Methods of Processing Kenaf Chopped Strand Mats for Manufacturing Test Specimens and Composite Structures

Joshua W. Heil
Utah State University

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METHODS OF PROCESSING KENAF CHOPPED STRAND MATS FOR
MANUFACTURING TEST SPECIMENS AND COMPOSITE STRUCTURES

by

Joshua W. Heil

A thesis submitted in partial fulfillment
of the requirements for the degree
of
MASTER OF SCIENCE
in
Mechanical Engineering

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Logan, Utah

2015
Abstract

Methods of Processing Kenaf Chopped Strand Mats for Manufacturing Test Specimens and Composite Structures

by

Joshua W. Heil, Master of Science
Utah State University, 2015

Major Professor: Dr. Thomas H. Fronk
Department: Mechanical and Aerospace Engineering

Bio-composites are increasing in demand due to governmental incentives across the globe for both environmental and human health reasons. The ideal bio-composite is renewable, recyclable, available, and non-toxic. To properly engineer bio-composite products, the physical properties of the fibers as well as fiber/matrix interactions must be known. The problem lies in the fact that many suitable natural fibers are not currently available in a material form that may be easily worked with. This research investigates methods to process raw kenaf (hibiscus cannabinus) on a scale that allows researchers to make more consistent samples for testing. Though kenaf is highlighted, these processing methods may be applied to any natural fiber. The raw fibers are processed into kenaf chopped strand mats (KCSM) by adapting basic paper-making techniques. KCSM exhibit paper-like qualities and mechanical properties and provide a material of uniform thickness for use in composite parts. Also presented are a basic understanding of natural fiber constituents and effects of mechanical and chemo-mechanical treatments on those constituents. To test KCSM, samples are made for the ASTM D3039 tensile testing and for testing in a dynamic material analyzer (DMA). Both mechanically and chemo-mechanically processed samples are made for the purpose of comparison. Also, I-beam bridges are built with KCSM to demonstrate
how KCSM may be used to create a structure. This is spurred on by the annual SAMPE bridge competition that includes special categories for natural fiber beams. The lay-up procedure is shown in detail to provide a framework that future competitors may use to build quality I-beams for this competition.

The properties obtained by using the KCSM are competitive with other reported properties for kenaf-based composites. A kenaf I-beam demonstrates a strength-to-weight ratio that is 65% of a fiberglass I-beam built to the same dimensions. Trade-offs of using KCSM are the random 2D-fiber orientation and brittle failure, which are not usually desirable in composite components. The chemically treated samples indicate a higher degree of crystallinity but demonstrate inferior mechanical properties when compared to the untreated samples.
Public Abstract

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Every year in the United States, 10 to 11 million vehicles reach the end of their life cycle. Nearly all of these go to a recycling facility. After the recycling process is complete an average of 25% of each vehicle, by weight, goes to waste. This demonstrates the need to use natural fibers in parts that could be recycled, like parts that can be made from kenaf. There are many reasons to consider using natural/bio-fiber composites (bio-composites). These reasons include human health and environmental factors as well as advantages that natural fibers have over fiberglass, such as: lower costs and densities, biodegradability, reduced energy consumption to produce, and a bonus advantage of sequestering carbon dioxide during its growth phase. Through better processing techniques and chemical treatments, the specific mechanical properties of natural fibers are nearing those of fiberglass, making bio-composites a viable substitute in structural applications.

This research investigates methods to process raw kenaf (hibiscus cannabinus) on a scale that allows researchers to make consistent samples for testing while also providing a means by which a complex structure can be built. The process includes a kenaf chopped strand mat (KCSM) that is formed using paper-making techniques. The samples created and tested with the KCSM offer competitive mechanical properties when compared to other
kenaf-based composites. This research also demonstrates that KCSM is a product that can be easily used to make composite components. I-beam bridges are built and tested in a 3-point bend test. This is spurred on by the annual SAMPE bridge competition that includes special categories for natural fiber beams. The lay-up procedure is shown in detail to provide a framework that future competitors may use to build quality I-beams for this competition.
To my wife Melissa and to my boys: Bryce, Cole, and Derek.

