An applied investigation of kenaf-based fiber/polymer composites as potential lightweight materials for automotive components

Yicheng Du

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AN APPLIED INVESTIGATION OF KENAF-BASED FIBER/POLYMER
COMPOSITES AS POTENTIAL LIGHTWEIGHT MATERIALS
FOR AUTOMOTIVE COMPONENTS

By
Yicheng Du

A Dissertation
Submitted to the Faculty of
Mississippi State University
in Partial Fulfillment of the Requirements
for the Degree of Doctor of Philosophy
in Forest Resources
in Department of Forest Products

Mississippi State, Mississippi
August 2010
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By

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AN APPLIED INVESTIGATION OF KENAF-BASED FIBER/POLYMER COMPOSITES AS POTENTIAL LIGHTWEIGHT MATERIALS FOR AUTOMOTIVE COMPONENTS

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Natural fibers have the potential to replace glass fibers in fiber-reinforced composite applications. However, the natural fibers’ intrinsic properties cause these issues: 1) the mechanical property variation; 2) moisture uptake by natural fibers and their composites; 3) lack of sound, cost-effective, environment-friendly fiber-matrix compounding processes; 4) incompatibility between natural fibers and polymer matrices; and 5) low heat-resistance of natural fibers and their composites.

This dissertation systematically studied the use of kenaf bast fiber bundles, obtained via a mechanical retting method, as a light-weight reinforcement material for fiber-reinforced thermoset polymer composites for automotive applications.

Kenaf bast fiber bundle tensile properties were tested, and the effects of locations in the kenaf plant, loading rates, retting methods, and high temperature treatments and their durations on kenaf bast fiber bundle tensile properties were evaluated.

A process has been developed for fabricating high fiber loading kenaf bast fiber bundle-reinforced unsaturated polyester composites. The generated composites
possessed high elastic moduli and their tensile strengths were close to specification requirements for glass fiber-reinforced sheet molding compounds.

Effects of fiber loadings and lengths on resultant composite’s tensile properties were evaluated. Fiber loadings were very important for composite tensile modulus. Both fiber loadings and fiber lengths were important for composite tensile strengths.

The distributions of composite tensile, flexural and impact strengths were analyzed. The 2-parameter Weibull model was found to be the most appropriate for describing the composite strength distributions and provided the most conservative design values. Kenaf-reinforced unsaturated polyester composites were also proved to be more cost-effective than glass fiber-reinforced SMCs at high fiber loadings.

Kenaf bast fiber bundle-reinforced composite’s water absorption properties were tested. Surface-coating and edge-sealing significantly reduced composite water resistance properties. Encapsulation was a practical method to improve composite water resistance properties. The molding pressure and styrene concentrations on composite and matrix properties were evaluated. Laser and plasma treatment improved fiber-to-matrix adhesion.
DEDICATION

Dedicate to my parents, Xingcai Du and Cuiping Sun, sister Lili Du, and brother-in-law Jianfeng Xu.
ACKNOWLEDGEMENTS

I would like to express the deepest appreciation and respect to my committee chair, Dr. Jilei Zhang. He continually and convincingly conveyed a spirit of adventure in regard to research. In addition, he imparted to me the principles of how to behave and carry out research professionally.

Special thanks were given to co-advisor Dr. Yibin Xue, and Dr. Thomas E. Lacy Jr., Dr. Charles U. Pittman Jr., Dr. Hossein Toghiani, and Dr. Mark F. Horstemeyer, who have served as my committee members, for their help in reviewing papers, teaching English, and directing the research.

I am very grateful to Dr. Hamid Borazjani, Dr. Susan Diehl, Dr. Steve Elder, and Dr. Chuji Wang for providing access to their labs and equipments, Franklin Quin, Bob Tackett, Dr. Fei Yu, George Miller, David Butler, and Linda Sites for their help in the research.

I also express special gratitude to Kengro Corporation, Ashland Chemicals, and AkzoNobel Corporate for donating materials, Mr. William A. Monroe for SEM photographing, and Mr. Lou Dodyk, the Manager of Meridian Automotive Systems, for his consistent technical support and providing technical data.

Finally, the author appreciates the financial support by the US Department of Energy (Grant No.: DE-FC26-06NT42755), the Center for Advanced Vehicular Systems, and department of forest products at Mississippi State University.
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<td>ASTM</td>
<td>American Society for Testing and Materials</td>
</tr>
<tr>
<td>CoNa</td>
<td>cobalt naphthenate</td>
</tr>
<tr>
<td>KBFB</td>
<td>kenaf bast fiber bundle</td>
</tr>
<tr>
<td>KPU</td>
<td>kenaf-PVAc-unsaturated polyester composite</td>
</tr>
<tr>
<td>KPV</td>
<td>kenaf-PVAc-unsaturated vinyl ester composite</td>
</tr>
<tr>
<td>MAPP</td>
<td>maleic anhydride grafted polypropylene</td>
</tr>
<tr>
<td>MC</td>
<td>moisture content</td>
</tr>
<tr>
<td>MEKP</td>
<td>methyl ethyl ketone peroxide</td>
</tr>
<tr>
<td>PRF</td>
<td>phenol-resorcinol-formaldehyde</td>
</tr>
<tr>
<td>PVAc</td>
<td>polyvinyl acetate</td>
</tr>
<tr>
<td>ROM</td>
<td>rule of mixture</td>
</tr>
<tr>
<td>SEM</td>
<td>scanning electron microscope</td>
</tr>
<tr>
<td>SMC</td>
<td>sheet molding compound</td>
</tr>
<tr>
<td>TBP</td>
<td>t-butyl perbenzoate</td>
</tr>
<tr>
<td>TGA</td>
<td>thermogravimetric analysis</td>
</tr>
<tr>
<td>UPE</td>
<td>unsaturated polyester</td>
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<tr>
<td>VE</td>
<td>vinyl ester</td>
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1.1 Background

Driven by environmental awareness, natural fibers have gained increasing acceptance as reinforcing materials for fiber-reinforced composites, due to their low density, high specific strength and stiffness, low-cost, renewability, and biodegradability. Based on location in a plant, natural fibers for reinforcement may be divided into 3 types (Mohanty 2005): bast (flax, hemp, jute, and kenaf), leaf (sisal, henequen, pineapple, and banana), and fruit or seed (cotton, kapok, and coir).

These natural fibers have the potential to replace petroleum-based or man-made fibers (such as glass fibers) in automotive structural parts applications. Automotive components made from light-weight natural fiber-reinforced composites can significantly reduce vehicle overall weight, which results in energy saving and low emission to the air.

1.2 Issues

Though automotive parts made from natural fiber-reinforced polymer composites have many benefits as compared to glass fibers, there are still several major technical issues which need to be addressed before the automotive industry gains full confidence to enable wide-scale acceptance, especially in the application of automotive exterior parts. These issues include: 1) variability of physical and mechanical properties of natural fibers, which results in larger variation of its composite’s properties; 2) relatively high moisture uptake by natural fibers and their composites which will affect composite
fabrication processes and final product properties; 3) lack of sound fiber-matrix compounding process (distributing fibers evenly within the matrix); 4) incompatibility between hydrophilic natural fibers and the hydrophobic polymer matrices; and 5) low heat-resistance of natural fibers and their composites. These problems need to be investigated, understood, and minimized before potential applications, structural parts and/or semi-structural parts, can be identified.

1.3 Literature review

1.3.1 Kenaf

Kenaf (Hibiscus cannabinus L.) is an annual herbaceous plant originally from Africa (Lloyd and Seber 1996). It is a newer crop to the United States. Kenaf is mainly cultivated in following southern temperate regions: Mississippi, Texas, California, Louisiana, New Mexico, and Georgia. It has a growing period of 90-150 days and may grow to 2.4 - 6 m in height. Its single, straight stem consists of an outer fibrous bark and an inner woody core that yield two distinct bast and core fibers respectively. The bast fiber constitutes about 26-35 wt% (weight percentage) of its stem, and genetic strains have been developed which yield 35 wt% or greater bast portions. The harvested kenaf stems are usually first decorticated to separate the bark from the core producing ribbons of kenaf bast 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). Drying is achieved by leaving the crop standing in the field. Presently, applications of kenaf products include paper pulp, cordage, grass, erosion mats, animal bedding, oil sorbents, potting media, animal litter, insulation boards, soilless sod, fillers for plastics, textiles, and livestock feed (Webber III et al. 2002).
1.3.1 Retting methods

Retting is a process which breaks down the chemical bonds that hold the bast fibers together using moisture, microorganisms, or chemistry. Five commonly used retting methods are water retting, dew retting, chemical retting, enzymatic retting, and mechanical retting.

1.3.1.1 Water retting

Water retting is also known as bacterial retting. The kenaf stalks are soaked in water at 30±2°C for 10 days, in which the action of anaerobic microorganisms hydrolyzed carbohydrates matrix and freed cellulose fibers (Morrison and Akin 1996). Then the stalks are washed in hot tap water until all the shiny, slippery residues are removed. After that they are air dried and are combed with a soft nylon brush to obtain uniform and high quality fibers (Ramaswamy 1994). The advantage of water retting is that it is easily to conduct and needs fewer treatments. However, due to several unsolved problems, e.g. contaminated water discharge (Fuller et al. 1946), time consuming and unpleasant working conditions due to odor, water retting has been largely abandoned in countries where labor is expensive or environmental regulations are enforced (Ehrensing 1998).

1.3.1.2 Dew retting

Dew retting, also called field retting, is used extensively for hemp and flax. This process doesn’t need water (Ehrensing 1998). During the dew retting process, stems are laid in the field so that rain or dew is used to keep the stems moist. The dew retting may take up to 2-5 weeks, depending on the environmental humidity and temperature. Dew retting is a geographical and weather-dependent method, which is restricted to
geographical regions with appropriate climate, and the resulting fibers are often inconsistent in quality with significant amounts of dirt and contaminants (Sharma 1988, Van Sumere 1992).

1.3.1.3 Chemical retting

Kenaf can also be chemically retted, where the kenaf is soaked in an alkali solution such as sodium hydroxide (Guzman et al. 1982, Ramaswamy et al. 1994, Morrison 1996). Chemical retting is faster than water retting. In this process, it only takes several hours for kenaf to be separated. However, chemical retting not only has a weakening affect on the fiber, and it also poses a potential ecological threat if precautions for neutralization are not taken (Abbot and Paterson 1985, Ramaswamy et al. 1994).

1.3.1.4 Enzymatic retting

Enzymatic retting uses enzymes to separate the fibers. Quality fibers with higher strengths can be obtained using this method. Enzymatic retting has been done on flax, but no study has been found in processing kenaf. Enzymatic retting is preferred over water and chemical retting for environmental reasons (Akin et al. 2000, Adamsen et al. 2002).

1.3.1.5 Mechanical retting

Mechanical retting, also known as green retting, is a simple and cost-effective method. The raw materials for this procedure can be field-dried, slightly retted plant straws, or technically dried straws. The bast fibers are mechanically separated from the woody part. This retting method is independent of weather conditions, and doesn’t need water, chemicals or any other additives; however, the fiber quality is not as fine as those retted with other methods.
1.3.1.2 Structure and chemistry of kenaf bast fiber bundles

In general, the kenaf bast fibers are hollow tubes averaging 2.6 mm in length, 21 μm in diameter with an average length/diameter aspect ratio of 124, very similar to softwood species. The core fibers, with an average length of 0.5 mm, closely match those of hardwoods (Lloyd and Seber 1996).

The major constituents of kenaf bast fiber bundles (KBFB) are cellulose, hemicellulose and lignin. The amount of each constituent can vary significantly due to cultivation environments, geographic origins, age, locations in the plant (from root to tip), and retting and separating techniques. 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 (31-39 wt%) and higher lignin (15-19 wt%) amounts. Rowell et al. (2000) reported 44-57 wt% cellulose, and 15-19 wt% lignin.

1.3.1.3 Mechanical properties of kenaf fibers and fiber bundles

Table 1.1 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 fibers. E-glass fibers had a higher specific strength, but kenaf had a higher specific Young’s modulus (36 GPa·cm³/g) than that of E-glass (28 GPa·cm³/g).
Table 1.1  Physical and mechanical properties of kenaf bast fiber bundles, commercial E-glass fibers, and unsaturated polyester resin.

<table>
<thead>
<tr>
<th>Fiber</th>
<th>Density (g/cm³)</th>
<th>Elongation (%)</th>
<th>Tensile strength (MPa)</th>
<th>Elastic modulus (GPa)</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kenaf</td>
<td>1.45</td>
<td>1.6</td>
<td>930</td>
<td>53</td>
<td>Mohanty et al. 2005</td>
</tr>
<tr>
<td></td>
<td>-</td>
<td>-</td>
<td>250-600</td>
<td>14-39</td>
<td>Ochi 2008</td>
</tr>
<tr>
<td></td>
<td>1.2</td>
<td>-</td>
<td>400</td>
<td>-</td>
<td>Lloyd and Seber 1996</td>
</tr>
<tr>
<td></td>
<td>0.75</td>
<td>-</td>
<td>223</td>
<td>14</td>
<td>Shibata et al 2005</td>
</tr>
<tr>
<td></td>
<td>-</td>
<td>1.2</td>
<td>200</td>
<td>13</td>
<td>Xue et al. 2009</td>
</tr>
<tr>
<td>E-glass</td>
<td>2.5</td>
<td>0.5</td>
<td>2,000-3,500</td>
<td>70</td>
<td>George et al. 2001</td>
</tr>
<tr>
<td>Unsaturated polyester</td>
<td>1.2-1.5</td>
<td>2</td>
<td>40-90</td>
<td>2-4.5</td>
<td>Mohanty et al. 2005</td>
</tr>
</tbody>
</table>

1.3.1.4 Factors affecting kenaf fiber and fiber bundle properties

The mechanical properties of kenaf fibers were studied and reported by Lloyd and Seber (1996), Mohanty et al. (2005), Ochi (2008), and Shibata et al. (2005). These properties vary significantly depending on cultivation environment, geographic origin, age, location on the plant (from root to tip), retting and separating techniques, cellulose/hemicellulose/lignin content, moisture content, temperature, and etc. Subsequently, these variations are likely to affect the physical and mechanical properties of the kenaf fiber-reinforced polymer composites.

1.3.1.4.1 Cultivation environments and geographic origins

Ochi (2008) investigated the effect of two different growth conditions (A and B) on the tensile strength of KBF Bs tested at a strain rate of 0.04/m. The growth locations of A (at an average temperature of 22°C) and B (30°C) were the Miyagi Prefecture and the Ehime Prefecture in Japan, respectively. The tensile strength and elastic modulus of kenaf grown at B (600 MPa and 39 GPa) were significantly greater than those grown at A (250 MPa and 14 GPa).
1.3.1.4.2 Fiber locations on the plant

Ochi (2008) investigated the effect of locations of the kenaf bast fibers on the plant (from root to tip) on the tensile strength. The tensile strength of fiber bundles from the upper portion was 80% of that from the bottom portion. Fiber bundles from the bottom section of the plant showed the tendency to have the greatest values of tensile strengths.

Ramaswamy et al. (1995) cut different kenaf fiber bundle sections from five locations along stems from the base to the tip, retted them by bacterial and chemical processes to extract fibers, and compared their bundle strengths. The location of fibers on the kenaf stalk had effects on the bundle breaking tenacity. The fiber bundle breaking tenacity increases as the location changes from the tip to the base. The breaking tenacity values were 23.0, 23.8, 24.8, 28.7, and 32.7 g/tex for five locations from the base to the tip, respectively. Tip and base sections showed clear differences in phenolics and carbohydrate contents, which could be related to maturity. Breaking strengths of bacterially retted KBFBs was significantly greater for the tip than for the base, with no difference in elongation between regions. This is opposite to the result from experimental results of Ochi (2008).

1.3.1.4.3 Retting processes

Ramaswamy et al. (1994) retted decorticated kenaf stalks by bacterial and chemical processes and compared their bundle strengths. Fiber bundle strengths were calculated according to the following equation:

\[
\text{Fiber bundle tenacity (g/tex)} = \frac{\text{breaking load (g) } \times \text{ length of bundle (mm)}}{\text{weight of bundle (g) } \times 1,000,000}
\]  
[1-1]
Kenaf bundle breaking strength was significantly lower for chemically retted fibers (12.9g/tex) as compared with bacterially retted fibers (28.2g/tex). This may be due to the significant reduction in gum contents caused by chemical retting. It was suggested that a method that combined bacterial and chemical processing might have some merits.

1.3.1.4.4 Environmental variables

Limited information is available related to high-temperature, especially its long duration effects on mechanical properties of kenaf fibers, kenaf bast fiber bundles, and its composites. Available information about temperature effects on mechanical properties were found mostly for solid wood and wood composites but not for kenaf. This wood literature is reviewed and documented below for future reference.

