississippi State Univ. This paper was received for publication in December 2009. Article no. 10711.

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fibers. These ribbons can be retted into fiber bundles or single fibers. It is preferable to harvest the kenaf crop once the fiber has been air dried (approximately 10% moisture content). This is achieved by leaving the crop standing in the field.

In general, the individual kenaf bast fibers are hollow tubes averaging 2.6 mm in length and 21 μm in diameter with an average length/diameter aspect ratio of 124, very similar to softwood species. The core fibers, with average lengths of 0.5 mm, closely match those of hardwoods (Lloyd and Seber 1996). The major constituents of kenaf bast fiber are cellulose, hemicellulose, and lignin. The amount of each constituent can vary significantly because of cultivation environment, geographic origin, age, location in the plant (from root to tip), and retting and separating technique. Lloyd and Seber (1996) reported weight percentages of 60.8 for cellulose, 20.3 for hemicellulose, 11.0 for lignin, 3.2 for extractives, and 4.7 for ash. Mohanty et al. (2000) reported lower cellulose (31% to 39%, wt/wt) and higher lignin (15% to 19%, wt/wt) amounts. Rowell et al. (2000) reported 44 to 57 percent (wt/wt) cellulose and 15 to 19 percent (wt/wt) lignin.

Table 1 summarizes physical and mechanical properties of kenaf fibers from the literature. In addition, the table lists the properties of commonly used E-glass fibers and the cured unsaturated polyester (UPE) resin used in composites for automotive parts. Holbery and Houston (2006) calculated the specific strength and elastic modulus of kenaf fibers from cited data and compared these values with those of E-glass fiber. E-glass fiber had a higher specific strength, but kenaf had a higher specific elastic modulus (36 GPa·cm^3/g) than that of E-glass (28 GPa·cm^3/g).

Based on the data contained in Table 1, the elastic modulus ratio for kenaf bast fiber to UPE ranges from 4 to 15 (Table 1). D'Almeida (2001) reported that matrix-fiber load transfer becomes more effective as the fiber-to-matrix elastic modulus ratio increases, which improves the composite's mechanical performance. At a fiber/matrix elastic modulus ratio of 5, a fiber volume fraction of 64 percent (i.e., 64%, vol/vol) is desired in order for the fibers to carry 90 percent of the applied load (Agarwal and Broutman 1980). High composite fiber volume fractions typically ensure effective fiber-matrix load transfer for natural fiber–reinforced composites.

Composite tensile properties are dominated by fiber volume fractions based on micromechanics approaches for predicting elastic modulus (Baiardo et al. 2004) and tensile strength (Haneefa et al. 2008) for randomly oriented short fiber composites. An increase in the amount of fiber generally increases the elastic modulus. However, increasing the volume fraction of fibers, especially short fibers, may also introduce more defects and reduce strengths. Fiber- to-matrix adhesion plays an important role in the changes in mechanical properties with fiber content.

D'Almeida (2001) performed a cost analysis indicating that inexpensive natural fiber–reinforced composites can be feasible alternatives to glass fiber–reinforced polyester composites particularly at high fiber volume fractions. Fibers with tensile strengths below 400 MPa require a minimum fiber fraction of 70 percent (vol/vol) in order to be competitive. A minimum fiber fraction of 50 percent (vol/vol) is necessary for fibers with tensile strengths ranging from 400 to 600 MPa. A minimum fiber fraction of 40 percent (vol/vol) is needed for fibers with tensile strengths greater than 600 MPa.

Natural fiber composite research has emphasized improving compatibility between hydrophilic fiber and hydrophobic polymer in order to enhance fiber/matrix adhesion (Misra et al. 2002, Aziz and Ansell 2004, Baiardo et al. 2004, John and Naidu 2004, Baley et al. 2006). Improving composite moisture resistance and dimensional stability while minimizing material and manufacturing costs are also major challenges in the development of structural natural fiber–reinforced composites.

A cost-effective fabrication process for producing KBFB/UPE composites with properties exceeding the minimum strength and stiffness required by industry standards must be developed in order to use kenaf fiber/unsaturated polymer composites in automotive applications. In this study, increasing fiber loading and using mechanically retted KBFBs were considered to be potential solutions to reduce manufacturing costs.