&

To the memory of my Mom, she always believed in me.
Acknowledgments

My sincerest appreciation goes to my wife, Melissa, and my boys: Bryce, Cole, and Derek. They share any credit due as they have waited on me patiently and rarely made me feel guilty about being gone all the time. Their love and cheers led me back to school and kept me going every step of the way. I always dreamed of being an engineer, but I would not be and could not be without them. I am grateful for my extended family that also offered their encouragement. I sincerely thank my Heavenly Father for blessing me with the abilities to complete this journey. I have been very blessed from above!

I am grateful to have had wonderful professors at Utah State University and especially Dr. Thomas Fronk who has given his time and allotted me the opportunity to help research kenaf fibers. I am also grateful to Dr. Steve Folkman and Dr. Barton Smith for being on my supervisory committee. I thank my father, Harry Heil, professor emeritus of Western State Colorado University, who was willing to help in processing the fibers, allowing me the use of his personal equipment, while offering tips and good information about paper-making. I acknowledge the support from WSCU’s art department for the use of their Hollander, which played a vital role in this research. I also acknowledge the support from the Microscopy Core Facility at Utah State University for the SEM work.

I thank my classmates and friends who helped me learn challenging material and offered moral support throughout my education. I owe a debt of gratitude to fellow researchers Thomas Loveless who helped with the DMA equipment and Manjunath Prasad for helping with the tensile tester. And last but not least, I thank Karen Zobell, the Administrative Staff Assistant, and Christine Spall, the Graduate Academic Advisor, for making everything run smoothly.

Joshua W. Heil
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<td>AFP</td>
<td>automated fiber placement</td>
</tr>
<tr>
<td>ASTM</td>
<td>American Society for Testing and Materials</td>
</tr>
<tr>
<td>CAGR</td>
<td>compound annual growth rate</td>
</tr>
<tr>
<td>CSM</td>
<td>chopped strand mats</td>
</tr>
<tr>
<td>DMA</td>
<td>dynamic material analyzer</td>
</tr>
<tr>
<td>DRE</td>
<td>data reduction equation</td>
</tr>
<tr>
<td>KCSM</td>
<td>kenaf chopped strand mats</td>
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<tr>
<td>MDF</td>
<td>medium density fiber board</td>
</tr>
<tr>
<td>RBA</td>
<td>relative bond area</td>
</tr>
<tr>
<td>SAMPE</td>
<td>Society for the Advancement of Material and Process Engineering</td>
</tr>
<tr>
<td>SEM</td>
<td>scanning electron microscope</td>
</tr>
<tr>
<td>USU</td>
<td>Utah State University</td>
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<tr>
<td>WSCU</td>
<td>Western State Colorado University</td>
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Chapter 1

Introduction

The global composite industry is experiencing steady growth. A management consulting and market research firm, Lucintel, has predicted that this market will witness a compound annual growth rate (CAGR)\(^1\) of 4.3% from 2015 to 2020 [4]. The U.S. composite materials market grew by 6.3% to $8.2 billion in 2014 and the dominating composite was glass fiber in a polyester resin [5]. Lucintel also predicts the bio-composite industry to grow at a CAGR of 11.2% from 2014 to 2019 [6]. Regardless of the driving forces behind this growth, research on natural fibers will continue to be an area of interest.

There are many reasons to consider using natural/bio-fiber composites (bio-composites), but the main driver of the natural fiber market is currently the automotive industry. In the United States alone, 10 to 11 million vehicles reach the end of their life cycle. Nearly all of these go to a recycling facility. After the recycling process is complete an average 25% of each vehicle, by weight, goes to waste [7]. This demonstrates the need to use natural fibers in parts that could be recycled, like parts that can be made from kenaf. Kenaf fibers are the focus of this research. They have been and are being used in passenger vehicles by Toyota, Ford, and BMW, just to name a few. Of the vehicles using kenaf, the most notable is the BMW i3. The BMW i3 has been designed so that 95% of the car is recyclable at the end of its life cycle. Its instrument panel surround and door trims are made of kenaf [8].

Besides the automotive industry, there are many other reasons to promote the use of bio-composites. These reasons include human health and environmental reasons. Alternatives such as fiberglass cause irritations to skin, eyes, and upper respiratory tract while both glass and carbon fibers do not degrade when released into the environment.

---

\(^1\)The CAGR is calculated by taking the \(n^{th}\) root of the total percentage growth rate, where \(n\) is the number of years in the period being considered.
Some of the advantages that natural fibers have over fiberglass are: lower costs and densities, biodegradability, reduced energy consumption to produce, and a bonus advantage of sequestering carbon dioxide during its growth phase. Through better processing techniques and chemical treatments the specific mechanical properties of natural fibers are nearing those of fiberglass making bio-composites a viable and reasonable substitute in structural applications [7].