Schaffer (1973) summarized the thermal changes, reported by many researchers, in dry wood in a non-oxidative atmosphere. The natural lignin structure was altered and hemicelluloses began to soften at 55 °C. Lignin slowly began to lose weight at 110 °C. At 120 °C, the hemicellulose content began to decrease, α-cellulose began to increase, and lignin began to soften. Lignin was melted and began to reharden at 160°C. At 180 °C, hemicellulose began to lose weight rapidly, lignin in the torus structures flowed, and wood began to lose weight rapidly. At 210 °C, lignin hardened, cellulose softened and depolymerized. Cellulose dehydrated above 210 °C. Cellulose crystallinity decreased and recovered at 225 °C. At 280 °C cellulose began to lose weight, and a temperature of 288 °C was assumed to be wood charring temperature. At 370 °C, cellulose had lost 83 wt% of initial weight, and wood was highly charred.

Results from thermogravimetric analyses (TGA) of hemp, flax, and linseed fibers in atmospheric air (Munder and Hempel 2006) indicated that there were 2 phases of mass
loss under the influence of temperatures. The change of mass was caused by evaporation of components, thermal degradation, or oxidation. The first phase of mass loss from the base fibers started at 30 °C and ended at a temperature of about 110 °C. The mass of the fiber sample was relatively constant in the temperature range from 110 to 170 °C. This meant the fibers were thermally stable in this range. Thermal degradation started above 170 °C. The thermogravimetry curve decreased steadily above the temperature of 175 °C. This steady decrease was caused by an increasing reaction rate. The maximum rate was in the range from 300 to 350 °C. The total mass loss which had occurred above 350 °C was approximately 60 wt%.

LeVan and Winandy (1990) reported that when wood was heated, chemical bonds began to break down at about 175 °C, and these reactions accelerated as the temperature increased. Above 200 °C, the carbohydrates break down yielding tars and flammable volatiles. The degradation of wood can be viewed as the sum of the degradation of its components. Lignin pyrolyzes at a slower rate than holocelluloses (α-cellulose plus the hemicellulose), but the degradation period begins earlier than that of holocelluloses. Below 350 °C, the wood began to degrade at slightly lower temperatures than alpha-cellulose. This lower degradation temperature of wood was primarily due to the hemicellulose. Celluloses decomposed in the temperature range from 260 to 350 °C, and hemicelluloses degraded in the approximate range of 200 to 260 °C. Dehydration reactions occurring above 200 °C were the primary process of lignin thermal degradation.

The influence of temperature on the modulus of elasticity (MOE) of Pinus sylvestris L. in a range from 25 to 300 °C was investigated by Moraes et al. (2004) using TMA. They reported that the MOE decreased from 100 °C onwards and this behavior could be explained by the softening and degradation of the wood polymers.
It was not the temperature alone which cause changes in the wood; there were additional factors which influence thermal degradation processes, e.g., time of treatment, atmosphere, pressure, and water content. Under certain conditions, changes in wood would be observed from 100 °C and above (Kollmann and Fengel 1965).

1.3.2 Polymer matrix

The role of a matrix in a fiber-reinforced composite is to: (1) transfer stress between fibers; (2) provide a barrier against an adverse environment; and (3) protect the surface of the fibers from mechanical abrasion (Mallick 1988). Polymeric matrices include thermoset and thermoplastic polymers. In this project, unsaturated polyester (UPE) was chosen as the matrix because of its advantages such as thermal stability, chemical resistance, less creep and stress relaxation, low price, and ease of processing.

UPE is generally manufactured by reacting unsaturated dibasic acids with saturated dihydric alcohols, or saturated dibasic acids with unsaturated dihydric alcohols, and then dissolving this mixture in a reactive monomer such as styrene (Mallick 1988). The styrene monomer and UPE can react to cure under the influence of heat or peroxide catalysts. The cured resin has good electrical properties, and has good resistance to corrosion and attack by chemicals.

1.3.2.1 Synthesis

Synthesis of unsaturated polyester is illustrated using an example of maleic anhydride and ethylene glycol. The maleic anhydride reacts with the glycol to form a UPE long chain and n-1 water molecules (Gu 1999).
This resultant polyester resin is very viscous solid or semisolid. In order to obtain a moderate viscosity of the resin, a dilution agent is necessary. The most commonly used dilution is styrene monomer, which also serves as a cross-linking agent during curing process (Gu 1999, Mallick 1988).

1.3.2.2 Curing

The polyester will not polymerize itself. With the help of initiator or heating, UPE and the styrene can react to form a copolymer. Resin changes from liquid to cross-linked 3-dimension polymer. The reaction is illustrated as follow (Gu 1999):

```
\[
\begin{array}{c}
\text{CH-C-O} + \text{CH}_2\text{-OH} & \rightarrow \ H \left[ O-\text{CH}_2\text{-CH}_2\text{-O-CH=CH-C} \right]_{n}\text{OH} + (n-1)\text{H}_2\text{O} \\
\end{array}
\]
```

1.3.2.3 Initiator and accelerator

Organic peroxide is usually used as an initiator to improve curing of unsaturated polyester. Benzoyl peroxide, methyl ethyl ketone peroxide, and cyclohexanone peroxide are commonly used initiators. Benzoyl peroxide is efficient and easy to handle. It is soluble in the styrene monomer, and stable at room temperature. Benzoyl peroxide
initiates the cure of the resin. In the UPE resin, the percentage of initiator may range from 0.5 to 2 wt% depending on the type of resin and monomer used. Methyl ethyl ketone peroxide (MEKP) is another commonly used initiator. At room temperature, it does not lead to a full cure by itself; however, with the help of a cobalt compound accelerator, MEKP will cause gelation up to complete curing within short periods of time. Therefore, MEKP can be used for large or complicated units where heat application is not possible. Dimethylaniline and cobalt naphthenate are initiators for benzoyl peroxide and MEKP respectively (Lubin 1969).

1.3.3 Sheet molding compounds (SMC)

Sheet molding compounds (SMC) are the mostly used process for manufacturing glass fiber-reinforced UPE composite for automotive components. The composition includes reinforcement, filler, and other additives.

1.3.3.1 Constituents

1.3.3.1.1 Polymer

The primary thermoset resin used in SMC process is unsaturated polyester. The resin content varies from 20 to 27 wt%.

1.3.3.1.2 Reinforcement

Glass fibers are the most often used reinforcement. Glass fibers used for SMC are chopped into lengths of 13 to 50 mm. The amount can vary from 25 to 60 wt%.

1.3.3.1.3 Filler

Fillers are used to: 1) reduce mold shrinkage; 2) control compound plasticity; 3) increase stiffness; 4) improve surface quality; and 5) reduce cost. The most common
filler in the SMC process is calcium carbonate (Mallick 1988). Examples of other fillers are clay, mica, and glass micro-spheres.

1.3.3.1.4 Other additives

The percentages of all other ingredients range from 3 to 5 wt%. Additional ingredients such as low-profile additives, cure initiators, inhibitors, thickening additives, and mold release agents are used to enhance the performance or processing of the material (SMC automotive alliance 1991). Varying the type and percentage of the ingredients will result in variations in mechanical properties or processability (SMC automotive alliance 1991).

1.3.3.2 SMC manufacturing process

The process for making SMCs is illustrated in Figure 1.1 (European Alliance for SMC 2001). All ingredients, except the glass fibers, are mixed together to form a resin paste. The resin paste is transferred to a doctor box where it is deposited onto a moving carrier film passing directly beneath. Simultaneously, glass fiber rovings are fed into a rotary chopper above the resin-covered carrier film. The chopped fibers are deposited randomly on the resin-covered carrier film.
Downstream from the chopping operation, a second carrier film is coated with resin paste and is laid, resin side down, on top of the chopped fibers. This stage of the process creates a resin paste and glass fiber “sandwich” which is then sent through a series of compaction rollers where the glass fibers are wet out with the resin paste and excess trapped air is squeezed out. At the end of the compaction rollers, the SMC sheet is taken up on a storage roll.

It takes about 3 to 5 days to chemically thicken the SMC to reach the desired molding viscosity. SMC is cut into pieces of predetermined size and shape. The cut pieces are then stacked and assembled into a specified charge pattern. After this charge is placed on the heated mold surface, the mold is closed and the SMC material is compressed. Typical mold pressures for a low-profile SMC are around 5.5-8.3 MPa.
The molding temperatures range from 121 to 163 °C. Table 1.2 gives a comparison of mechanical properties for a typical low-profile SMC (R25), a more structural SMC (SMC R50), and steel (SAE 1008 low carbon/cold rolled) (SMC automotive alliance 1991).

Table 1.1 SMC properties comparison table (SMC automotive alliance. 1991)

<table>
<thead>
<tr>
<th>Property</th>
<th>SMC R25</th>
<th>SMC R50</th>
<th>Steel SAE 1008</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile strength (MPa)</td>
<td>65-90</td>
<td>124-204</td>
<td>330.7</td>
</tr>
<tr>
<td>Tensile modulus (MPa)</td>
<td>10-12.5</td>
<td>12.2-19.1</td>
<td>206.7</td>
</tr>
<tr>
<td>Flexural strength (MPa)</td>
<td>155-200</td>
<td>248-380</td>
<td>-</td>
</tr>
<tr>
<td>Flexural modulus (MPa)</td>
<td>8.5-14.0</td>
<td>11.6-16.4</td>
<td>-</td>
</tr>
<tr>
<td>Notched IZOD (J/m)</td>
<td>500-1000</td>
<td>725-1360</td>
<td>-</td>
</tr>
<tr>
<td>Specific gravity</td>
<td>1.8-2.0</td>
<td>1.85-2.15</td>
<td>7.86</td>
</tr>
</tbody>
</table>

1.3.4 Natural fiber-based polyester composites

1.3.4.1 Current fabrication processes

One of the main issues of using natural fibers as a reinforcement in the SMC process is the difficulty with fiber chopping and dispersion. Natural fibers are normally discontinuous and so it is difficult to automatically chop natural fibers with methods like those used for glass fibers. The dispersion of natural fibers in the resin is also difficult because most natural fibers such as hemp, kenaf, and flax can interlock which results in poorer final product performance (Liu et al. 2007).

A bio-composite sheet molding compound process was invented by Drzal et al. (2007). The manufacturing process was based on a traditional SMC process for making SMCs. Because the formatting of glass fibers and natural fibers are different, a natural fiber feeder system was designed to replace the chopped glass fiber feeder system. The natural fiber feeder system comprised a feed hopper, a twin screw feeder, a fiber distribution chute and a vibratory feeder. The resultant sheet material made from this
process looked very good and was ready for compression molding. The fiber loading documented is 35 wt%.

The other process is making natural fibers into a non-woven mat. A measured quantity of polymer resin is poured on the pre-weighed amount of fiber mats. Trapped air is squeezed out using a roller. This resin-infused fiber mat is then cured in a mold. This process is usually used in labs (Dhakal et al. 2007).

1.3.4.2 Process variables

Mehta et al. (2004) compressed and cured hemp fiber/unsaturated polyester composites at 0.55 MPa for 2 h at 100 °C followed by post-curing for 2 h at 150 °C to investigate the effect of novel sizing on the mechanical and morphological characteristics of the composite. A combination of natural fibers (sisal) and wollastonite whiskers has been used by Singh et al. (2003) as an alternative to glass fiber reinforcement in polyester dough moulding compounds. The compound casting was done by spreading doughy materials over the brass plate mould to a desired thickness and compressing in a hydraulic press at 100 °C with a pressure of 2 MPa.

The temperature used for molding fiber-reinforced UPE composites varies depending on the catalyst used. Different levels of molding pressure for fabricating natural fiber-reinforced composites were found in literature. However, limited literature was found for evaluating the effect of molding pressures on composite mechanical properties.
1.3.4.3 Methods to improve fiber-matrix interfacial bonding and moisture absorption resistance

A major disadvantage of lignocellulosic natural fibers is their hydrophilic nature, which makes them incompatible with non-polar polymers and poorly resistant to moisture. The poor compatibility results in lack of adhesion between the fibers and the matrix which, in turn, leads to low mechanical properties and poor moisture resistance (George et al. 2001).

Chemical surface modification of natural fibers is one of the ways to improve fiber-matrix adhesion. The basic mechanisms of improving fiber to matrix bonding are: 1) improvement of fiber wetting by the polymer; 2) formation of covalent bonds between both materials. The chemicals for treating natural fibers include: alkali (Valadez-Gonzalez et al. 1999, Agrawal et al. 2000, Aziz and Ansell 2003), silanes (Valadez-Gonzalez et al. 1998, Misra et al. 2006, Drzal et al. 2007), acetylation (Sreekala et al. 2000), maleated coupling agents (Gassan and Bledzki 1997, Rowell et al. 1999, Feng et al. 2001, Sameni et al. 2003) (for thermoplastic matrix); and isocyanates (Maldas et al. 1989).

In general, surface chemical treatment of fibers improves mechanical properties and water resistance properties simultaneously. Literature was found covering the treatment of natural fibers using chemicals like silane (Demir et al. 2006), maleated coupling agents (Misra et al. 2000), acid chloride (Y. Zhang et al. 2009), and potassium permanganate (Paul et al. 1997).

1.3.5 Tensile property modeling of short fiber reinforced composites

Table 1.3 chronologically lists the empirical and numerical equations which have been reported for predicting tensile properties of man-made fiber-reinforced composites.
These classical equations suggested that the tensile properties of fiber-reinforced composites were governed by the elastic constants of the reinforcing fibers and the matrix, fiber volume percentages, fiber aspect ratios, fiber orientations, and fiber array patterns based on the assumption that fibers and the matrix are perfectly bonded.

Table 1.2  Mostly cited classical equations for predict tensile modulus and strength for fiber reinforced composite

<table>
<thead>
<tr>
<th>Model</th>
<th>Fiber form</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rule of mixtures</td>
<td>Unidirectional; continuous</td>
<td></td>
</tr>
<tr>
<td>Modified rule of mixtures</td>
<td>Randomly; long straight thin fibers</td>
<td>Cox 1952</td>
</tr>
<tr>
<td>Nielsen-Chen</td>
<td>Random; fiber length above critical length</td>
<td>1968</td>
</tr>
<tr>
<td>Halpin-Tsai</td>
<td>Unidirectional; discontinuous</td>
<td>1969</td>
</tr>
<tr>
<td>Mori-Tanaka</td>
<td>Unidirectional; discontinuous</td>
<td>Nielsen 1970</td>
</tr>
<tr>
<td>Self-Consistent</td>
<td></td>
<td>Hill 1965</td>
</tr>
</tbody>
</table>

| Tensile strength          |                                     |             |
| Rule of mixtures          | Unidirectional; continuous          |             |
| Kelly-Tyson               | Unidirectional; discontinuous       | 1965        |
| Bowyer and Bader          | Unidirectional or randomly; discontinuous | 1972        |

These equations have recently been cited to describe mechanical properties of natural fiber-reinforced thermoplastic or thermoset polymer composites. Joseph et al. (2002) studied elastic moduli and tensile strengths of treated sisal fiber-reinforced polypropylene composites with fiber loadings from 0 to 40 wt%. They concluded that composite elastic moduli and tensile strengths increased with increasing fiber loadings. Experimentally obtained tensile strengths were close to values predicted by a few theoretical functions. Baiardo et al. (2004) fabricated flax fiber-reinforced polyester composites with fiber volume percentages (v%) from 1 to 37.5. Experimental moduli
were predicted accurately by the modified rule of mixture (ROM) equation (Cox 1952) for fiber loading from 12.5 to 37.5 v%. However, the strength decreased when the fiber loading was higher than 12.5 v% due to lack of fiber/matrix adhesion.

Shibata et al. (2006) used the ROM equation (Cox 1952) to predict flexural properties of short kenaf fiber-reinforced starch-based polymer composites with fiber loadings from 0 to around 67.5 v%. Flexural moduli increased with increasing fiber loading up to 60 v%. The maximum flexural strength was reached when the fiber loading was close to 40 v%. Flexural moduli and strengths increased with increasing fiber lengths. Flexural moduli closely matched the values predicted by theory. Xue et al. (2007) studied tensile and flexural properties of aspen fiber-polypropylene-MAPP composites with fiber loadings from 0 to 60 wt%. These composites exhibited increased tensile moduli with increasing fiber loadings from 0 to 60 wt%. Tensile strengths increased with increasing fiber loadings increasing from 0 to 50 wt%, and then decreased with higher loadings. Elastic moduli predicted by the Halpin-Tsai model (1969) correlated well with experimental data at fiber loadings ranging from 0 to 30 wt%, and were slightly lower than the experimental results when the fiber loading is higher than 30 wt%. Haneefa et al. (2008) studied the mechanical properties of short banana/glass hybrid fiber-reinforced polystyrene composites with fiber loadings from 0 to 30 wt%. Mechanical properties increased with increasing fiber loadings. Experimental tensile properties showed good agreement with values calculated using the series method, the Hirsch model (Hirsch 1962), the Halpin-Tsai equation (Halpin 1969), the modified Halpin-Tsai equation (Nielsen 1970), and the Bowyer-Bader model (Bowyer and Bader 1972).
Foregoing studies suggested that moduli of natural fiber-reinforced composites, in general, increased with increasing fiber loadings, but the effect of fiber loadings on composite strengths varied, especially at high levels of fiber loadings when fibers could not be sufficiently wetted. Increasing fiber lengths improved both the moduli and strengths. The published literature is limited for mechanical properties of kenaf fiber-reinforced unsaturated polyester (UPE) composites with fiber weight fraction up to 65%.