Limited studies were found for developing fabrication processes aimed at increasing KBFB content. A biocomposite sheet molding compound (SMC) process was invented by Drzal et al. (2007) with a 35 percent (wt/wt) fiber loading. The manufacturing process was created based on an existing SMC manufacturing process for fabricating glass fiber–reinforced composites. Resin transfer molding and vacuum-assisted resin transfer molding are also increasingly used to manufacture thermoset composites in the automotive industry (Holbery and Houston 2006). These processes allow the use of low shear during compounding and employ temperatures that do not result in fiber degradation. It is possible to achieve fiber loadings of up to 70 percent (wt/wt), but this requires a significant investment in equipment (capital costs; Holbery and Houston 2006).

The SMC combines a thermoset resin (such as UPE) with glass fibers, fillers, and other additives to form a fiber-reinforced composite (European Alliance for SMC 2001). These ingredients are compounded into sandwich-like resin/glass fiber/resin prepregs. After 3 to 5 days of maturation,

Table 1.—Physical and mechanical properties of kenaf bast fiber bundles, commercial E-glass fibers, and unsaturated polyester resin.

Fiber	Density (g/cm^3)	Elongation (%)	Tensile strength (MPa)	Elastic modulus (GPa)	Reference
Kenaf	1.45	1.6	930	53	Mohanty et al. (2005)
	—	—	250–600	14–39	Ochi (2008)
	1.2	—	400	—	Lloyd and Seber (1996)
	0.75	—	223	14	Shibata et al. (2005)
	—	1.2	200	13	Xue et al. (2009)
E-glass	2.5	0.5	2,000–3,500	70	George et al. (2001)
Unsaturated polyester	1.18	7.3	24	3.5	

these prepregs are cut into pieces with predetermined shapes and weights. The cut pieces are then stacked, assembled into a laminated prepreg, and compression molded at elevated temperatures and pressure.

Table 2 summarizes studies from the literature on fiber loadings of natural fiber–reinforced composites. These studies indicated that the tensile strengths and moduli of the composites generally increased as the fiber content increased. However, flexural strengths and moduli of the composites began to decrease or level off at 40 percent by volume or 30 to 40 percent by weight. The fabrication techniques used in these studies are on laboratory scale and are not feasible for high-volume production.

The primary objectives of this study were to explore the maximum fiber loading in KBFB/UPE composites and to develop fiber preforming techniques to increase fiber loadings. The ultimate goal is to develop a continuous cost-effective production process for manufacturing high KBFB loading/UPE composites using the process variables developed in this study.

Materials and Methods

Materials

The raw materials used in this study consisted of KBFBs, UPE, styrene, catalysts, binders, and a few additives. Mechanically retted KBFBs were supplied by Kengro Corporation (Charleston, Mississippi). The measured tensile strength and elastic modulus of dry KBFB were 260 MPa and 19.2 GPa, respectively. The UPE (Aropol Q-6585) was provided by Ashland Chemical Company. The measured tensile strength and modulus of the cured UPE, mixed with the formulation used in this work, were 24 MPa and 3.5 GPa, respectively. Styrene (purity, >99%) was purchased from Fisher Scientific, and a *t*-butyl perbenzoate catalyst was supplied by AkzoNobel Corporate. A polyvinyl acetate emulsion (PVAc) adhesive with 46 percent (wt/wt) solids content, provided by Tailored Chemical, was used as a binder for making mat preforms.

Composite fabrication

The composite fabrication process consists of four main steps.

Fiber bundle preparation.—The long KBFBs were ground into short KBFBs using a Thomas Wiley Mill (Model 4) with a 6-mm sieve. Then the majority of the undersized fibers were removed using a vibrating 30-mesh screen. The average length of the resultant short KBFBs was 3.3 mm (±1.5 mm) over 20 measurements, equivalent to an average aspect ratio of 43. The measured moisture content of KBFBs was approximately 10 percent.