1.1 The Motivation

To properly engineer bio-composite products, the physical properties of the fibers as well as fiber/matrix interactions must be known. This task is not simple. Each type of natural fiber differs from other types, not to mention that each fiber is a composite in and of itself. The problem is that many of the suitable natural fibers are not currently available in a form that is production ready, or rather, in a material that is ready-to-use. As a result, manufacturing test specimens becomes a challenge. Aside from this, differing techniques can have an affect on results. Variations in the way samples are prepared could explain the large variations in the reported data for natural fiber properties, see Table 2.3.

1.2 Manufacturing Kenaf Chopped Strand Mats

One objective of this research is to demonstrate a method for manufacturing kenaf chopped strand mats (KCSM) that are in a ready-to-use form to make composite parts. When researching fiber/matrix interactions, composite test specimens are required. The shapes and sizes of these samples vary from test to test and are difficult to produce. As such, the temptation may arise to test a smaller set of samples than is necessary for obtaining trustworthy results. This paper details a simple way to make kenaf-based composite samples of any shape and size. Test samples are made for both the dynamic mechanical analyzer (DMA), from which the storage modulus is obtained and tensile specimens for ASTM D3039 testing, from which the ultimate strength, Young’s modulus, and the secant modulus are obtained. Also presented, are a basic understanding of natural fiber constituents and effects of mechanical and chemo-mechanical processing on those constituents.
1.3 Manufacturing a Kenaf-Fiber Structure

To complement the research completed in the first objective, three small I-beams are built and tested using either KCSM or fiberglass. Two of the three I-beams are made to the same dimensions: one using the KCSM and the other using a fiberglass mat. These two I-beams are tested in a 3-point bend test for comparison. This objective is spurred on by an annual competition held by the Society for the Advancement of Material and Process Engineering (SAMPE), an international professional member society that provides information on new materials and processing technology. This research may aid future students who wish to compete in one of the natural fiber categories. Although manufacturing techniques presented here are specific to I-beams made using the KCSM, they can be applied to any category of the competition and some techniques may be useful for any composite related project.
Chapter 2

Kenaf

Hibiscus cannabinus is a plant in the Malvaceae family which has the common name kenaf. Kenaf is a warm-season annual fiber crop that is related to cotton, okra, and hibiscus. Though it is probably native to east-central Africa it is grown in large quantities in the United States of America, India, Indonesia, Bangladesh, Malaysia, South Africa, Thailand, Vietnam, and numerous parts of Africa. It has dozens of uses which include, but are not limited to: general paper uses like newsprint and cardboards, lining for roofs in felt paper, hardboard panels, rigid molded products, furniture, insulation panels, traditional cordage uses, filtering products, absorbing agents, animal litters, and fillers. It is also used to control terrain erosion (hydro seeding), aid in burning biomass, produce ethyl alcohol using ligno-cellulose, and create various cellulose products [9].

One of the reasons kenaf is a suitable material for use in the bio-composite industry is because of its abundance and short growth cycle. It can be grown in tropical and sub-tropical areas. In sub-tropical areas the kenaf may be planted after danger of frost has passed. If the kenaf is to be harvested as livestock feed it should be cut 75 to 100 days after planting for optimum protein production. Around this same time, the fibers begin to build up in the stem and the leaf-to-stem ratio changes while the protein levels start to drop. The plant reaches maturity in about 150 days and can be 8 to 20 feet tall. The kenaf fibers used for this research are from Malaysia and were obtained from Bast Fiber LLC. The fibers arrived untreated and are approximately 2.5 to 3 inches in length [10].

2.1 Constituents of Kenaf

To have a better understanding of what kenaf is capable of, each of its components are briefly examined. Many natural fibers have these constituents in common, but in varying
It is also important to note that these amounts can vary by the batch. The percentages of these constituents are presented in Table 2.1. As can be seen, there is a fair spread in the data provided.