1.4 Objectives

The main objectives of this research are to evaluate KBFB tensile properties, to develop a cost-effective and environmentally-friendly composite fabrication process, and to explore some alternatives for improving fiber-matrix bonding and decreasing moisture uptake in KBFB composites.

The detailed objectives are to: 1) evaluate the tensile behavior of KBFBs obtained from different retting processes; 2) investigate the effects of loading rates, locations, and combinations of temperature and duration on tensile properties of KBFBs, 3) develop a pre-forming method of compounding KBFBs into an unsaturated polyester matrix-based sheet molding compound; 4) evaluate effects of KBFB loadings and aspect ratios on composite tensile properties; 5) study composite strength properties and evaluate cost-effectiveness of KBFB-reinforced unsaturated polyester composites; and 6) improve composite water resistance properties and improve composite mechanical properties by optimizing fabrication parameters, and improve fiber-matrix adhesion by physical methods.
CHAPTER II
STATISTICAL EVALUATIONS OF TENSILE PROPERTIES
OF KENAF BAST FIBER BUNDLES

2.1 Materials and methods

2.1.1 Materials and experimental design

Three types of KBFB samples were studied: KBFB retted by bacterial, mechanical, and enzymatic methods. Bacterially-retted KBFBs in this study were originally from Bangladesh. They were commercially available in the Asian market. Mechanically-retted Kenaf bast fibers were supplied by Kengro Corporation. Enzymatically-retted KBFBs were obtained by retting kenaf ribbons using an enzyme. The enzyme retting was performed at University of Georgia.

Bacterially-retted KBFBs were used for evaluating the effects of loading rates on KBFB tensile properties. Samples were randomly cut from 15 cm inside from both ends of plant-size fiber bundles. Three loading rates were selected: 2.5 μm/s, 25 μm/s, and 250 μm/s. At least 30 replicates were prepared at each loading rate.

KBFB samples for evaluating the effects of locations on tensile properties were also cut from bacterially-retted KBFBs. Samples were extracted from 3 locations along the full plant-size fiber bundles: top, middle, and bottom. At least 30 replicates were prepared from each location.

The effect of retting methods on tensile properties of KBFB was also evaluated. KBFB samples from all three retting methods were prepared. These KBFB samples were
tested at low moisture contents close to 0%. The aim was to obtain elastic constants of mechanically-retted KBFBs at low moisture contents, which mimic the KBFBs used in composite fabrication.

2.1.2 Sample preparations

Long KBFBs were cut into 30-mm long KBFBs samples. All short KBFB samples were free of obvious defects observed from an optical microscope. The KBFB cross-section areas were measured at 3 longitudinal points under an optical microscope (Nikon Eclipse E 600). At each point, the maximum diameter was measured first and another measurement was taken at about 90° from the first measurement, assuming the cross section was in an elliptical shape. These 2 measurements were considered to be major and minor diameters of the ellipse. The area is then calculated using $A = \pi ab/4$, where $a$ and $b$ are the diameters along the major and minor axes of the ellipse.

KBFB samples were made into specimens by bonding them to paper tabs, which acted as frames to hold fibers, using a phenol-resorcinol-formaldehyde (PRF) adhesive. The nominal gauge length, length between two adhesive droplets, was 20 mm. Figures 2.1a and 2.1b show KBFB samples as-cut, a schematic of one KBFB specimen, and a specimen mounted on a paper tab as a frame for the grip and test assembly.
These KBFB specimens were conditioned in an environment with an ambient temperature of 23 ºC and a relative humidity of 30 to 40% for over 48 h prior to tests except for the specimens used for evaluating the effect of retting methods. The measured moisture content of KBFBs was around 8.5% after this conditioning.

Specimens for investigating tensile properties of KBFBs, obtained via 3 retting methods, were dried in an oven (Blue M Power-O-Matic-60) at 103 ºC for over 3 h. After drying, they were taken from the oven and sealed immediately in plastic bags separately to avoid moisture absorption prior to tensile testing.

2.1.3 Tensile testing

Tensile strengths and Young's moduli of KBFBs were evaluated according to ASTM Standard C1557-03 (ASTM 2003). Tests were performed on a stepper motor-driven micromechanical testing system (Bio Syntech Mach-1 V500cs). The instrument has a maximum loading capacity of 9.8 N at a precision of 0.98 mN. Precision of displacement control is about 0.5 µm. Specimens were gripped on 2 adhesive droplets. After being mounted on the grips, the paper tab was disconnected using a pair of scissors.
A 0.02 N preload was applied to remove slack of the fiber. The tests were conducted under displacement control. The loads and displacements were recorded at a frequency of 20 Hz.

KBFB specimens for evaluating loading rate effects were tested at 3 crosshead speeds, 2.5, 25, and 250 µm/s, which are equivalent to strain rates of $1.25 \times 10^{-4}$, $1.25 \times 10^{-3}$, and $1.25 \times 10^{-2}$/s when the nominal gauge length is 20 mm. KBFB specimens for evaluating effects of location and retting methods were tested with a crosshead speed of 25 µm/s.

Stress was calculated using the load and the averaged cross-section area of each specimen. Strain was calculated using displacement and the measured gauge length. The slope of the best linear-fit straight line through a subjectively chosen initial linear portion of stress-strain curves was considered to be the Young’s modulus. The maximum stresses during test were considered to be tensile strengths.

The micromechanisms of tensile failure were evaluated by examining the fracture surfaces of samples using a field emission gun scanning electron microscope (FEG-SEM) with fracture surfaces sputter coated with gold.

2.2 Results and discussions

2.2.1 Loading rates

Three failure mechanisms were clearly distinguishable in the uniaxial tensile tests: (1) a single fracture within the gauge section; (2) failure at or very close to the PRF resin droplets; and (3) catastrophic failure of the sample. Table 2.1 summarizes the values of Young’s moduli, tensile strengths, and elongation at three strain rates. In general, specimens failing close to or at the resin droplets had lower strengths. This type of
failure was caused by some unexpected residual stresses during resin curing, which
induced the earlier failure at resin droplets during testing. Most of the time, there was no
clear indication that the failure occurred right at the grip. Therefore, these test results are
still reported in Table 2.1. Specimens that failed in a catastrophic mode had, on average,
higher Young’s moduli and much higher tensile strengths and elongations, indicating that
failure occurred almost simultaneously at multiple locations along the KBFB. Failure by
either mechanism (1) or (3) indicates uniform loading along the entire length of the
specimen. Therefore, a separate statistical analysis of tensile behavior was performed on
these specimens alone (Table 2.1, asterisk-marked). The wide range of properties is
attributed to the large variation of natural fiber constituents and distribution of the
constituents, including inconsistencies in the diameter and length of individual single
kenaf fibers.

Table 2.1  Tensile properties of KBFB at 3 levels of strain rates (1.25×10−4/s,
1.25×10−3/s, and 1.25×10−2/s).

<table>
<thead>
<tr>
<th>Loading Rate μm/s</th>
<th>Young’s Modulus Mean GPa</th>
<th>COV %</th>
<th>Tensile Strength Mean MPa</th>
<th>COV %</th>
<th>Elongation Mean %</th>
<th>COV</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.5</td>
<td>13.5</td>
<td>13</td>
<td>146.4</td>
<td>31</td>
<td>1.12</td>
<td>27</td>
</tr>
<tr>
<td>2.5*</td>
<td>13.4</td>
<td>12</td>
<td>153.8</td>
<td>27</td>
<td>1.18</td>
<td>20</td>
</tr>
<tr>
<td>25</td>
<td>12.7</td>
<td>22</td>
<td>161.2</td>
<td>45</td>
<td>1.23</td>
<td>33</td>
</tr>
<tr>
<td>25*</td>
<td>13.5</td>
<td>22</td>
<td>200.2</td>
<td>31</td>
<td>1.46</td>
<td>21</td>
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<tr>
<td>250</td>
<td>15.3</td>
<td>24</td>
<td>188.4</td>
<td>41</td>
<td>1.19</td>
<td>32</td>
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<tr>
<td>250*</td>
<td>17.2</td>
<td>13</td>
<td>223.0</td>
<td>29</td>
<td>1.31</td>
<td>28</td>
</tr>
</tbody>
</table>

Note: * Indicates that these specimens failed in the middle of the gauge or failed in a
catastrophic mode.

Young’s moduli of the KBFBs listed in Table 2.1 marked with asterisks remained
almost the same until the loading rate reached 10−2/s. However, the tensile strengths of
the KBFBs increased gradually as the loading rates increased from 10−4/s to 10−2/s. We
may define the loading rate lower than $10^{-2}$/s as quasi-static loading. The yield strength must be reported along with the loading rate unless a conventional quasi-static loading rate is defined. This phenomenon demonstrates that the kenaf bast fiber bundle, a composite of $\alpha$-cellulose, holocelluloses, and lignin materials by itself, might have some general viscoelastic behavior with a damage mechanism highly dependent upon the loading rate.

2.2.2 Locations

Table 2.2 summarizes the average values of both Young’s moduli and tensile strengths of the KBFBs obtained from 3 locations along the plant-size fiber bundles. KBFBs from the middle have the highest Young’s moduli and tensile strengths compared to those from other 2 locations.

Table 2.2 also lists estimated distribution parameters of normal and 2-parameter Weibull distribution functions based on the maximum likelihood method and the p-values of goodness-of-fit tests for these 2 distributions. The 2-parameter Weibull distribution was only rejected at 5% significance level for Young’s modulus of top KBFBs. When the 2-parameter Weibull distribution was rejected, the normal distribution was also rejected for the same data set. Figure 2.2 presents histograms of tensile strengths of KBFBs from 3 locations. All of them showed a slight right-skewness. The probability density functions of these 2 distributions were also plotted for tensile strengths in Figure 2.2. The 2-parameter Weibull distribution was better at describing the most frequently appearing experimental data and non-negative values for material strengths. The 2-parameter Weibull distribution appears to be slightly preferable to the normal distribution.
from foregoing calculated statistics and the distribution plots. However, the normal
distribution cannot be rejected in most cases.

Table 2.2 Young’s moduli, tensile strengths, and elongations of KBFBs, with values of the goodness-of-fit of normal and 2-parameter Weibull distribution.

<table>
<thead>
<tr>
<th></th>
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<th></th>
<th>Middle</th>
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<th>Bottom</th>
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<td>47</td>
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<tr>
<td>Cramer-von Mises</td>
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<td>0.1</td>
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<td>&gt;0.250</td>
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<td>Anderson-Darling</td>
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<td>200.5</td>
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<td>&gt;0.250</td>
<td>&gt;0.250</td>
<td>&gt;0.250</td>
<td>&gt;0.250</td>
</tr>
</tbody>
</table>

Note: Values with the same capital letter are not statistically significant at a 5% significance level.
Figure 2.2  Histogram of tensile strengths for KBFBs from 3 locations along plant stem KBFBs: a) top, b) middle, and c) bottom of the kenaf plant.
2.2.3 Retting methods

Table 2.3 summarizes tensile properties of KBFBs retted via 3 methods. It should be noticed that dried KBFBs obtained by bacterial retting have an average Young’s modulus of 33.0 GPa and an average tensile strength of 420.3 MPa, significantly higher than Young’s moduli and tensile strengths for evaluating the location effect. The moisture content of KBFBs significantly affects the tensile properties of KBFBs.

KBFBs retted bacterially have the highest Young’s modulus, tensile strength, and best elongation of the 3 retting methods. Enzyme-retted KBFBs have a Young’s modulus in between those of the other methods but the lowest strength and elongation. Mechanically retted KBFBs have the lowest Young’s modulus and tensile strength and elongation in the middle.

<table>
<thead>
<tr>
<th>Retting method</th>
<th>Young’s modulus GPa</th>
<th>COV (%)</th>
<th>Tensile strength MPa</th>
<th>COV (%)</th>
<th>Elongation %</th>
<th>COV (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bacterial</td>
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<td>17</td>
<td>420.3</td>
<td>36</td>
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<td>Enzyme</td>
<td>25.4</td>
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<td>196.9</td>
<td>44</td>
<td>1.01</td>
<td>22</td>
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</tbody>
</table>

Note: Values with the same capital letter are not statistically significant at 5% significance level.

2.2.4 Failure Mechanisms

The SEM micrographs display two failure modes in the KBFBs, as shown in Figure 2.3: fiber pullout from the bundle (Figures 2.3 a and c) and fiber breaks (Figures 2.3 b and d). Accompanying fiber pullout from the bundle, there exists a large region of fiber separation at the fiber/matrix interfaces. On the possible broken fiber section
(figure 2.3 c), there is only a slight hint of fibrous debonding. Figure 2.3 a also shows that debonding and fiber failure coexist.

Figure 2.3  SEM micrographs of KBFB failure surfaces.

2.3 Summary and conclusions

Tensile behavior of KBFBs was investigated. The effects of loading rates, fiber location, and retting methods on KBFB tensile properties were evaluated. A statistical summary of elastic properties was presented based on a relatively large sample set due to a large scatter in KBFB tensile properties. The KBFBs are fairly brittle and demonstrated a strain rate-dependence of Young’s moduli and tensile strengths in the strain rate range of $10^{-4}$ to $10^{-2}$/s. The Young’s moduli and tensile strengths were dependent on the
location in the plant stem from which the fibers were extracted. Retting methods affect
the KBFB tensile properties. Based on the SEM images, it can be concluded that two
failure mechanisms exist: fiber pullout and fiber breakage.
CHAPTER III

TEMPERATURE-DURATION EFFECTS ON KENAF BAST FIBER BUNDLE TENSILE PROPERTIES

3.1 Materials and methods

3.1.1 Materials and experimental design

Fiber bundles in this study were originally obtained from Bangladesh. They were obtained directly from kenaf stems via a bacterial-retting technique. A 5 by 3 factorial experiment was designed to evaluate the effect of the temperature and the duration held at each temperature on KBFB tensile properties. Five levels of temperature (110, 130, 150, 170, and 190 °C) at 3 duration levels (3, 6, and 9 h) were investigated (Table 3.1). Each of the 15 temperature and duration treatment combinations was performed using 20 replicates.

3.1.2 Specimen preparation and property testing

KBFB samples, 30-mm long, were cut from the middle portion of plant-size long fiber bundles. The KBFB diameters were measured at three locations under a microscope, and cross section areas were calculated using measured average diameters. Then, specimens were treated in an oven (Blue M Power-O-Matic-60) in accordance with the 15 temperature-duration combinations listed in Table 3.1.
Table 3.1  Failure locations and number of samples for kenaf bast fiber bundle specimens tested at each temperature-duration combination and specimen moisture contents (MC).

<table>
<thead>
<tr>
<th>Temperature ºC</th>
<th>Duration h</th>
<th>Failure mode</th>
<th>MC %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Middle</td>
<td>Grip</td>
<td></td>
</tr>
<tr>
<td>110</td>
<td>3</td>
<td>5</td>
<td>16</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>2</td>
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<td></td>
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<td>11</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>5</td>
<td>8</td>
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<tr>
<td></td>
<td>9</td>
<td>2</td>
<td>10</td>
</tr>
<tr>
<td>170</td>
<td>3</td>
<td>4</td>
<td>7</td>
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<td></td>
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<td></td>
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<td>190</td>
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<td>3</td>
<td>6</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>2</td>
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</tr>
<tr>
<td></td>
<td>9</td>
<td>2</td>
<td>2</td>
</tr>
</tbody>
</table>

After being heated in the oven, the KBFBs were moved and placed in a desiccator. Individual KBFB samples were mounted upon paper tabs with a PRF adhesive. The nominal gauge length between the 2 adhesive droplets was 20 mm. Then, the specimens were stored in ambient conditions with a temperature of 25 ºC and a relative humidity of 50-60% for over 36 hours to allow the glue to cure.

Samples were fragile after being treated at high temperatures, especially those treated at 170 ºC or above. Some samples broke during specimen preparation or testing. The actual sample size of specimens tested for each temperature and duration combination is shown in Table 3.1.

The color of KBFBs treated at 170 ºC changed from light blond to golden brown. The color of KBFB samples treated at 190 ºC were brown, and the color turns darker as
duration at 190 °C increased from 3 to 9 h. The color change suggested the carbonization began for KBFBs treated at 170 °C and above. No obvious color changes were observed for KBFB specimens heated at 150 °C or below.

Tensile testing was performed on the micro-mechanical testing machine shown in Figure 3.1 in accordance with the ASTM Standard C1557-03 (ASTM 2003). The test speed was 20 μm/s, equivalent to a strain rate of 0.001/s. Loads and displacements were recorded. Tensile strengths were calculated using ultimate loads at the KBFB failure point and averaged cross-sectional areas since the stress-strain relationship was linear up to failure point as shown in a typical stress-strain curve in Figure 3.2. Young’s modulus was determined from the slope of best linear-fit line of the stress-strain curve. The moisture contents (MC) for each of 15 tested specimen groups was measured. Scanning electron micrographs of specimen fracture surfaces were taken using a Carl Zeiss EVO-40XVP scanning electron microscope with the fracture surfaces sputter-coated with gold.