Mat preforming.—The KBFB preforming process is illustrated in Figure 1. First, 100 g (100 parts) of KBFBs were dispersed evenly and randomly by hand into a 381-mm-wide by 432-mm-long wooden forming box placed on a stainless steel sheet (Fig. 1a). This sheet had been precoated with a nonstick mold release agent. Then 12.5 g of PVAc adhesive (46% [wt/wt] solid content) were diluted with additional 37.5 g (37.5 parts) water. The diluted 50 g (5.75 parts of solid and 44.25 parts of water) PVAc was sprayed on the loose KBFB mat's upper surface after the wooden frame was removed (Figs. 1b and 1c). Next, after covering the sprayed surface with another steel sheet (Fig. 1d), the whole stack was turned upside down, and the upper steel sheet was removed (Fig. 1e). Another layer of diluted 50 g of PVAc was then sprayed on the upper surface (Fig. 1f). Now, the KBFBs were pressed into a 381 by 432 by 3-mm^3 preformed KBFB mat under a pressure of 0.15 MPa at a temperature of 175°C for 4 minutes. Finally, eight 102 by 178 by 3-mm^3 mat sections (Fig. 2) were cut from this full-size mat. The average weight per section was 11.3 g (10 parts of fibers and 1.3 parts of solid PVAc). Table 3 shows each ingredient weight of a full-size mat for each step and a single dry mat. The target dry fiber weight of a single 102 by 178-mm^2 mat was 10 g.

UPE resin application.—The UPE resin and its ingredients were mixed using a stirrer using the following formulation: 65 parts of UPE, 10 parts of styrene, 0.05 part of inhibitor, and 1.5 parts of peroxide catalyst by weight. The viscosity of the prepared UPE resin was measured to be 335 cP (25°C). After being mixed thoroughly, an excess amount liquid resin mixture was transferred to a spray gun.

The preformed mats were dried in an oven at 103°C for over 3 hours before resin application. After they were removed from the oven, the mats were placed on a balance, and the resin application was performed immediately to avoid the KBFB's moisture uptake. The predetermined amount of resin was sprayed on the mats. Figure 3 shows a prepreg schematic of five stacked mats and the corresponding amount of resin by weight. The resin penetrated into the mats smoothly and without apparent difficulty but probably somewhat nonuniformly. Nevertheless, the initial resin distribution was not an issue because during hot pressure curing, the resin flowed readily throughout the mats becoming well distributed prior to curing. This was determined from multiple scanning electron microscopy (SEM) observations of composite cross sections. The average weight of each ingredient for the preforming processes and the desired fiber loading prior to compression molding is summarized in Table 4.

Compression molding.—Each prepreg was placed in a mold precoated with a silicone mold release agent. The platens were preheated to 175°C. The pressure was raised to 5 MPa on these prepregs within 10 seconds, and then the press's heating switch was turned off. Since the final laminate composite obtained only was 3 to 4 mm thick, heat

Table 2.—A list of studies on fiber loadings of natural fiber–reinforced composites.

Fiber type	Matrix	Fiber loading (%)	Source
Jute	Polyester	60, vol/vol	Roe and Ansell (1985)
Banana	Polyester	48, wt/wt	Zhu et al. (1995)
Pineapple	Polyester	40, wt/wt	Devi et al. (1997)
Hybrid ramie-cotton	Unsaturated polyester	60, vol/vol	Paiva Júnior et al. (2004)
Flax	Polyester	37.5, vol/vol	Baiardo et al. (2004)
Banana and sisal	Unsaturated polyester	50, vol/vol	Idicula et al. (2005)