Figure 3.1   Test setup for evaluating kenaf fiber bundle tensile properties with and a mounted specimen.
3.2 Results and discussion

3.2.1 Failure locations and modes

Under tensile loads, KBFBs had three main failure locations as shown in Figure 3.3: fiber broken at the grips, fiber broken in the middle, and catastrophic failure. Table 3.1 summarizes the numbers of KBFBs which failed at each of the 3 failure locations. Tensile strengths and elongations were calculated from test data where the KBFBs failed in the middle of the gauge or broke in a catastrophic failure mode. Tensile moduli were calculated for all tested specimens.
SEM micrographs show the 3 typical failure modes observed in the KBFB tensile testing: splintering tension (Figure 3.4a), combined splintering and brash tension (Figure 3.4b), and brash tension (Figure 3.4c). The splintering mode was mostly observed in specimens heated at 110 °C. The combined failure mode was frequently found in specimens heated at 150 and 170 °C, and brash mode occurred in specimens heated at 190 °C. These three failure modes seem to be related to fiber color changes (from light blond to golden brown, then to brown) observed after the heat treating process, which indicated changes occurring in the cellulose, hemicellulose, and lignin of KBFBs.
Figure 3.4  SEM micrographs of tensile fracture surfaces of KBFB specimens: a) splintering tension; b) combined splintering and brash tension; and c) brash tension.
3.2.2 Data analysis

Table 3.2 summarizes the mean values of Young’s moduli, tensile strengths, and elongations of specimens tested at each of the 15 temperature and duration combinations. The numbers of specimens used to calculate mechanical properties were also given for each property. The coefficients of variation (COV) of Young’s moduli ranged from 14 to 25% for the temperatures of 170 °C and below, but large COVs (42 to 59%) occurred at 190 °C. The COV range for tensile strengths spanned from 16 to 50%, while elongations had a COV range from 11 to 38%.

Table 3.2 Mean mechanical property values for tested kenaf bast fiber bundles at each combination of temperature and duration.

<table>
<thead>
<tr>
<th>Temperature °C</th>
<th>Duration h</th>
<th>Tensile Modulus Mean GPa</th>
<th>COV %</th>
<th>N.</th>
<th>Tensile Strength Mean MPa</th>
<th>COV %</th>
<th>N.</th>
<th>Elongation Mean %</th>
<th>COV %</th>
<th>N.</th>
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<td>41.4</td>
<td>42</td>
<td>2</td>
<td>0.47</td>
<td>1</td>
<td></td>
</tr>
</tbody>
</table>

The moisture content (MC) of the specimens was also summarized in Table 3.2. The specimens of each temperature-duration combination had different average equilibrium MC. In general, the equilibrium MC decreased as temperatures increased.
Mechanical properties of woody materials are affected by moisture content below the fiber saturation point. Differences in MC for tested specimens would cause variations in mechanical properties. In order to analyze the effect of temperature and heating duration on tensile properties, the effect of the difference in the MC on tensile properties needs to be eliminated. Kretschmann and Green (1996) observed that the Young’s modulus of southern pine had a very slight decrease of 0.64% when the MC increased from 7 to 12%. The tensile strength increased by 7.35% when the MC increased from 7 to 12%. Since there is no data is available founding regard to the effect of MC on mechanical properties for KBFB, all individual experimental data points were adjusted to value at 12% MC based on the data of southern pine. Based on this assumption, each 1% MC change results in 1.47% change in tensile strengths and 0.13% change in elastic moduli. Elongation was adjusted by the ratio of tensile strength to Young’s modulus. The analysis of variance (ANOVA) was performed on the adjusted data instead of the original recorded data.

A 2-factor unbalanced ANOVA general linear model procedure was performed for adjusted individual data to analyze both the main effects and the interaction factors on the means of Young’s modulus, tensile strength, and elongation, respectively. The results indicated that the 2-factor interactions of temperature and duration in analyses of Young’s modulus and tensile strength as the dependent variables were statistically significant (p-value = 0.0808) and marginally significant (p-value = 0.1373) at the 10% significance level. Temperature was the only significant effect in the elongation analysis. Therefore, the effects of two significant interactions were examined for Young’s modulus and tensile strengths, respectively; while the main effect of temperature on elongation
was evaluated. The least squares means (LSMEANS) multiple comparison method was performed to determine the mean differences of the treatment combinations.

3.2.3 Temperature effects

3.2.3.1 Young’s moduli

In general, as the temperature increased from 110 to 170 °C, no significant change was observed in KBFB’s Young’s moduli. This non-significant change in KBFB’s Young’s moduli is because celluloses are thermally stable in this temperature range (Schaffer 1973, LeVan and Winandy 1990, Munder and Hempel 2006), and stiffness of KBFBs is mainly governed by cellulose, which acts as a reinforcement, if the KBFBs is assumed to be a composite. Therefore, the stiffness of KBFBs is less sensitive to the temperature change compared to hemicellulose and lignin within the temperature range from 110 to 170 °C. Significant drops in Young’s moduli occurred when the temperature increased from 170 to 190 °C. The average drops in the Young’s modulus were 39%, 47%, and 55% for the duration of 3, 6, and 9 h, respectively. This sharp decrease in Young’s moduli can be explained by the significant mass loss in KBFBs due to the severe degradation of cellulose, hemicellulose and lignin, especially the cellulose at high temperatures (Schaffer 1973, LeVan and Winandy 1990, Munder and Hempel 2006) for long duration.

3.2.3.2 Tensile strengths

Figure 3.5 plots the mean separation results for temperatures on tensile strengths for each duration level. In general, tensile strengths declined as the temperature increased for 6 and 9 h duration, and different declining rates were observed as the temperature increased. The tensile strength declined at an averaged rate of 1.19 MPa/°C
as the temperature increased from 110 to 170 °C for the 6 h duration. As the temperature increased from 170 to 190 °C, the average declining rate increased to 3.07 MPa/°C. There was a 51% drop in tensile strengths when the temperature increased from 150 to 190 °C. An average drop of 44% in tensile strengths occurred when the temperature increased from 170 to 190 °C, but this drop was not statistically significant due to the large variation of the data.
Figure 3.5  Mean comparisons for temperature effects on tensile strength for each duration of a) 3 h, b) 6 h, and c) 9 h (Values with the same capital letter are not statistically different at 10% significance level).
In the case of 9 h duration, tensile strengths declined in an average rate of 0.72 MPa/°C for the temperature range from 110 to 150 °C, and the strength decline was not statistically significant in this temperature range. The declining rate increased to 2.72 MPa/°C when the temperature was higher than 150 °C. A 38% decline in tensile strengths occurred as the temperature increased from 150 °C to 170 °C. As the temperature increased to 190 °C, an average drop of 54% in tensile strengths occurred, but this decline was not statistically significant due to fewer replicates at 190 °C and the large data scatter.

No significant tensile strength decline was observed for 3 h duration as the temperature increased from 110 to 170 °C, and a significant 66% drop in tensile strengths occurred when the temperature increased to 190 °C.

The gradual tensile strength decline observed for 6 and 9 h duration could be explained by the gradual degradation of lignin and hemicellulose starting at lower temperatures (Schaffer 1973, LeVan and Winandy 1990), which caused the mass loss of these components. The weight loss resulted in the reduction in the stiffness of lignin and hemicellulose, which act as the matrix if the KBFB is considered to be a composite. Subsequently, this gradual stiffness decrease causes the decrease in KBFB tensile strengths (Stellbrink 1996). The significant drop in tensile strengths can be explained by the severe degradation of cellulose, hemicellulose and lignin.

3.2.3.3 Elongation

The temperature had a significant effect on KBFB elongation (Figure 3.6). The elongation decreased as the temperature increased. A significant decline in elongation started when the temperature reached 150 °C. The strain continued to drop significantly
as the temperature increased from 150 °C to 170 °C. No further significant decline was observed as temperature increased to 190 °C.

Figure 3.6  Effect of temperature on the ultimate strain of the kenaf fiber bundles (Values with the same capital letter are not statistically different at 10 percent significance level).

3.2.4 Duration effects

3.2.4.1 Young’s moduli

There was no significant changes in Young’s moduli for temperatures ranging from 130 to 190 °C when the duration changed from 3 to 9 h (Table 3.3). A significant duration effect on the Young’s modulus was observed at the temperature of 110 °C, where the Young’s modulus had a significant 28% increase when the duration increased from 3 to 6 h, and then had a significant 16% drop as duration kept increasing to 9 h. The increase in the Young’s modulus at 110 °C for 6 h duration might be due to the hemicellulose content decrease, and the alpha-cellulose increase (Schaffer 1973). At 190
˚C, Young’s moduli declined as the duration increased, while the decline was not statistically significant. This decline trend in the stiffness would be caused by the possible cellulose degradation because of long heat treatment duration.

Table 3.3  Mean comparisons for duration on Young’s modulus and tensile strength for each level of temperature.

<table>
<thead>
<tr>
<th>Temperature °C</th>
<th>Duration (Hour)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>3</td>
</tr>
<tr>
<td>Tensile modulus (GPa)</td>
<td></td>
</tr>
<tr>
<td>110</td>
<td>12.0</td>
</tr>
<tr>
<td>130</td>
<td>13.8</td>
</tr>
<tr>
<td>150</td>
<td>14.4</td>
</tr>
<tr>
<td>170</td>
<td>15.0</td>
</tr>
<tr>
<td>190</td>
<td>9.2</td>
</tr>
<tr>
<td>Tensile strength (MPa)</td>
<td></td>
</tr>
<tr>
<td>110</td>
<td>139.9</td>
</tr>
<tr>
<td>130</td>
<td>165.7</td>
</tr>
<tr>
<td>150</td>
<td>142.6</td>
</tr>
<tr>
<td>170</td>
<td>152.7</td>
</tr>
<tr>
<td>190</td>
<td>52.5</td>
</tr>
</tbody>
</table>

Note: Values with the same capital letter are not statistically different at the 10% significance level.

3.2.4.2 Tensile strengths

The duration had no significant effect on tensile strengths (Table 3.3) for the temperature range from 130 to 190 °C. A significant increase in tensile strengths was observed when the duration increased from 3 to 6 h at 110 °C. A non-significant, high percentage drop in tensile strengths was observed at temperatures of 170 and 190 °C when the duration increased from 6 to 9 h.

3.2.4.3 Elongations

In general, the duration had no significant effect on KBFB elongations.
3.3 Summary and conclusions

Effects of high-temperature and treatment duration treatment on KBFB tensile properties were investigated. Experimental results indicated that the temperature and the treatment duration had a significant interaction effect on the Young’s modulus, while had a marginal significant interaction effect on tensile strengths at a 10% significance level. The tensile elongation was significantly affected by the temperature.

There was no significant change in KBFB Young’s moduli as the temperature increased from 110 to 170 °C. A significant decrease in Young’s moduli was observed when the temperature increased from 170 to 190 °C, and the declines in Young’s moduli were 39%, 47%, and 55% for 3, 6, and 9 h duration, respectively. The treatment duration seemed to have no significant effect on KBFB Young’s moduli at the temperature of 130 °C and above. The different KBFB stiffness behavior can be explained by the KBFB’s cellulose degradation process.

KBFB tensile strengths generally declined as the temperature increased, and the different declining behavior was observed among 3 duration levels. Significant KBFB tensile strength drops started at 170, 170, and 150 °C for 3, 6, and 9 h duration, respectively. The duration seemed to have no significant effect on KBFB tensile strengths at the temperature of 130 °C and above. The KBFB tensile strength decline due to the temperature change can be interpreted by the degradation process of KBFBs’ cellulose, hemicellulose, and lignin.

In general, the KBFB elongation decreased as the temperature increased. A significant decline in KBFB elongations started at 150 °C.
CHAPTER IV
PROCESSING TECHNIQUES FOR HIGH KENAF-FIBER LOADING
UNSATURATED POLYESTER COMPOSITES

4.1 Materials and methods

4.1.1 Materials

The raw materials used in this study consisted of KBFBs, UPE, styrene, catalysts, binders, and a few additives. Mechanically-retted Kenaf bast fibers were supplied by Kengro Corporation. 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 wt% solids content, provided by Tailored Chemical, was used as a binder for making mat pre-forms. Maleic anhydride grafted polypropylene (MAPP) (Epolene G-3015, MW = 47000, maleic anhydride <1.0 wt%), provided by Eastman Chemical Company, was used as a thermoplastic binder, and also as a coupling agent to compatibilize fibers with the matrix resin. The melting point of MAPP is between 155 °C and 158 °C (Keener et al. 2004); this was confirmed by Pimenta et al. (2008) using DSC with a ramping rate of 10 °C/min, whereby the melting point peaks were at 153 °C and 162 °C.
4.1.2 Composite fabrication

The composite fabrication process consists of four main steps.

4.1.2.1 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 KBFBs was 3.3 mm. The measured moisture content of KBFBs was approximately 10 %.

4.1.2.2 Mat pre-forming

Two mat pre-forming methods (involving a PVAc adhesive and a MAPP binder respectively) were developed in this study.

The first KBFB pre-forming process is illustrated in Figure 4.1. First, 100 g (100 parts) 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 (Figure 4.1a). This sheet had been pre-coated with a non-stick mold release agent. Then, 12.5 g of PVAc adhesive (46 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 (Figures 4.1b and c). Next, after covering the sprayed surface with another steel sheet (Figure 4.1d), the whole stack was turned upside down and the upper steel sheet was removed (Figure 4.1e). Another layer of diluted 50 g PVAc was then sprayed on the upper surface (Figure 4.1f). Now, the KBFBs were pressed into a $381 \times 432 \times 3$ mm$^3$ performed KBFB mat under a pressure of 0.15 MPa at a temperature of 175 °C for 4 min. Finally, eight $102 \times 178 \times 3$ mm$^3$ mat sections (Figure 4.2) 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 4.1 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 × 178 mm² mat was 10 g.

Figure 4.1 Fabrication of a polyvinyl acetate (PVAc) pre-formed 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; f) another layer of PVAc was sprayed on the top surface.
Figure 4.1 (Continued).
Figure 4.1  (Continued).

Figure 4.2  Pre-formed 102 × 178 × 3 mm3 kenaf bast fiber bundle mats
Table 4.1  Average weight of each ingredient in a full-size and single kenaf bast fiber bundle mat

<table>
<thead>
<tr>
<th>Weight (g)</th>
<th>Total (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PVAc Fiber (10% moisture content)</td>
<td>PVAc (48% solid content)</td>
</tr>
<tr>
<td>Wet mat</td>
<td>Dry mat (381×432 mm²)</td>
</tr>
<tr>
<td>Solid</td>
<td>Water</td>
</tr>
<tr>
<td>91</td>
<td>9</td>
</tr>
<tr>
<td>91</td>
<td>0</td>
</tr>
<tr>
<td>10</td>
<td>0</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>MAPP</th>
<th>Dried fiber</th>
<th>MAPP</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mat (381×432 mm²)</td>
<td>91</td>
<td>15.18</td>
</tr>
<tr>
<td>Single mat (102×178 mm²)</td>
<td>10</td>
<td>1.67</td>
</tr>
</tbody>
</table>

MAPP was employed as a thermoplastic binder in a similar mat pre-forming method. MAPP was chosen as a binder because it can also serve as a coupling agent to the fibers, which could improve mechanical and water resistance properties. First, MAPP was ground into 20 mesh size powder using a mill. Then, 15.18 g of MAPP powder was mixed with 91.07 g KBFBs in a sealed shaking box. The weight ratio of KBFBs to MAPP was 6 to 1. Second, the 106.25 g KBFB/MAPP mixture was evenly dispersed into the 381 × 432 mm² wood frame placed on a metal sheet covered with a polytetrafluoroethylene (PTFE) sheet. Third, another sheet of PTFE was placed to cover the loose mat when the wood frame was removed. After the whole stack was loaded into the press, the metal sheet was carefully removed from underneath. Fourth, the loose mat was pressed at 150 °C with a pressure of 0.5 MPa for 4 min, and the whole stack was removed from the press and cooled in ambient temperature for a few minutes to let MAPP solidify. A pre-formed mat was obtained after the PTFE sheets were removed. Eight 102 × 178 × 3 mm³ mats were cut from the full-size mat. These mats were placed
in a sealed plastic bag immediately to avoid moisture absorption. The average weight of a single mat was 11.67 g (10 parts of KBFB and 1.67 part of MAPP).

4.1.2.3 UPE resin application

The UPE resin and its ingredients were mixed using a stirrer employing the formulation: 65 parts of UPE, 10 parts of styrene, 0.05 part of inhibitor, and 1.5 parts of peroxide catalyst by weight. After being mixed thoroughly, the liquid resin mixture was transferred to a spray gun.

Mats pre-formed using PVAc were dried in an oven at 103 °C for over 3 h before resin application. The resin was sprayed on the mats immediately after they were removed from the oven. Figure 4.3a shows a schematic of the stacking pattern of 5 stacked mats and the corresponding amount of resin by weight.