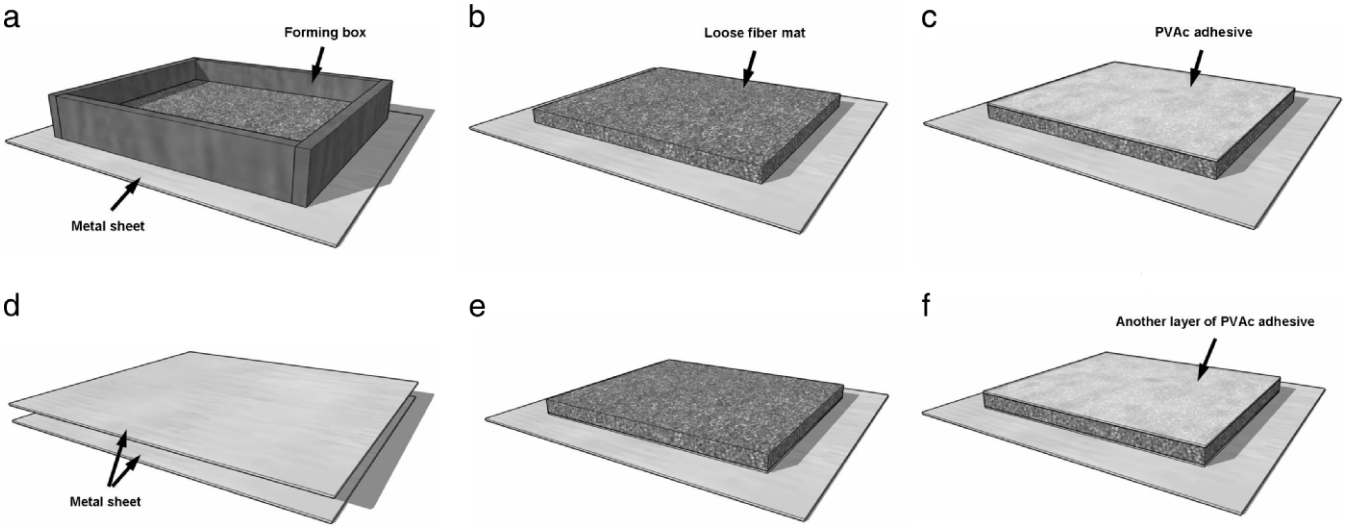


Figure 1.—Fabrication of a polyvinyl acetate (PVAc) preformed mat: (a) ground fiber bundles were dispersed on a metal sheet within a wooden frame, (b) a loose fiber mat before PVAc was applied, (c) one layer of PVAc was sprayed on the top surface, (d) another metal plate was placed to cover the loose mat, (e) the stack was turned upside down, and (f) another layer of PVAc was sprayed on the top surface.

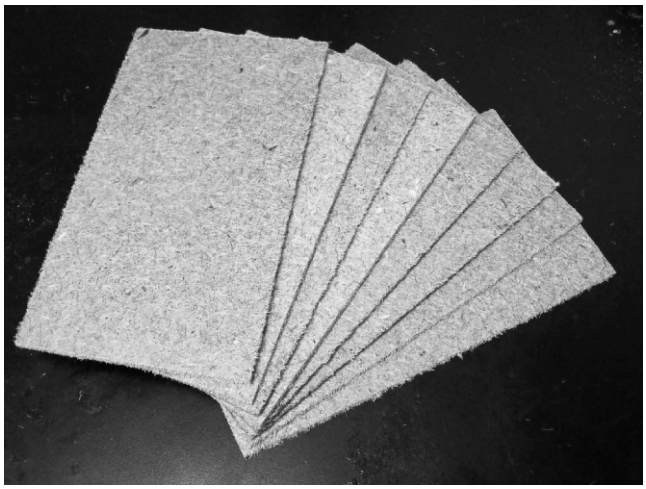


Figure 2.—Preformed 102 by 178 by 3-mm^3 kenaf bast fiber bundle mats.

transfer throughout the curing stack was very fast. The composite was maintained at this pressure for 1 hour. The pressure was released when the mold had cooled to about 100°C. At this point, each composite panel was removed from the mold. The fiber loadings of the resultant composites were actually higher than the fiber loadings expected based on the initial weighed amounts of the components because small amounts of UPE resin were squeezed out along the four lateral edges of the composites during compression molding. Therefore, the weight and size of each composite panel were measured after curing and trimming away this squeezed out resin (no fibers from the mats were squeezed out). Then the actual fiber loading was recalculated. The four resultant KBFB/PVAc/UPE (KPU) composites are shown in Figure 4.

Tensile testing

Twenty dog-bone tensile specimens were cut from the KPU composite panels. All specimens were tested on an Instron 5869 Universal Testing Machine in accordance with ASTM Standard D638-03 (American Society for Testing and Materials 2004). The tensile strain was recorded by an Instron 2630-100 series extensometer. The testing speed was set at 5 mm/min.

SEM observations

Micrographs of composite cross sections were examined to observe resin penetration into the mat and coverage on fiber bundles. Two types of specimen cross sections were prepared: representative fracture surfaces of tested tensile specimens and smooth surfaces cut using a Leica Ultracut E Ultramicrotome. Specimens were mounted on aluminum stubs with carbon tape and coated with gold-palladium in a Polaron E 5100 Sputter Coater for 60 seconds. SEM images

Table 3.—Average weight of each ingredient in a full-size and single kenaf bast fiber bundle mat.