![Diagram of stacked mats and resin application](image)

**Figure 4.3** A schematic of the stacking pattern of stacked pre-formed kenaf bast fiber bundle mats and the corresponding amounts of unsaturated polyester resin applied on each mat: a) pre-formed mat using PVAc; b) pre-formed mat with MAPP.
Four 102 × 178 mm² mats pre-formed with MAPP were stacked to assemble a laminated prepreg. The UPE resin was sprayed on the mats immediately after they were taken from the sealed plastic bag. The amounts of UPE resin sprayed on each mat are shown schematically in Figure 4.3b. Table 4.2 shows the average weights of each ingredient for these two pre-forming processes and the desired fiber loadings prior to compression molding.

<table>
<thead>
<tr>
<th></th>
<th>Weight (g)</th>
<th>Desired fiber loading (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Fiber</td>
<td>PVAc</td>
</tr>
<tr>
<td>KPU</td>
<td>50</td>
<td>6.3</td>
</tr>
<tr>
<td>KMU</td>
<td>40</td>
<td>6.7</td>
</tr>
</tbody>
</table>

**4.1.2.4 Compression molding**

Each laminated prepreg was placed between two mold platens pre-coated with silicone mold release agent. The platens were preheated to 175 °C. The pressure was raised to 5 MPa on these prepregs within 10 sec and then the press’s heating switch was turned off. The composite was maintained at this pressure for 1 h. 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 cured UPE resin which had squeezed out was sanded off along 4 lateral edges of the panel. Then, the weight and size of each composite panel were measured to calculate the actual fiber loading. Representative composite samples are shown in Figure 4.4. Four KBFB-PVAc-UPE (KPU) composite panels and four KBFB-MAPP-UPE (KMU) composite panels were fabricated in this study.
4.1.3 Tensile testing

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

4.1.4 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 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 sec. Scanning electron microscope (SEM) images were taken on Carl Zeiss SMT EVO 50 instrument (EHT: 5-15 kV).

4.2 Results and discussion

4.2.1 Fiber loading

Table 4.3 summarizes mean values of measured fiber loadings of both KPU and KMU composites. The fiber loadings of KPU composites ranged from 64.4 to 67.1 wt% with an average value of 65.3 wt%. The KMU composites had fiber loadings ranging from 61.0 to 63.1 wt% with an average value of 62.2 wt%. Thus, the composite fiber loading can be successfully increased up to 65 wt% using the fabrication technique explored in this study.

Table 4.3 Calculated fiber loadings in each composite group after compression

<table>
<thead>
<tr>
<th></th>
<th>Weight (g)</th>
<th>Average fiber loading</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Fiber</td>
<td>COV (%)</td>
</tr>
<tr>
<td>KPU</td>
<td>50</td>
<td>76.8</td>
</tr>
<tr>
<td>KMU</td>
<td>40</td>
<td>64.3</td>
</tr>
</tbody>
</table>

The SEM micrographs of the KPU composite cross sections (Figures 4.5a and b) show good resin penetration throughout the specimen thickness and uniform resin wetting on fiber bundle surfaces. KMU composites (Figures 4.5c and d) are similar to KPU composites in wetting or penetration. Close inspection of Figures 4.5a and c reveals the presence of a few small voids.
Figure 4.5 SEM images of composites at different magnification: a) cross-section smoothed by microtome (kenaf-PVAc-UPE), b) fracture surfaces of a tensile specimen (kenaf-PVAc-UPE); c) cross-section smoothed by microtome (kenaf-MAPP -UPE), d) fracture surfaces of tensile specimens (kenaf-MAPP –UPE).

PVAc and MAPP both bind KBFB together to form a loose pre-formed solid mat at low pressure. These mats are strong enough to assure 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 pre-forming process ensures uniform properties in the resultant composite and speeds the molding cycle. Thicker composites can be produced by simply increasing the number of pre-formed mats in the laminate. Composites with lower fiber loadings can be
made using lower molding pressure or employing high-accuracy molds with reduced mold gaps.

4.2.2 Physical and tensile properties

Table 4.4 summarizes the physical and mechanical properties of KPU and KMU composites fabricated in this study, mechanical property data from a SMC/BMC design manual (European Alliance 2001), and specification requirements for glass fiber/UPE composites from an automotive part manufacturer. The densities of KPU (1.22 g/cm³) and KMP (1.18 g/cm³) composites are significantly lower than those of glass fiber/UPE composites (1.8 - 2.15 g/cm³). This could result in large percentage weight savings if the same volume of each composite could be used in finished products. In general, the tensile properties of KPU composites were better than those of KMU composites. This could be ascribed to small MAPP particles which were found from the surface of the KMU composites because of the low mat pre-forming temperature (150 °C) which was below the reported melting point of MAPP (Pimenta et al., 2008; Keener et al., 2004). These MAPP particles caused stress concentration when a load is applied on the composite. Increasing the pre-forming temperature higher than the MAPP melting point would have avoided this difficulty.
Table 4.4  Mean values of physical and mechanical properties of tested composites and specifications of glass fiber reinforced unsaturated polyester composites

<table>
<thead>
<tr>
<th></th>
<th>KPU&lt;sup&gt;a&lt;/sup&gt;</th>
<th>KMU&lt;sup&gt;b&lt;/sup&gt;</th>
<th>SMCs&lt;sup&gt;c&lt;/sup&gt;</th>
<th>Specific requirement&lt;sup&gt;d&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density</td>
<td>1.22</td>
<td>1.18</td>
<td>1.8 - 2.15</td>
<td>1.85 ± 0.05</td>
</tr>
<tr>
<td>Elastic modulus</td>
<td>12.1</td>
<td>8.65</td>
<td>10 - 19.1</td>
<td>9</td>
</tr>
<tr>
<td>COV</td>
<td>21</td>
<td>13</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Specific modulus</td>
<td>9.92</td>
<td>7.33</td>
<td>(5.56-8.88)</td>
<td>(4.86)</td>
</tr>
<tr>
<td>Tensile strength</td>
<td>54.6</td>
<td>38.9</td>
<td>65 - 204</td>
<td>58</td>
</tr>
<tr>
<td>COV</td>
<td>8</td>
<td>6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Specific strength</td>
<td>44.8</td>
<td>33.0</td>
<td>(36.1-94.9)</td>
<td>(31.4)</td>
</tr>
<tr>
<td>Elongation</td>
<td>0.62</td>
<td>0.55</td>
<td></td>
<td></td>
</tr>
<tr>
<td>COV</td>
<td>22</td>
<td>29</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<sup>a</sup>Kenaf-PVAc-unsaturated polyester composites.
<sup>b</sup>Kenaf-MAPP-unsaturated polyester composites.
<sup>c</sup>Sheet molding compounds (Contains filler, unsaturated polyester, and 25-50 wt% glass fiber): Values in parentheses are calculated from the lower limit of the properties range (SMC automotive alliance 1991).
<sup>d</sup>Specification requirements for glass fiber/UPE composites (Fiber content: 25-30%) from an automotive part manufacturer: values in parentheses are calculated by properties and densities by requirement.

The elastic modulus of the KPU composites (12.1 GPa) was 21.0% higher than the lower limit of the SMC products (10 GPa). Moreover, the elastic modulus of KPU was 34.4% higher than that of the manufacturer’s specification requirements (9 GPa). Similarly, the specific modulus of KPU (9.92 GPa·cm³/g) was 78.7% higher than the lower limit for SMCs (5.55 GPa·cm³/g), and 104.1% higher than the specification requirement (4.86 GPa·cm³/g). The KPU composite’s tensile strength (54.6 MPa) was lower than the lower limit for the SMCs (65 MPa) and is just below the manufacturer’s specifications (58 MPa) (Table 6). However, the specific strength of the KPU composite (44.8 MPa·cm³/g) was still attractive compared to that of SMCs (36.1 MPa·cm³/g) and the manufacturer’s requirement (31.4 MPa·cm³/g).

The tensile elastic modulus of the KMU composite (8.65 GPa) was 13.5% lower than the lower limit of SMC composites, and 3.9% lower than the specification requirement. The KMU composite’s low elastic modulus versus that of the KPU
composite could be due to the KMU composite’s lower fiber loading (62.2 wt%) versus that of the KPU composite (65.3%). However, the specific modulus of the KMU composite (7.33 GPa·cm³/g) favorably compared with that of SMCs (5.55 GPa·cm³/g) and manufacturer specification (4.86 GPa·cm³/g). The tensile strength of KMU composite (38.9 MPa) was much lower than that of both the SMCs (65 - 204 MPa) and the manufacturer’s requirement (58 MPa). The specific strength of KMU composite (33.0 MPa·cm³/g) was also lower than that of the SMCs (36.1 MPa·cm³/g), but higher than that of the manufacturer’s requirement (31.4 MPa·cm³/g).

4.2.3 Failure mechanisms

SEM images of tensile specimen fracture surfaces of the KPU and KMU composites (Figure 4.6) show four typical failure modes of the KBFB/polyester composites: fiber splintering tension breakage, brash tension breakage, fiber/matrix debonding, and fiber pull-out. The strength of fiber-reinforced resin composites is a complex function of the material and geometric inhomogeneities, fiber-to-matrix adhesion, fiber/matrix interphase structure, fiber volume fraction (Kaw 2006), resin strength, fiber strength, variability of fiber strengths (U.S. DOD 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.
Figure 4.6  Tensile fracture surfaces of kenaf-PVAc-UPE (KPU) and Kenaf-MAPP-UPE (KMU) composites: a) fiber tension breakage in KPU composites; b) fiber tension breakage in KMU composites; c) fiber pullout breakage in KPU composites; d) voids after fiber pullout breakage of KMU composites.

The first two failure modes observed in this work (Figures 4.6a and b) are attributed to fiber fractures. The latter two mechanisms (Figures 4.6c and d) result from moderate interfacial matrix-to-fiber adhesion. Failure mechanisms in KMU composites were similar to those in KPU composites. It appears that the tensile strengths of these composites are dominated by both (a) the UPE matrix-to-fiber bundle interfacial bonding and (b) the fiber bundle tensile strengths. Hence, improving the interfacial adhesion in this class of composites is one of the ways to improve composite strength (Baiardo et al. 2004, John and Naidu 2004, Baley et al. 2006, Aziz and Ansell 2004, Misra et al. 2002).
Voids (Figures 4.6a and c) and gaps (Figure 4.6d) between the fibers and the matrix were observed. Clearly, the compatibility between fiber and matrix still needs improvement. These observations suggest that low tensile strengths could be attributed to voids and limited fiber-to-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 potentially have mechanical properties exceeding those of SMCs.

4.3 Summary and conclusions

A composite fabrication process for high kenaf bast fiber bundle 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 pre-forming using either a PVAc emulsion adhesive or a MAPP binder, (3) UPE resin application, and (4) laminate compression molding.

The process provided relatively good resin coverage on kenaf bast fiber bundle surfaces for average fiber loadings up to 65 wt%. Such high fiber loadings could substantially reduce raw material and manufacturing costs in comparison with traditional automotive composites while also increase 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. KPU composites displayed better tensile properties than KMU specimens. Both of these composite types had lower densities which yielded favorable specific moduli and strengths versus a glass fiber/UPE composite. 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.
CHAPTER V
EFFECTS OF FIBER LOADINGS AND ASPECT RATIOS ON TENSILE PROPERTIES OF KENAF BAST FIBER BUNDLE-REINFORCED UNSATURATED POLYESTER COMPOSITES

5.1 Materials and methods

5.1.1 Materials

In this study, mechanically-retted KBFBs, provided by Kengro Corporation, were used as reinforcing fibers. The average diameter of these KBFBs was measured to be 78.6 μm. The UPE (Aropol Q-6585), provided by Ashland Chemical Company, was used as the matrix polymer. The diluent, styrene, and the catalyst, t-butyl perbenzoate (TBP), were purchased from Fisher Scientific Inc. Methyl ethyl ketone peroxide (MEKP), purchased from Sigma-Aldrich Corporation, and cobalt naphthenate (CoNa), purchased from Fisher Scientific Inc., were used as a second catalyst system. A poly(vinyl acetate) (PVAc) water emulsion (solid content: 46%) provided by Tailored Chemical was used as the mat pre-forming binder.

5.1.2 Experimental

5.1.2.1 KBFB tensile properties

Short KBFB samples were randomly cut from long mechanically-retted KBFBs. Twenty replicates were used. Tensile specimens were prepared using a previously published method (Du et al. 2008). These specimens were dried at 103 °C for 3 h to
assure the moisture content of these KBFB specimens was negligible. After drying, each specimen was sealed immediately in individual plastic bags to avoid moisture uptake. KBFB tensile properties were obtained on a micromechanical testing machine according to ASTM Standard C 1557-03 (ASTM 2003).

5.1.2.2 Cured unsaturated polyester tensile properties

The UPE resin was mixed with a stirrer into the following formulation (by weight): 100 parts of UPE, 15 parts of styrene, 0.5 part of CoNa, and 1.0 part of MEKP. The mixture was poured into a mold and cured into bars at room temperature for 24 h followed by postcuring at 80 ºC for 3 h. After cooling, the bars were cut into eight strips with nominal dimensions of 165×19×6.4 mm³ using a band saw. They were shaped into dog-bone tensile specimens using a computer numerical controlled (CNC) machine. All tensile specimens were tested on an Instron 5869 universal testing machine under displacement control conditions in accordance with ASTM Standard D638-03 (ASTM 2004). Tensile strains were recorded by an Instron 2630-100 series extensometer.

5.1.2.3 KBFB-UPE shear bonding strengths

Eight specimens were prepared and tested. Kenaf strips (50×3 mm²) were cut from kenaf bast fiber ribbons. One side of these strips at one end was smoothed by sandpaper (120-Grit). These strips were dried in an oven for 5 h at 103 ºC to remove residual moisture. Shear specimens were prepared by pasting these kenaf strips onto cured 13×13×6.4 mm³ UPE blocks using a UPE resin paste of the same formulation used in the UPE tensile specimens. These specimens were cured at a room temperature for 24 h and followed by postcuring at 80 ºC for 3 h. A shear specimen schematic is shown in Figure 5.1. Testing was conducted on an Instron 5566 testing machine under displacement
control conditions. Shear failure loads were recorded and shear bonding strengths were calculated using:

\[
\tau_{fm} = \frac{F}{LW}
\]  \[5-1\]

where \( \tau_{fm} \) is the fiber-matrix shear bonding strength, and \( F, L, \) and \( W \) were the failure load, overlap surface length and width respectively.

Figure 5.1  A shear specimen schematic for fiber-matrix shear bonding strength testing.

5.1.2.4 Composite tensile properties

Three fixed fiber lengths were used: 1.72, 2.75, and 3.30 mm, equivalent to 22, 35 and 42 aspect ratios, respectively. At least two fiber loading levels were employed at each aspect ratio. At least five replicates were tested at each fiber length and loading combination.

Short KBFBs were prepared by grinding long KBF Bs in a Thomas Wiley Mill (Model 4). The short KBFBs were divided into three batches by screening the ground short KBFBs using three different sieves. Fifty short KBFBs were randomly selected
from each KBFB batch, and the average lengths of three KBFB batches were measured using a microscope. Figure 5.2 shows the length distribution of each KBFB batch.

![Figure 5.2 Length distributions of three batches of reinforcing kenaf bast fiber bundle.](image)

Composites were fabricated as previously described (Du et al. 2010a). The UPE resin was prepared employing 100 parts of UPE, 15 parts of styrene, and 1.5 parts of TBP catalyst by weight. Short KBFBs were made into pre-forms using the PVAc adhesive and were compounded with various amounts of UPE resin. Five resin-infused pre-formed mats were stacked to assemble one prepreg. The prepregs were then compression molded to kenaf-PVAc-UPE (KPU) composites at 175 °C under a pressure of 5 MPa. The KPU composite fiber loadings were calculated after the squeezed-out resin was sanded off.

Tensile specimens for tensile property testing were cut from 102×178 mm² KPU composites and shaped to dog-bone specimens using a CNC machine. Tensile testing was performed on an Instron 5869 universal testing machine under displacement control conditions according to ASTM standard 638-03 (ASTM 2004). The tensile strains were recorded by an Instron 2630-100 series extensometer.
5.3 Results and discussion

5.3.1 Fiber and matrix tensile properties, and fiber-matrix shear properties

Table 5.1 summarizes the tensile properties of KBFBs and the neat cured UPE, and the fiber-matrix shear bonding strength. The average density of KBFBs was 0.806 g/cm³ (tested at Micromeritics Analytical Services). Typical KBFB and cured UPE stress-strain curves are plotted in Figure 5.3. Figure 5.4 contains images of KBFB and neat cured UPE tensile specimen fracture surfaces. KBFBs clearly displayed brittle fiber breakage and the UPE specimens all failed due to flat (cleavage) fracture. According to the stress-strain plots and fracture surfaces, both KBFBs and the UPE are considered to be brittle materials.