	Weight (g)				
	Fiber (10% moisture content)		PVAc (48% solid content)		
Mat	Solid	Water	Solid	Water	Total
Wet	91	9	11.5	88.5	200
Dry (381 by 432 mm^2)	91	0	11.5	0	102.5
Single (102 by 178 mm^2)	10	0	1.26	0	11.26

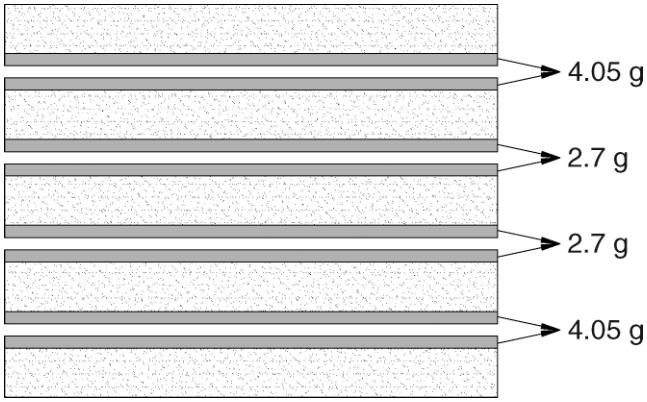


Figure 3.—A schematic of the stacking pattern of preformed kenaf bast fiber bundle mats and the corresponding amounts of unsaturated polyester resin applied on each mat.

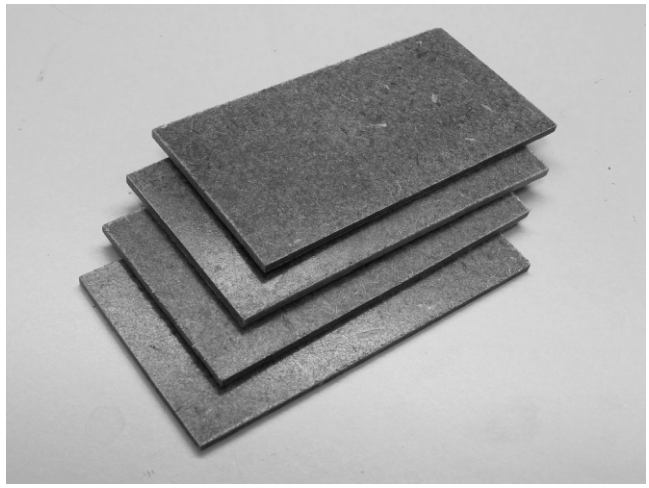


Figure 4.—Kenaf bast fiber bundle/unsaturated polyester composites.

were taken on a Carl Zeiss SMT EVO 50 instrument (EHT; 5 to 15 kV).

Results and Discussion

Fiber loading

The mean values of measured fiber loadings of resultant composites are summarized in Table 4. The fiber loadings of KPU composites ranged from 64.4 to 67.1 percent (wt/wt) with an average value of 65.3 percent (wt/wt). Thus, the composite fiber loading was successfully increased up to a nominal value of 65 percent (wt/wt) using the fabrication techniques explored in this study.

The SEM micrographs of the KPU composite cross sections (Figs. 5a and 5b) show good resin penetration throughout the specimen thickness and uniform resin wetting on fiber bundle surfaces. Close inspection of Figures 5a and 5b reveals the presence of a few small voids.

The PVAc adhesive binds KBFBs together to form a loose preformed solid mat at a low pressure. These mats are strong enough to ensure easy handling in subsequent procedures, such as resin application and molding. Nevertheless, resin can still freely flow into the voids of the loose mat during resin application and compression molding. The preforming process ensures uniform properties in the resultant composite and speeds the molding cycle. Thicker composites can be produced by increasing the number of preformed mats in the laminate. Composites with lower fiber loadings can be made by increasing the amount of UPE resin added to each fiber mat. Then compression at the same pressure and temperature will result in thicker composites with lower fiber weight fractions.

Physical and tensile properties

Table 5 summarizes the physical and mechanical properties of the KPU composites fabricated in this study, mechanical property data of SMCs from a SMC/BMC design manual (European Alliance for SMC 2001), and specification requirements for glass fiber/UPE composites from an automotive part manufacturer. The densities of KPU (1.22 g/cm^3) composites are significantly lower than those of glass fiber/UPE composites (1.8 to 2.15 g/cm^3). This could result in a large percentage of weight savings if the same volume of each composite could be used in finished products.