Table 5.1 Physical and mechanical properties of kenaf and neat cured unsaturated polyester

<table>
<thead>
<tr>
<th>Properties</th>
<th>Data</th>
<th>Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kenaf fiber bundle</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Density (g/cm³)</td>
<td>0.806</td>
<td>Tested at Micromeritics Analytical Services</td>
</tr>
<tr>
<td>Elastic modulus (GPa)</td>
<td>19.2</td>
<td>Measured</td>
</tr>
<tr>
<td>Diameter (μm)</td>
<td>78.6</td>
<td>Measured</td>
</tr>
<tr>
<td>Strength (MPa)</td>
<td>260</td>
<td>Measured</td>
</tr>
<tr>
<td>Elongation (%)</td>
<td>1.22</td>
<td>Measured</td>
</tr>
<tr>
<td>Unsaturated polyester</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Density (g/cm³)</td>
<td>1.18</td>
<td>Measured</td>
</tr>
<tr>
<td>Elastic modulus (GPa)</td>
<td>3.46</td>
<td>Measured</td>
</tr>
<tr>
<td>Tensile strength (MPa)</td>
<td>24.4</td>
<td>Measured</td>
</tr>
<tr>
<td>Elongation (%)</td>
<td>0.725</td>
<td>Measured</td>
</tr>
<tr>
<td>Poisson ratio</td>
<td>0.37</td>
<td>Pascault 2002</td>
</tr>
<tr>
<td>Shear modulus (GPa)</td>
<td>1.26</td>
<td>Calculated, $G=E/2(1+\mu)$</td>
</tr>
<tr>
<td>Polyvinyl acetate</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Density (g/cm³)</td>
<td>1.19</td>
<td>Brandrup and Immergut 1989</td>
</tr>
<tr>
<td>Elastic modulus (GPa)</td>
<td>1.28-2.26</td>
<td>Daniels 1987</td>
</tr>
<tr>
<td>Tensile strength (MPa)</td>
<td>29.4-49.0</td>
<td>Daniels 1987</td>
</tr>
<tr>
<td>Fiber-matrix</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Shear strength (MPa)</td>
<td>6.36</td>
<td>Measured</td>
</tr>
<tr>
<td>Critical length (mm)</td>
<td>1.61</td>
<td>Calculated, $L_c = \sigma_{uf} r / \tau_{um-f}$</td>
</tr>
</tbody>
</table>
Figure 5.3  Typical stress-strain plots for kenaf bast fiber bundle (KBFB) and unsaturated polyester (UPE) tensile specimens.

Figure 5.4  Typical failure modes of kenaf bast fiber bundles (KBFBs) and the neat cured unsaturated polyester (UPE): a and b) scanning electron microscope (SEM) fracture surfaces of representative KBFB tensile specimens; c) fracture surfaces of UPE tensile specimens.
5.3.2 Kenaf bast fiber bundle/unsaturated polyester composites

Figures 5.5 a and b show two scanning electron microscopy (SEM) images of the composite tensile specimen fracture surfaces (Du. et al. 2010). Both fiber pullout and fiber breakage can be observed (Figure 5.5a). The composites had a moderate fiber-matrix interfacial bonding. The reinforcing fibers were mostly randomly aligned in a two-dimensional mat form (Figure 5.5b).

![SEM images of kenaf bast fiber bundle/unsaturated polyester composites](image_url)

Figure 5.5 A fracture surface of one kenaf bast fiber bundle-reinforced unsaturated polyester composite tensile specimen: a) composite failed with fiber breakage and fiber pullout; b) a fracture surface at low magnification (75X).

The polymer matrix in the composite can be considered as a polymer blend of the UPE and the PVAc. The PVAc, a fiber mat pre-forming binder has a similar density, modulus of elasticity, and tensile strength as the UPE matrix (Table 5.1). PVAc volume fraction is small in all cases (5.2-5.9 v%). Therefore, the UPE properties were taken as matrix properties in the composite tensile property calculation. Fiber weight percentages in the composites were converted to volume percentages using the densities of KBFBs and UPE (Table 5.1).
Composite tensile moduli were predicted using the Halpin-Tsai (Halpin 1969), the m-ROM (Cox 1952), the Mori-Tanaka (1973), and the self-consistent (Hill 1965) models.

The Halpin-Tsai model (Halpin 1969) for predicting the longitudinal and transverse stiffness of aligned short fiber-reinforced composites may be expressed as:

\[
\frac{E_l}{E_m} = \frac{1+2(l/d)\eta_f V_f}{1-\eta_f V_f} \tag{5-2}
\]

Where \( \eta_l = \frac{(E_f / E_m) - 1}{(E_f / E_m) + 2(l/d)} \)

\[
\frac{E_t}{E_m} = \frac{1+2\eta_t V_f}{1-\eta_t V_f} \tag{5-3}
\]

Where \( \eta_t = \frac{(E_f / E_m) - 1}{(E_f / E_m) + 2} \)

where \( E_l \) and \( E_t \) are composite longitudinal and transverse moduli of a unidirectional short fiber-reinforced composite; \( \eta_l \) and \( \eta_t \) is the length efficiency factor; \( V_f \) and \( E_f \) are the fiber volume fraction and the fiber tensile modulus; \( E_m \) is the matrix tensile modulus; \( l/d \) is the fiber aspect ratio. For the case where short fibers are randomly oriented in a two-dimensional mat, the composite’s modulus \( E_c \), can be computed using the following empirical equation (Agarwal et al. 2006):

\[
E_c = \frac{3}{8} E_l + \frac{5}{8} E_t \tag{5-4}
\]

Composite tensile modulus may also be calculated using the m-ROM (Cox 1952):

\[
E_c = \eta_o \eta_f V_f E_f + (1-V_f) E_m \tag{5-5}
\]
\[ \eta_l = 1 - \tanh[n(l / d)]/[n(l / d)] \quad [5-5] \]

\[ n = \frac{2G_m}{\sqrt{E_f \ln(2R/d)}} \quad [5-7] \]

where \( \eta_l \) is the length efficiency factor; \( \eta_\theta \) is the fiber orientation factor, which is assumed to be 0.33 for a two-dimensional random fiber composite (Cox 1952); KBFBs are assumed to be arranged in a square array, so \( 2R/d = [\pi/(4V_f)]^{0.5} \); the matrix shear modulus \( G_m \) (GPa) was estimated from:

\[ G_m = \frac{E_m}{2(1 + \nu)} \quad [5-8] \]

where \( \nu \) is the UPE’s Poisson ratio, taken as 0.37 (Pascault et al. 2002).

The effective moduli of KBFB/UPE composites were calculated using the classical Mori-Tanaka and Self-Consistent micromechanical models (cf., Mura 1991; Nemat-Nasser and Hori 1993; Qu 2006, Du et al. 2010b). Both of these effective medium approaches are based upon the Eshelby (1957) solution for the stress and strain fields inside and surrounding an ellipsoidal inclusion. In the Mori-Tanaka method (Mori and Tanaka 1973) the fourth-rank elastic stiffness tensor, \( \bar{L} \), for an effective continuum with (\( N \)-1) distinct aligned ellipsoidal heterogeneities may be expressed as:

\[ \bar{L} = \sum_{r=0}^{N} c_r L_r T_r \left[ \sum_{n=0}^{N} c_n T_n \right]^{-1} \quad [5-9] \]

where \( T_n = \left[ I + S_n (M_0 L_n - I) \right]^{-1} \)

is the fourth-rank local strain concentration tensor for the \( n^{th} \) phase, \( S_n \) is the fourth-rank Eshelby tensor for the \( n^{th} \) phase, \( L_n \) is the fourth-rank elastic stiffness tensor for the \( n^{th} \)
phase, \(c_n\) is the volume fraction of the \(n^{th}\) phase, and \(M_0\) is the fourth rank elastic compliance tensor for the matrix (cf., Mura 1991; Nemat-Nasser and Hori 1993; Qu 2006). Here \(n = 0\) is used to denote matrix properties. The fourth-rank effective compliance tensor, \(\mathbf{M}\), may be determined by inverting the effective stiffness tensor [5-9]. After performing requisite orientation averaging to account for the effect of two-dimensional randomly oriented fibers on the calculated effective properties (Fisher 2002), the compliance tensor may be expressed as a 6 by 6 matrix using Voigt notation. The in-plane effective modulus for the composite may be expressed as:

\[
E_c = \frac{1}{\mathbf{M}_{11}^{-1}}
\]  

[5-10]

Similarly, in the self-consistent model (Hill, 1965) the fourth-rank stiffness tensor for the effective continuum may be expressed as

\[
\mathbf{L} = \mathbf{L}_0 + \sum_{r=1}^{N} c_r \left( \mathbf{L}_r - \mathbf{L}_0 \right) \mathbf{T}_r
\]  

[5-11]

where

\[
\mathbf{T}_r = \left[ I + S_r \mathbf{L}_r^{-1}(\mathbf{L}_r - \mathbf{L}_0) \right]^{-1}
\]  

[5-12]

is the global strain concentration tensor. The effective compliance tensor and effective in-plane modulus for the composite may be determined in a fashion similar to that for the Mori-Tanaka method. Both the Mori-Tanaka and self-consistent methods account for the effect of weak interactions between adjacent fibers on the bulk composite properties. While these approaches are best suited for composites with fiber volume fractions, \(V_f < 0.5\), Mori-Tanaka and self-consistent estimates for \(E_c\) were developed in this study for
comparison purposes. Similarly, Voight upper bound and Reuss lower bound ROM estimates for $E_c$ were calculated in order to bound the range of elastic properties (cf., Agarwal et al. 2006), i.e.,

$$E_{cUPR} = V_f E_f + (1-V_f)E_m \quad \text{(Voigt Upper Bound)} \quad [5-13]$$

$$E_{cLWR} = \frac{E_f E_m}{V_f E_m + (1-V_f)E_f} \quad \text{(Reuss Lower Bound)} \quad [5-14]$$

The predicted tensile moduli by the Halpin-Tsai, m-ROM, Mori-Tanaka, Self-Consistent models against fiber loadings are plotted in Figure 5.6. The upper and lower bound ROM estimates for composite tensile moduli are also plotted for reference purposes; these models are best suited for predicting unidirectional continuous fiber-reinforced composite properties. Included in the figure are experimental data for KBFB composites. The relative errors between predicted and experimental tensile moduli are summarized in Table 5.2. The predicted tensile moduli by Halpin-Tsai, Mori-Tanaka, and Self-Consistent models agreed well with experimental values, though a large scatter exists.
Figure 5.6 Kenaf bast fiber bundle-reinforced unsaturated polyester composite experimental and computed tensile moduli versus fiber loadings. The curves plotted by the modified rule of mixtures (m-ROM), Halpin-Tsai (H-T), Mori-Tanaka (M-T), and Self-Consistent (S-C) models were computed tensile moduli of composites reinforced by KBFBs with aspect ratios of 20, 30, and 40, respectively, from bottom to top.

Table 5.2 Relative errors between composite experimental and theoretical Young’s modulus values.

<table>
<thead>
<tr>
<th>Aspect ratio</th>
<th>Fiber wt%</th>
<th>Fiber v%</th>
<th>Experiment (GPa)</th>
<th>Predicted (GPa/v%)</th>
<th>H-Ta</th>
<th>M-Tb</th>
<th>S-Cc</th>
<th>m-ROMd</th>
</tr>
</thead>
<tbody>
<tr>
<td>22</td>
<td>59.1</td>
<td>67.9</td>
<td>11.9</td>
<td>11.8 (0)</td>
<td>10.9 (-8)</td>
<td>12.0 (1)</td>
<td>5.3 (-56)</td>
<td></td>
</tr>
<tr>
<td>22</td>
<td>56.7</td>
<td>65.8</td>
<td>8.8</td>
<td>11.5 (31)</td>
<td>10.6 (21)</td>
<td>11.6 (32)</td>
<td>5.2 (-41)</td>
<td></td>
</tr>
<tr>
<td>35</td>
<td>62.0</td>
<td>70.6</td>
<td>12.3</td>
<td>12.4 (1)</td>
<td>11.4 (-7)</td>
<td>12.5 (2)</td>
<td>5.4 (-56)</td>
<td></td>
</tr>
<tr>
<td>35</td>
<td>60.8</td>
<td>69.4</td>
<td>13.3</td>
<td>12.2 (-8)</td>
<td>11.2 (-16)</td>
<td>12.3 (-8)</td>
<td>5.4 (-60)</td>
<td></td>
</tr>
<tr>
<td>43</td>
<td>65.3</td>
<td>73.5</td>
<td>12.1</td>
<td>12.9 (6)</td>
<td>11.9 (-2)</td>
<td>13.1 (8)</td>
<td>5.5 (-55)</td>
<td></td>
</tr>
<tr>
<td>43</td>
<td>67.7</td>
<td>75.5</td>
<td>12.0</td>
<td>13.3 (11)</td>
<td>12.3 (3)</td>
<td>13.5 (13)</td>
<td>5.6 (-53)</td>
<td></td>
</tr>
<tr>
<td>43</td>
<td>62.0</td>
<td>70.6</td>
<td>10.3</td>
<td>12.4 (20)</td>
<td>11.4 (10)</td>
<td>12.5 (21)</td>
<td>5.4 (-47)</td>
<td></td>
</tr>
</tbody>
</table>

a H-T: Halpin-Tsai.
b M-T: Mori-Tanaka.
c S-C: Self-consistent.
d m-ROM: modified rule of mixtures.

Values in parentheses were relative errors between predicted and experimental values.
The m-ROM significantly underestimated the experimental values. Baiardo et al. (2004) and Shibata et al. (2006) found good agreement between natural fiber-reinforced composite experimental tensile moduli and computed values by the m-ROM. Results from this study suggested Halpin-Tsai, Mori-Tanaka, and Self-Consistent models were better approximated the measured data over the range of fiber loadings considered here.

All models showed that the composite tensile moduli increase gradually with increasing fiber loadings, while the m-ROM suggested the lowest dependency of moduli on fiber loadings. In this work, fiber loadings as high as 75 v% still helped improve natural fiber-reinforced composite tensile moduli.

The predicted composite tensile moduli by the Halpin-Tsai, Mori-Tanaka, Self-Consistent, and m-ROM models (Figure 5.6) were insensitive to changes in fiber aspect ratio in the range 20-40. Figure 5.7 contains a plot of the calculated tensile modulus based on the Halpin-Tsai model as a function of fiber aspect ratio for a number of different fiber loadings. As can be seen from the figure, the composite modulus is asymptotically approaches a constant value, for a given fiber loading once the aspect ratio exceeds 15.
Composite tensile strengths were predicted using the Kelly-Tyson (1965) and Bowyer-Bader (1972) models that incorporated fiber aspect ratios and loadings. The Kelly-Tyson Model composite strength model may be expressed as:

\[ \sigma_c = \sigma_f \eta_l \eta_m V_f + E_m \varepsilon_c (1-V_f) \]  \hspace{1cm} [5-15]

where \( \sigma_c \) is the ultimate composite tensile strength; \( \sigma_f \) is the fiber tensile strength; \( E_m \) is the matrix tensile modulus; \( \varepsilon_c \) is the composite ultimate tensile strain; \( \eta_l \) is the fiber orientation factor whose values may lie between 0.167 and 1, when fibers are randomly aligned in a two-dimensional mat, then \( \eta_l = 1/3 \) (Cox 1952); \( \eta_l \) is the fiber length factor,

\[ \eta_l = 1 - L_c / (2L) \hspace{1cm} (L > L_c) \]  \hspace{1cm} [5-16]

\[ \eta_l = L_c / (2L) \hspace{1cm} (L < L_c) \]  \hspace{1cm} [5-17]

where \( L \) is the fiber length; and \( L_c \) is the critical fiber length in a matrix.
The critical fiber length, i.e., the length which is necessary for the maximum stress in the fiber to reach the fiber fracture stress $\sigma_f$, may be determined using (Kelly and Tyson 1965):

$$L_c = \frac{\sigma_f r}{\tau_{m-f}}$$  \hspace{1cm} [5-18]

where $r$ is the fiber radius, and $\tau_{m-f}$ is the fiber-matrix shear bonding strength. In this study, the shear bonding strength was found to be 6.36 MPa. Therefore, the KBFB critical length in the UPE matrix is 1.61 mm. The average fiber lengths from each fiber batch were used in the strength determination. The Bowyer-Bader composite strength model takes into account variable fiber lengths:

$$\sigma_c = \eta_0 \int_0^{L_c} \frac{\tau x V_x}{2r} dx + \eta_0 \int_{L_c}^{\infty} E_f \varepsilon_f (1 - \frac{E_f \varepsilon_f r}{2x \tau}) V_x dx + E_m \varepsilon_c (1 - V_f)$$  \hspace{1cm} [5-19]

where $L_i$ and $L_j$ are used to denote fiber lengths below and above the critical length, and $V_i$ and $V_j$ are their fiber corresponding volume fractions, $r$ is the radius of fibers and $\tau$ is the fiber-matrix shear strength.