The average elastic modulus of the KPU composites (12.1 GPa) was 21.0 percent higher than the lower limit of the SMC products (10 GPa). Moreover, the average elastic modulus of KPU composites was 34.4 percent higher than that of the manufacturer's specification requirements (9 GPa). Similarly, the specific modulus of KPU (9.92 GPa·cm^3/g) was 78.7 percent higher than the lower bound for SMCs (5.55 GPa·cm^3/g) and 104 percent higher than the specification requirement (4.86 GPa·cm^3/g). The KPU composite's tensile strength (54.6 MPa) was lower than the lower bound for the SMCs (65 MPa) and is just below the manufacturer's specifications (58 MPa; Table 5). However, the specific strength of the KPU composite (44.8 MPa·cm^3/g) was still attractive compared with that of SMCs (36.1 MPa·cm^3/g) and the manufacturer's requirement (31.4 MPa·cm^3/g).

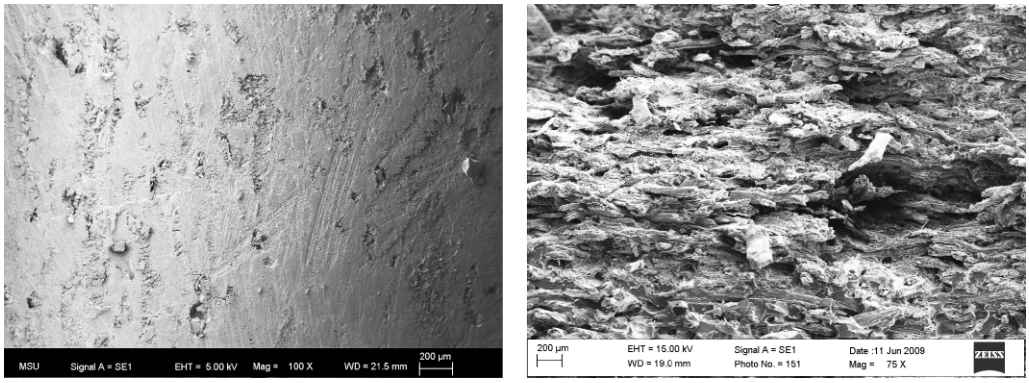
Failure mechanisms

SEM images of tensile specimen fracture surfaces of the KPU composites (Fig. 6) show four typical failure modes of the KBFB/UPE composites: fiber splintering tension breakage, fiber brash tension breakage, fiber/matrix debonding, and fiber pullout. The strength of fiber-reinforced composites is a complex function of the material and geometric inhomogeneities, fiber-to-matrix adhesion, fiber/

Table 4.—Weights of each ingredient in a laminated prepreg and its desired and calculated actual fiber loading for each composite.

Weight (g)					Fiber loading (%, wt/wt)	
Fiber	PVAc	UPE	Prepreg	Composite	Desired	Actual
50	6.3	27	83.3	76.8	60	65.3 (1.88)[a]

[a] Coefficient of variation is presented in parentheses.



a b

Figure 5.—SEM images of composites at different magnification: (a) cross section smoothed by microtome and (b) a tensile specimen fracture surface.

Table 5.—Mean values of physical and mechanical properties of tested composites and specifications of glass fiber–reinforced unsaturated polyester composites.

Property	KPU[a]	SMCs[b]	Specific requirement[c]
Density (g/cm^3)	1.22	1.8–2.15	1.85 ± 0.05[d]
Elastic modulus (GPa)	12.1	10–19.1	9
COV (%)[e]	21		
Specific modulus (GPa·cm^3/g)	9.92	(5.56–8.88)	(4.86)
Tensile strength (MPa)	54.6	65–204	58
COV (%)	8		
Specific strength (MPa·cm^3/g)	44.8	(36.1–94.9)	(31.4)
Elongation (%)	0.62		
COV (%)	22		

[a] KPU = Kenaf/PVAc/UPE composites.
[b] SMCs = sheet molding compounds (contains filler, unsaturated polyester, and 25% to 50% [wt/wt] glass fiber). Values in parentheses are calculated from the lower limit of the properties range (SMC Automotive Alliance 1991).
[c] Specification requirements for glass fiber/UPE composites (fiber content, 25% to 30%) from an automotive part manufacturer. Values in parentheses are calculated by properties and densities by requirement.
[d] Mean ± standard deviation.
[e] COV = coefficient of variation.

matrix interphase structure, fiber volume fraction (Kaw 2006), resin strength, fiber strength, and variability of fiber strengths (US Department of Defense 1999), among other factors. Such factors govern crack initiation, crack growth, and the absorption of energy by damage accumulation prior to failure. Usually, there is inherent scatter in composite strength measurements due to the stochastic nature of the failure process as well as bifurcations between local stress state–dependent failure mechanisms.

The first failure mode observed in this work (Fig. 6a) is attributed to fiber fractures. The latter mechanism (Fig. 6b) results from moderate interfacial matrix-to-fiber adhesion. All fracture surfaces ran across (through) the laminates, and no delamination fractures were observed. It appears that the tensile strengths of these composites are dominated by both (1) the UPE matrix-to-fiber bundle interfacial bonding and (2) the fiber bundle tensile strengths. Hence, improving the interfacial adhesion in this class of composites is one of the ways to improve composite strength (Misra et al. 2002, Aziz and Ansell 2004, Baiardo et al. 2004, John and Naidu 2004, Baley et al. 2006).

Voids and gaps (Figs. 6a and 6b) between the fibers and the matrix were observed. Clearly, eliminating voids and improving UPE matrix-to-fiber adhesion still needs improvement. These observations suggest that low tensile strengths could be attributed to voids and limited fiber-to-

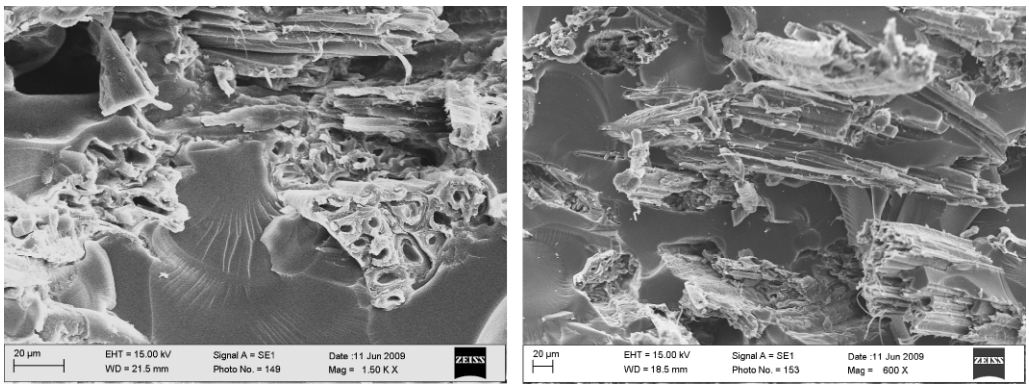


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Figure 6.—Tensile fracture surfaces of kenaf/PVAc/UPE (KPU) composites: (a) fiber tension breakage and (b) fiber pullout breakage.

matrix adhesion. If reduced void content and enhanced interfacial bonding can be achieved through improved processing techniques, natural fiber composites similar to those developed in this work should have mechanical properties competing against those of SMCs.

Summary and Conclusions

A composite fabrication process for high KBFB loadings in an UPE matrix has been explored for automotive part applications. This process had four major steps: (1) short KBFBs preparation, (2) KBFB mat preforming using a PVAc emulsion adhesive, (3) UPE resin application, and (4) laminate compression molding.

This process provided relatively good resin coverage on KBFB surfaces for average fiber loadings up to 65 percent (wt/wt). Such high fiber loadings could substantially reduce raw material and manufacturing costs in comparison with traditional automotive composites while also increasing mechanical properties.

KPU composites fabricated using this process had higher elastic moduli and tensile strengths that were close to specification requirements for glass fiber–reinforced sheet molding compounds. These composites had lower densities versus glass fiber/UPE composites and provided favorable specific moduli and strengths. SEM images of KBFB-reinforced UPE composites suggest that further improvements in tensile properties may be achieved by enhancing fiber bundle/resin adhesion and reducing void content. This is a focus for future work. This study demonstrates that KBFB-reinforced UPE composites have the potential to be a low-cost alternative to glass fiber–reinforced UPE composites for automotive applications.

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