The fiber volume fraction, $V(x)$, of fibers of length, $x$, may be expressed using a two-parameter Weibull distribution model:

$$V(x) = \frac{k \left( \frac{x}{\lambda} \right)^{k-1} \exp \left[ - \left( \frac{x}{\lambda} \right)^k \right]}{\lambda}$$  \hspace{1cm} [5-20]

where $k$ and $\lambda$ are shape and scale parameters, respectively. The shape and scale parameters were determined from the lengths of 50 randomly-selected specimens from each KBFB batch. Table 5.3 summarizes the estimated parameters used in the composite strength computations.
Table 5.3  Estimated scale and shape parameters of fiber length distributions from three kenaf bast fiber bundle batch.

<table>
<thead>
<tr>
<th></th>
<th>Shortest</th>
<th>Medium</th>
<th>Longest</th>
</tr>
</thead>
<tbody>
<tr>
<td>k</td>
<td>2.825</td>
<td>2.235</td>
<td>2.509</td>
</tr>
<tr>
<td>λ</td>
<td>1.932</td>
<td>3.114</td>
<td>3.803</td>
</tr>
</tbody>
</table>

The predicted composite tensile strengths obtained using the Kelly-Tyson model and experimental values are plotted as a function of fiber loading in Figure 5.8. The composite strengths computed by the Kelly-Tyson and Bowyer-Bader models and corresponding errors compared to the experimental results were listed in Table 5.4.

Figure 5.8  Kenaf bast fiber bundle-reinforced unsaturated polyester composite experimental and computed tensile strengths versus fiber loadings. The three curves are computed by the Kelly-Tyson model for composites reinforced by KBFBs with aspect ratios of 20, 30, and 40 respectively, from bottom to top.
### Table 5.4: Relative errors between composite experimental and theoretical tensile strength values.

<table>
<thead>
<tr>
<th>Aspect ratio</th>
<th>Fiber Loading</th>
<th>Composite tensile strength Predicted (GPa/%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Experiment</td>
<td>Predicted Kelly-Tyson</td>
</tr>
<tr>
<td>22</td>
<td>59.1</td>
<td>51.0</td>
</tr>
<tr>
<td>22</td>
<td>67.9</td>
<td>51.0</td>
</tr>
<tr>
<td>35</td>
<td>62.0</td>
<td>49.7</td>
</tr>
<tr>
<td>35</td>
<td>70.6</td>
<td>53.1</td>
</tr>
<tr>
<td>35</td>
<td>60.8</td>
<td>53.7</td>
</tr>
<tr>
<td>43</td>
<td>62.0</td>
<td>56.3</td>
</tr>
<tr>
<td>43</td>
<td>67.7</td>
<td>52.3 (11)</td>
</tr>
</tbody>
</table>

Note: *Values in parentheses were relative errors between predicted and experimental values.

At the lowest fiber aspect ratio (22), the Kelly-Tyson model was conservative with relative errors of -26 and -12%. As fiber lengths increased, the predicted values gradually approached experimental values. The relative errors descended to -1 to -9% for an aspect ratio of 35 and -3 to 11% for 42. Experimentally-obtained tensile strengths were fairly accurately predicted by the Kelly-Tyson model when the fiber aspect ratio is above 35.

The Bowyer-Bader model underestimated the composite tensile strengths, approximately 15%, than the Kelly-Tyson model. However, similar to the Kelly-Tyson model, the relative errors decreased with increasing aspect ratios.

The composite strengths, in general, increased with increasing fiber loadings except one abnormal finding when the fiber aspect ratio was 35. This might caused by variation. Literature showed that fiber loadings are generally beneficial to composite tensile strengths. However, at high loading levels, the results might vary. Some researchers reported composite tensile strengths consistently increased with fiber loadings. Takagi and Ichihara (2004) reported composite tensile strengths increased as
the fiber loading increased from 0 to 50 wt%. Devi et al. (1997) reported increasing tensile strengths as the fiber loadings increased from 10 to 40 wt%. However, decreasing tensile strengths at higher fiber loadings were also reported (Baiardo et al. 2004, Xue et al. 2007, Shibata et al. 2006). This study shows a persistent enhancement trend in composite tensile strengths as fiber loading increase to 75 v%.

Experimental and computed composite tensile strengths are plotted as a function of fiber aspect ratios in Figure 5.9. Theoretically, as the fiber aspect ratios increase, the computed tensile strengths increase at a decreasing rate. At aspect ratios of 40 and above, the ascending trend flattens and becomes linear. Further investigation on the effect of fiber aspect ratio on composite tensile strengths is needed due to lack of experimental results at fixed fiber loading levels.

![Figure 5.9](image)

**Figure 5.9** Kenaf bast fiber bundle-reinforced unsaturated polyester composite experimental and calculated tensile strengths versus fiber aspect ratios. The three curves were computed by the Kelly-Tyson model for composites with fiber loadings of 65, 70, and 75 v%, respectively, from bottom to top.
5.4 Summary and conclusions

Tensile properties of KBFB-reinforced UPE composites with fiber loadings high up to 75 v% were studied. Both composite tensile moduli and strengths increased with increasing fiber loading persistently. Composite tensile moduli were not sensitive to fiber aspect ratios in the studied range. Experimental results were compared to computed values using classical models. Composite tensile moduli were accurately predicted by the Halpin-Tsai, the Mori-Tanaka, and the Self-Consistent models. Composite tensile strengths were accurately computed by the Kelly-Tyson model at higher aspect ratio levels. This study confirmed the possibility of the concept that to increase fiber loadings in natural fiber-reinforced composites reduces the consumption of petroleum-based polymer which in turn saves the manufacture cost, while still results in improved composite tensile properties.
CHAPTER VI
RELIABILITY AND COST-PERFORMANCE OF SHORT KENAF FIBER-REINFORCED UNSATURATED POLYESTER COMPOSITES

6.1 Materials and methods

6.1.1 Materials

KBFBs were supplied by Kengro Corporation. The UPE resin (Aropol Q-6585), provided by Ashland Chemical Company, was used as the matrix polymer. The diluent, styrene, and the free radical initiator, t-butyl perbenzoate (TBP), were purchased from Fisher Scientific Inc. A poly(vinyl acetate) (PVAc) water emulsion (solid content: 46%), provided by Tailored Chemical Products, Inc., was used as a mat pre-forming binder.

6.1.2 Composite fabrication

Composites were fabricated as previously described (Du et al. 2010a). Long KBFBs were first ground into short fibers with an average length of 3.3 mm. Then, these short KBFBs were pre-formed to loose mats using the PVAc adhesive, and these pre-formed mats were infused with predetermined amounts of the UPE resin. The prepregs, assembled by mats, were then compression-molded into composites. The detailed process parameters for the UPE resin formulation, KBFB mat pre-formatting, and compression molding were reported previously. The target fiber loadings were 50-60 wt%. The composite fiber loadings were calculated after curing and all the squeezed-out
resin was sanded off. A total of 25, 44, and 40 specimens, respectively, were prepared for tensile, flexural, and impact testing.

6.1.3 Testing

The resultant composite samples were cut to tensile specimens using a CNC machine and were tested on an Instron 5869 universal testing machine under displacement control conditions in accordance with ASTM Standard D638-03 (ASTM 2004). The testing speed was set at 4 mm/min, and the tensile strain was recorded by an Instron 2630-100 series extensometer. Flexural test specimens were studied on the same Instron 5869 machine under displacement control conditions in accordance with ASTM Standard D790-03 (ASTM 2003). The support span was set at 60 mm, and the testing speed was set at 4 mm/min. Impact properties were tested using an Izod fixture on an Instron Dynatup 9250HV Instrumented Drop Tower in accordance with ASTM Standard D256-06 (ASTM 2006).

6.2 Results and discussion

6.2.1 Statistical distribution

Table 6.1 summarizes the fitted distribution parameters for each evaluated strength property. The experimental data histograms, together with their normal, two-parameter Weibull, gamma, and lognormal distribution function fitted curves, are plotted in Figures 6.1a, b, and c, for the composites’ tensile, flexural, and impact strengths, respectively. The two-parameter Weibull function-fitted curves showed a skew to the left for all strength properties. The lognormal and gamma tensile and flexural strength distribution curves were almost symmetrical, very close to normal distribution curves.
Gamma and lognormal impact strength distribution curves skewed slightly to the right, but they were still very close to the normal distribution curves.

Table 6.1 Summary of distribution parameters of each distribution for composite tensile, flexural, and impact strengths, corresponding fiber loading range, and samples size of tested specimens.

<table>
<thead>
<tr>
<th></th>
<th>Tensile strength (MPa)</th>
<th>Flexural strength (MPa)</th>
<th>Impact strength (kJ/m(^2))</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Normal</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mean</td>
<td>53.10</td>
<td>83.64</td>
<td>6.25</td>
</tr>
<tr>
<td>COV (%)</td>
<td>9.52</td>
<td>6.78</td>
<td>12.71</td>
</tr>
<tr>
<td>Fiber loading (wt%)</td>
<td>58-63</td>
<td>62-68</td>
<td>53-63</td>
</tr>
<tr>
<td><strong>Two-parameter Weibull distribution</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Scale parameter</td>
<td>55.35</td>
<td>86.22</td>
<td>6.59</td>
</tr>
<tr>
<td>Shape parameter</td>
<td>11.93</td>
<td>16.79</td>
<td>9.10</td>
</tr>
<tr>
<td>Mean</td>
<td>53.03</td>
<td>83.54</td>
<td>6.24</td>
</tr>
<tr>
<td><strong>Gamma distribution</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Scale parameter</td>
<td>0.47</td>
<td>0.38</td>
<td>0.10</td>
</tr>
<tr>
<td>Shape parameter</td>
<td>113.65</td>
<td>219.43</td>
<td>60.90</td>
</tr>
<tr>
<td>Mean</td>
<td>53.10</td>
<td>83.64</td>
<td>6.25</td>
</tr>
<tr>
<td><strong>Lognormal distribution</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Scale parameter</td>
<td>3.97</td>
<td>4.42</td>
<td>1.82</td>
</tr>
<tr>
<td>Shape parameter</td>
<td>0.10</td>
<td>0.07</td>
<td>0.13</td>
</tr>
<tr>
<td>Mean</td>
<td>53.11</td>
<td>83.64</td>
<td>6.25</td>
</tr>
<tr>
<td>Sample size</td>
<td>25</td>
<td>44</td>
<td>40</td>
</tr>
</tbody>
</table>
Figure 6.1  Probability density functions of: a) tensile strength, b) flexural strength, and c) impact strength for kenaf bast fiber bundles-reinforced unsaturated polyester composites.
The majority of experimental tensile strengths ranged from 45 to 60 MPa but a few specimens possessed tensile strengths above 60 or below 45 MPa (Figure 6.1a). The probability density curve drops rapidly when composite tensile strengths exceed 60 MPa, unlike the gradual ascending pattern for specimens with strengths less than 50 MPa.

Most measured composite flexural strengths were found in the range from 75 to 90 MPa (Figure 6.1b). The probability density curve increased rapidly from an initial flexural strength of 65 MPa to maximum values occurring near 83 MPa. The most frequent values occurred from 85 to 90 MPa, and the number of occurrences decreased as the flexural strength approached 100 MPa. The peak of the two-parameter Weibull distribution probability density curve coincided with that of experimental data’s histogram.
Impact strengths ranged from 4 to 8 kJ/m$^2$ with the most frequent measurements falling between 6.4 to 7.2 kJ/m$^2$ (Figure 6.1c). The impact strength histogram skewed to the left, exhibiting an unsymmetrical distribution mode. Only the two-parameter Weibull function’s peak closely matched that of the histogram and displayed the skew to the left.

6.2.2 Reliability analysis and design value

The distribution parameter estimators were calculated based on the maximum likelihood method using the commercially-available program-SAS. This method has been frequently used for the two-parameter Weibull distribution (Rust et al. 1989). The two-parameter Weibull survival probability curves for all three strength properties are plotted in Figures 6.2a, b, and c. These fits of the two-parameter Weibull distribution function survival curves closely matched the experimental data.
Figure 6.2  Weibull distribution function fitted survival curves for the experimental data from kenaf bast fiber bundle-reinforced unsaturated polyester composite: (a) tensile, (b) flexural, and (c) impact strengths with experimental data.
The five-percentile values (95% probability of survival) have normally been chosen as design values in structural engineering (Ellingwood 2000). Table 6.2 summarizes the design values based on five-percentile values for two-parameter Weibull, normal, gamma, and lognormal distributions, where a conservative estimate of strength is typically desired. The value of the two-parameter Weibull five-percentile tensile strength was 3% less than the five-percentile experimental value. Other distribution models gave five-percentile tensile strength values greater than the five-percentile experimental value. All four distribution models gave five-percentile flexural strength values less than the five-percentile experimental value. The Weibull distribution provided the most conservative value, which was 3.9% lower than the five-percentile experimental value. The Weibull design value of the impact strength was 0.4% less than the five-percentile experimental value and this was also the closest of the models to the five-percentile experimental value. Other model distributions provided higher five-percentile impact strength values than the five-percentile experimental value. The design values based on ten-percentile values are also summarized in Table 6.2 for reference. The two-parameter Weibull model still gave the most conservative design values among other three models. The two-parameter Weibull distribution provided the most conservative design values for all these strength properties. This is the same results found by Alqam et al.’s results (2002).
Table 6.2  Five and ten-percentile values for each distribution.

<table>
<thead>
<tr>
<th>Model</th>
<th>Tensile strength (MPa)</th>
<th>Flexural strength (MPa)</th>
<th>Impact strength (kJ/m²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Five-percentile value</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Experimental</td>
<td>44.50</td>
<td>75.19</td>
<td>4.77</td>
</tr>
<tr>
<td>Normal</td>
<td>44.78</td>
<td>74.31</td>
<td>4.94</td>
</tr>
<tr>
<td>Two-parameter Weibull</td>
<td>43.15</td>
<td>72.24</td>
<td>4.75</td>
</tr>
<tr>
<td>Gamma</td>
<td>45.18</td>
<td>74.57</td>
<td>4.99</td>
</tr>
<tr>
<td>Lognormal</td>
<td>45.13</td>
<td>74.54</td>
<td>4.99</td>
</tr>
<tr>
<td>Ten-percentile value</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Experimental</td>
<td>46.49</td>
<td>76.36</td>
<td>4.91</td>
</tr>
<tr>
<td>Normal</td>
<td>46.62</td>
<td>76.37</td>
<td>5.23</td>
</tr>
<tr>
<td>Two-parameter Weibull</td>
<td>45.83</td>
<td>75.40</td>
<td>5.15</td>
</tr>
<tr>
<td>Gamma</td>
<td>46.82</td>
<td>76.49</td>
<td>5.25</td>
</tr>
<tr>
<td>Lognormal</td>
<td>46.73</td>
<td>76.42</td>
<td>5.24</td>
</tr>
</tbody>
</table>

6.2.3 Tensile and flexural strength discrepancy

The two-parameter Weibull strength distribution curves can be plotted according to following equation (Weibull 1939).

\[
S(X) = \exp[-(X / \lambda)^k]
\]  

[6-1]

Here \(S(X)\) is the probability of survival, and \(X\) is an arbitrary applied stress.

The Weibull scale parameters, \(\lambda\) (Table 6.1), fitted from experimental tensile strengths were referenced to calculate the flexural strength scale parameters according to the specimen dimensions using three flaw distribution models: volume, edge, and surface, respectively (Robinson 1972). Table 6.3 summarizes the calculated flexural strength scale parameters. The corresponding predicted survival curves are shown in Figure 6.3.
Table 6.3 Scale parameters $\lambda$, computed by three models.

<table>
<thead>
<tr>
<th>Model</th>
<th>Tensile MPa</th>
<th>Flexural MPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>Volume</td>
<td>55.35</td>
<td>90.51</td>
</tr>
<tr>
<td>Surface</td>
<td>55.35</td>
<td>73.21</td>
</tr>
<tr>
<td>Edge</td>
<td>55.35</td>
<td>71.61</td>
</tr>
<tr>
<td>Experimental</td>
<td>55.35</td>
<td>86.22</td>
</tr>
</tbody>
</table>

Shape parameter $k$: 11.93

Figure 6.3 Composite flexural strength survival curves of kenaf bast fiber-reinforced unsaturated polyester composites computed with tensile strengths using volume, surface, and edge models versus the experimental results.

The scale parameter obtained using the volume flaw model (90.51) was the closest to experimental result-fitted value (86.22). At lower flexural strength levels, the volume flaw model’s predicted values agreed well with experimental data. At higher flexural strengths, the predicted values of the scale parameters gradually became higher than those from experimental data. This difference at higher strength levels was ascribed to the different shape parameters. Experimental flexural strengths have a shape parameter of 16.79 (Table 6.2), higher than that of tensile strengths (11.93). Thus, tensile
strengths had a larger scatter than flexural strengths. The tensile strength distribution shape parameters were not suitable for flexural strength distributions. This finding does not conform to the results of Knight and Hahn (1975) or those of Bullock (1974), where similar shape parameters were found for both of these strengths.

6.2.4 Cost analysis

Although possessing lower absolute strengths, natural fiber-reinforced composite materials can compete with glass fiber-reinforced composites in terms of specific properties and costs. D’Almeida (2001) proposed cost analysis equation \([6-2]\) to calculate the price per unit length of a composite specimen, \(Q\), subjected to an applied three-point bending load \(P\).

\[
Q = \frac{3}{2} \frac{L \rho_c}{t} \frac{X_c}{\sigma_c} P \tag{6-2}
\]

In Equation 6-2, \(L\) and \(t\) are the test span and the specimen thickness, \(\rho_c\) and \(\sigma_c\) are the composite’s density and flexural strength, and \(X_c\) is the total price of all components. Equation [6-2] can also be written as:

\[
Q = \beta \cdot P \tag{6-3}
\]

\[
\beta = \frac{3}{2} \frac{L \rho_c}{t} \frac{X_c}{\sigma_c} \tag{6-4}
\]

The cost parameter \(\beta\) is a constant for a given composite geometry.

Similarly, the price per unit of a composite tensile specimen can be calculated.
In these equations, \( \rho_c \) and \( \sigma_c \) are the composite’s density and tensile strength, and \( X_c \) is the total price of all components. Note that the cost parameter \( \beta \) for a composite subjected to a tensile load is independent of the composite’s geometry.

In the same manner, the price per unit of a composite can be calculated for the material subjected to impact loads.

\[
Q = \beta \cdot E
\]  \hspace{1cm} [6-8]

\[
\beta = \frac{\rho_c}{I_c} X_c
\]  \hspace{1cm} [6-9]

In equations [6-8] and [6-9], \( E \) is the absorbed energy, and \( I_c \) is the composite impact strength.

The composite with a lower value of parameter \( \beta \) will be more cost-effective to carry the same load or to resist the same amount of impact energy. A cost-effectiveness coefficient \( k \), defined in equation [6-10] is proposed to compare kenaf fiber-reinforced UPE composites with commercial glass fiber-reinforced SMCs of each type of load.

\[
K = \frac{\beta_c}{\beta_{SMC}}
\]  \hspace{1cm} [6-10]
Here, $\beta_C$ and $\beta_{SMC}$ are the cost parameters for the KBFB-reinforced composite and the glass fiber-reinforced SMC. The cost-effectiveness coefficients of kenaf fiber-reinforced UPE composites subjected to tensile ($K_T$), flexural ($K_F$), and impact loads ($K_I$) can be calculated by equations [6-11] to [6-13].

\[
K_T = \frac{\rho_c / \sigma_{C-Tension} \cdot X_C}{\rho_{SMC} / \sigma_{SMC-Tension} \cdot X_{SMC}} \quad [6-11]
\]

\[
K_F = \frac{\rho_c / \sigma_{C-Flexure} \cdot X_C}{\rho_{SMC} / \sigma_{SMC-Flexure} \cdot X_{SMC}} \quad [6-12]
\]

\[
K_I = \frac{\rho_c / I_C \cdot X_C}{\rho_{SMC} / I_{SMC} \cdot X_{SMC}} \quad [6-13]
\]

When a KBFB-reinforced composite is more cost-effective than the glass fiber-reinforced SMC, then the corresponding value of $k$ is less than 1.

Table 6.4 summarizes the price of each constituent of the kenaf fiber-reinforced composite and a typical glass fiber-reinforced SMC (R25 SMC). Prices of UPE and filler for SMC were obtained from one automotive component manufacturer. The weight percentage of each component, as well as tensile, flexural, and impact strengths of R25 SMCs are listed in Table 6.4. These data were used to calculate the cost-effectiveness coefficients for kenaf fiber-reinforced composites for comparison to glass fiber-reinforced R25 SMCs. The cost coefficients $K_T$, $K_F$, and $K_I$ are also plotted against fiber loadings for tensile, flexural, and impact loads (Figures 6.4a, b, and c).
Table 6.4  The component prices, densities and mechanical properties of kenaf bast fiber bundle (KBFB) and glass fiber-reinforced unsaturated polyester (UPE) composites.

<table>
<thead>
<tr>
<th>KBFB-reinforced composites</th>
<th>Price ($/kg)</th>
<th>Composite cost ($/kg)</th>
<th>Density (g/cm$^3$)</th>
<th>Tensile strength (MPa)</th>
<th>Flexural strength (MPa)</th>
<th>Impact (kJ/m$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.44-0.55</td>
<td>1.03</td>
<td>1.2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Kenaf fiber</td>
<td>2.65</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>UPE</td>
<td>1.30</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Zampaloni et al. 2007</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Composite cost ($/kg)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>UPE</td>
<td>2.65</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PVAc</td>
<td>1.30</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Manufacturer</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SMC (R25)</td>
<td>3.25</td>
<td>1.59</td>
<td>1.85</td>
<td>73</td>
<td>178</td>
<td>75</td>
</tr>
<tr>
<td>Price ($/kg)</td>
<td>2.65</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>UPE (25%)</td>
<td>0.22</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mohanty et al. 2000</td>
<td></td>
<td></td>
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<td></td>
<td></td>
<td></td>
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<tr>
<td>European Alliance 2001</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tensile strength (MPa)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Flexural strength (MPa)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Impact (kJ/m$^2$)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Figure 6.4 Cost coefficients, KT, KF, and KI, of kenaf bast fiber bundle-reinforced unsaturated polyester composites for carrying: (a) tensile loads, (b) flexural loads, and (c) impact loads.
In general, these coefficients decrease, i.e., KBFB composites become more cost-effective with increasing fiber loading. KBFB-reinforced composites become more cost-effective than glass fiber-reinforced SMCs for tensile and flexural applications when the fiber loading exceeds 51.2 wt% and 56.3 wt%, respectively. These observations suggest kenaf fiber-reinforced composites are viable candidate replacement materials for glass fiber-reinforced SMCs for carrying tensile or flexural loads within the fiber weight fraction range considered here (50-70 wt%).

KBFB-reinforced composites were less cost effective than glass fiber-reinforced SMCs subjected to impact loads. The cost coefficient $K_I$ for composites carrying impact loads is always substantially higher than 1, although this coefficient decreased from 6.50 to 3.19 as the fiber weight percentage increased from 50 to 70 %. This could possibly attribute to energy absorption due to the distributed brittle fracture of the glass fibers; KBFB have no similar energy dissipation mechanism. Thus, kenaf fiber-reinforced composites cannot compete in cost with glass fiber-reinforced SMCs for carrying impact loads.

6.3 Summary and conclusions

The statistical distributions of KBFB-reinforced UPE composites’ tensile, flexural, and impact strengths were studied. The two-parameter Weibull model was found to be appropriate to describe the unsymmetrical distribution in observed composite strengths. The five-percentile values calculated from the two-parameter Weibull model were used to determine reliable tensile, flexural, and impact strengths design values: 43.15 MPa, 72.24 MPa, and 4.75 kJ/m², respectively. The composite’s flexural strengths
scale parameter can be calculated by tensile strengths using the volume flaw model. However, the tensile strength had a larger scatter than the flexural strength.

Cost-effectiveness analysis suggested that KBFB-reinforced UPE composites were more cost-effective than glass fiber-reinforced SMCs for carrying tensile and flexural loads when their fiber loadings were higher than 51.24 and 56.29 wt%. However, these composites were less cost-effective than SMCs for applications as impact resistant materials.
CHAPTER VII
WATER RESISTANCE AND METHODS FOR MECHANICAL
PROPERTIES IMPROVEMENT OF NATURAL FIBER/
THERMOSET POLYMER COMPOSITES

7.1 Materials and methods

7.1 1 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 = 47000, maleic anhydride <1.0 wt%), provided by Eastman Chemical Company, were used as mat pre-forming binders. Polyurethane (Helmsman spar urethane), purchased from Lowe’s store, and wax (Mainstays candle), purchased from Wal-Mart store, 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.
7.1.2 Experimental design

7.1.2.1 Composite fabrication

Composites were fabricated as previously described in full detail (Du et al. 2010a). Long 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 pre-forms using PVAc or MAPP binders. UPE and VE resins were prepared employing 100 parts of UPE or VE, 15 parts of styrene, and 2 parts of TBP by weight. Mats were pre-formed using either PVAc or MAPP. Five pre-formed mats were infused with UPE or VE resins to form one prepreg. The resultant preps were then compression molded into composites.

7.1.2.2 Water absorption

Kenaf-PVAc-UPE composites were cut into $25.4 \times 76.2 \text{ mm}^2$ 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, $25.4 \times 76.2 \text{ mm}^2$ 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 (ASTM 2006). Specimens were immersed in de-ionized water at 23°C, and the weight of each specimen was recorded after 2h and 24 h. Intermediate measurements were recorded for some kenaf-PVAc-UPE composite. A few kenaf-PVAc-UPE and
kenaf-PVAc-VE specimens were kept submerged under water until their moisture contents reached equilibrium.

7.1.2.3 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 7.1 summarizes the amount of each component and the corresponding styrene content for each UPE resin formulation. The mixed UPE resins were cured at ambient temperature for 24 h and postcured in an oven at 80 ºC for 3 h.

Table 7.1 Formulation of unsaturated polyester (UPE), styrene, methyl ethyl ketone peroxide (MEKP), cobalt naphthenate, and styrene concentration of the cured UPE.

<table>
<thead>
<tr>
<th></th>
<th>Parts (weight)</th>
</tr>
</thead>
<tbody>
<tr>
<td>UPE</td>
<td>100 100 100 100</td>
</tr>
<tr>
<td>UPE solid</td>
<td>65  65  65  65</td>
</tr>
<tr>
<td>Styrene (in UPE)</td>
<td>35  35  35  35</td>
</tr>
<tr>
<td>Styrene (additional)</td>
<td>0  5  15  30</td>
</tr>
<tr>
<td>MEKP</td>
<td>1  1  1  1</td>
</tr>
<tr>
<td>cobalt naphthenate</td>
<td>0.5 0.5 0.5 0.5</td>
</tr>
<tr>
<td>Styrene concentration (wt%)</td>
<td>34.5 37.6 42.9 49.4</td>
</tr>
</tbody>
</table>

The cured resins were shaped to dog-bone tensile specimens using a computer numerical controlled (CNC) 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.
7.1.2.4 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, impact properties of resultant composites were determined by ASTM Standard D790-03 (ASTM 2003) and D256-06 (ASTM 2006).

7.1.2.5 Fiber surface treatment

Eighteen $50 \times 3 \text{ mm}^2$ kenaf strips were cut from kenaf bast fiber ribbons. One side at the end of these strips ($3 \times 5 \text{ mm}^2$) was smoothed by sand paper (120-Grit). Six strips were dried in an oven at 103 °C for 5 h to remove the residual moisture.

The remaining twelve strips were prepared for investigating the fiber-matrix adhesion improvement via laser and plasma radiation. The laser beam was generated by an Nd: YAG pumped dye laser with a single pulse energy of 10-15 mJ. The laser had a single 532 nm wavelength with a 20 Hz pulse repetition rate. The smoothed $3 \times 5 \text{ mm}^2$ surfaces of six kenaf strips were treated with the oscillating 0.5-1 mm$^2$ laser beam focal point for 30 s.

The remaining six samples were treated by an atmospheric pressure argon microwave-induced plasma jet, which was excited by a 2.45 GHz plasma source. The plasma power was 48 W. The argon flow rate was 0.1-0.3 L/min. The non-thermal plasma gas temperature was close to 450 K. The plasma jet is shown in Figure 7.1. 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 smoothed $3 \times 5 \text{ mm}^2$ surfaces of kenaf strips were exposed to a combination of thermal heating and chemical bath for 1 s within the vicinity of the plasma tip.
Shear specimens were prepared by pasting all eighteen control or treated kenaf strips onto 13×13×6.4 mm³ UPE blocks using the UPE resin as the adhesive. These specimens were cured at ambient temperature for 24 h and postcured at 80ºC for 3 h. Shear forces were determined on an Instron 5566 universal testing machine and shear strengths were calculated by:

$$\tau = \frac{F}{(L \cdot W)}$$

where \(F\), \(L\), and \(W\) were the failure force, overlap length and width respectively.

7.2 Results and discussion

7.2.1 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 7.2. The average weight increase of kenaf-PVAc-UPE composites after 24 h of water immersion was 22.1 wt%. The average weight increases of the kenaf-MAPP-UPE and kenaf-PVAc-VE
composites were 10.2 to 11.8 wt%, respectively, both significantly lower than that of kenaf-PVAc-UPE. 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.

![Figure 7.2](image1.png)

**Figure 7.2** 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.

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 which can promote adhesion to the hydroxyls of the lignocellulosic fibers (Mallick 1988). The lower water uptake by the kenaf-MAPP-UPE composites is the results of MAPP reaction on the fiber surfaces which improves compatibility between KBFBs and UPE.

Figure 7.3 shows the water uptake of UPE-coated kenaf-PVAc-UPE composites after 24 h. The control specimens (edges were not sealed) had an average water uptake of 8.3% versus the water uptake of 4.3, 3.3, and 2.1 wt%, respectively, for the UPE-
coated kenaf-PVAc-UPE composites which were edge-sealed by wax, polyurethane (PU), and UPE. However, none of these absorbed less than 0.8 wt% water, which is the specification requirement for glass fiber/UPE composites from an automotive part manufacturer.

![Weight increase of UPE-coated kenaf-PVAc-unsaturated polyester (KPU) composites after edge-sealing after a 24-hour water immersion test](attachment:image)

**Figure 7.3** Weight increases of UPE-coated kenaf-PVAc-unsaturated polyester (KPU) composites after edge-sealing after a 24-hour water immersion test (Control: edge not sealed. Wax: edge-sealed by wax. PU: edge-sealed by poly urethane. UPE: edge-sealed by unsaturated polyester).

After 24 h 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 7.4. After 650 h, the composite moisture contents have almost approached equilibrium. Approximately, half of the water was absorbed in the first 24 h.

![Figure 7.4](image_url)  
Weight increases of kenaf-PVAc-unsaturated polyester (KPU) and kenaf-PVAc-vinyl ester (KPV) composites versus time during a long-term water immersion test.

### 7.2.2 Styrene content

Tensile moduli and strengths of the cured UPE resin (no fibers) versus styrene content and typical stress-strain curves are plotted in Figure 7.5. 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 37.6 wt%. Thus, five parts styrene per 100 parts of the commercial UPE resin was the optimized blending
ratio for styrene and this UPE resin in order to achieve the maximum matrix tensile properties. This differs with the results of Sanchez et al. (2000), where the optimized styrene content was 24 wt%. The cured UPE failed in cleavage fracture modes. This leads to larger scatter in tensile strengths than moduli.

Figure 7.5  Tensile properties of cured unsaturated polyester (UPE) versus styrene concentrations by weight: a) tensile properties versus styrene concentrations; b) typical stress-strain plots of UPE specimens at four styrene concentration levels.

7.2.3 Molding pressure

Figure 7.6 illustrates the relationship between densities, impact strengths, and flexural properties of the kenaf-PVAc-UPE composites and molding pressures. 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 (Deus et al. 2005).
Figure 7.6  Composite properties versus compression molding pressures: a) densities, b) impact strengths, c) flexural strengths, d) flexural modulus.

Composite densities increased as molding pressures increased from 3 to 5 MPa (Figure 7.6a). High pressures reduced the void volume and improved the fiber-matrix interface in the composite, thereby increasing composite densities. Densities leveled off when the molding pressure increased from 5 to 7 MPa, so the composites weren’t further compressed.

Composite impact and flexural strengths increased as molding pressures increase from 3 to 7 MPa (Figures 7.6 b and c). Statistical analyses showed that, at the 5% significance level, increasing the molding pressures from 3 and 5 MPa significantly improved composite flexural and impact strengths. A further increase in molding pressures from 5 and 7 MPa caused only a marginally significant increase in composite flexural and impact strengths. 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 increase significantly as molding pressures increase from 3 to 5 MPa (Figure 7.6d), and then slightly decrease as molding pressures are increased to 7 MPa. However, this decrease was not statistically significant. The results indicate a 5 MPa molding pressure was the optimum to achieve the highest flexural moduli, and 7 MPa was the best for flexural and impact strengths.

### 7.2.4 Laser and plasma treatments

The fiber-matrix shear strengths of control and laser-treated and argon plasma-exposed kenaf strips and the UPE matrix are illustrated in Figure 7.7. It is easily seen that the laser and plasma treated kenaf strips have greatly improved fiber-matrix shear strengths 91.6 and 83.3%, respectively. Statistical analyses suggested these laser and plasma treatment led to significant and marginally significant fiber-matrix shear strength increases. These experimental results were similar to conclusions reached by Morales et al. (2006). The laser and plasma exposures are environmentally-friendly methods for enhancing interface compatibility in natural fiber-reinforced composites.

![Graph showing shear strength comparison](image)

**Figure 7.7** The interfacial shear strengths of untreated and laser or plasma radiation treated kenaf strips and the cured unsaturated polyester resin.
7.3 Summary and conclusions

The large water uptake by the kenaf-PVAc-UPE composites was due to the hydrophilic nature of both kenaf fiber bast fibers and PVAc. Encapsulation was an effective way to improve composite water resistance. The employment of a VE matrix or MAPP as a mat pre-forming binder significantly improved composite water resistance properties.

The commercial UPE used in this study exhibited highest tensile properties when the styrene content is 37-38 wt%. 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.