<table>
<thead>
<tr>
<th>Cellulose</th>
<th>Hemicellulose</th>
<th>Lignin</th>
<th>Pectin</th>
<th>Ash</th>
<th>Source</th>
</tr>
</thead>
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<tr>
<td>60.8</td>
<td>20.3</td>
<td>11.0</td>
<td>3.0</td>
<td>4.7</td>
<td>[11]</td>
</tr>
<tr>
<td>63.5 ±0.5</td>
<td>17.6 ±1.4</td>
<td>12.7 ±1.5</td>
<td>4.0 ±1.0</td>
<td>2.2 ±0.8</td>
<td>[12]</td>
</tr>
<tr>
<td>45.0-57.0</td>
<td>21.5</td>
<td>8.0-13.0</td>
<td>3.0-5.0</td>
<td>-</td>
<td>[13]</td>
</tr>
<tr>
<td>69.2</td>
<td>27.2</td>
<td>2.8</td>
<td>-</td>
<td>0.8</td>
<td>[13]</td>
</tr>
</tbody>
</table>

**Cellulose**

Cellulose is an organic compound known as a polysaccharide. It is the major chemical and structural component of the primary cell wall of green plants and is the most abundant organic polymer on Earth. Cellulose is mainly used to produce paper and paperboard. It was also used to create celluloid, the first thermoplastic polymer, by the Hyatt Manufacturing Company in 1870. In the 1890s it was first used to make artificial silk, commonly known as rayon. In 1912, it was used to create cellophane. Cellulose may also be chemically synthesized without the use of any biologically derived enzymes [14].

Cellulose is hydrophilic, chiral, and degradable. It has a specific gravity of 1.45 [15]. Another one of the characteristic properties of cellulose is a broad chemical variability which is a result of high donor reactivity of hydroxyl groups. This helps to stiffen the chain and forms strong intra- and inter-molecular hydrogen bonds. The final result is a set of linear cellulose chains (Figure 2.1) with hydroxyl groups in a partially crystalline and partially amorphous fiber structure as seen in Figure 2.2.

Various cellulose containing fibers have different degrees of crystallinity. The degree of crystallinity refers to the structural order in a solid. It influences hardness, density, transparency, and diffusion. Chemo-mechanical treatments may be applied to natural fibers to increase the degree of crystallinity. One source states that raw kenaf has a degree of
Fig. 2.1: A linear cellulose chain is formed by repeating this molecule.

Fig. 2.2: Crystalline and amorphous portions of the cellulose macrofibril (representation). Crystallinity of 48.2% and can be increased by chemo-mechanical methods to 81.4% [12]. This procedure is detailed in Section 3.2.

**Hemicellulose**

Like cellulose, hemicellulose is also a polysaccharide but it has a completely amorphous structure. Together with lignin, these two form a matrix surrounding the cellulose fibrils. Compared to cellulose it has little strength and is easily hydrolyzed (chemically broken down by water). It consists of short chains of 200-3,000 mixed sugar units and is a branched polymer (Figure 2.3). Cellulose has 7,000-15,000 pure glucose units and is an unbranched polymer.

Fig. 2.3: An example of a xylan-type hemicellulose. Note the branches. There are several different types of hemicellulose.
polymer [1]. For further comparison of cellulose to hemicellulose refer to Table 2.2. Most chemical treatments aid in the removal of hemicellulose.

Table 2.2: Cellulose vs. Hemicellulose [1] and [2].

<table>
<thead>
<tr>
<th></th>
<th>Cellulose</th>
<th>Hemicellulose</th>
</tr>
</thead>
<tbody>
<tr>
<td>Monomer</td>
<td>Pure glucose</td>
<td>Mixed Sugars</td>
</tr>
<tr>
<td>Polymer chain length</td>
<td>Long</td>
<td>Short</td>
</tr>
<tr>
<td>Polymer topology</td>
<td>Linear</td>
<td>Branched</td>
</tr>
<tr>
<td>Polymer morphology</td>
<td>Crystalline &amp; amorphous</td>
<td>Amorphous</td>
</tr>
<tr>
<td>Solubility</td>
<td>Low</td>
<td>High</td>
</tr>
<tr>
<td>Reactivity</td>
<td>Less reactive</td>
<td>More reactive</td>
</tr>
<tr>
<td>Hydrolysis</td>
<td>Partial</td>
<td>Readily Susceptible</td>
</tr>
<tr>
<td>Hydrophilic</td>
<td>Less</td>
<td>More</td>
</tr>
</tbody>
</table>

Lignin

Lignin is a complex polymer (a phenolic compound) of aromatic alcohols known as monolignols. It is normally found in the secondary cell walls of plants as is the case for kenaf fibers. Lignin is hydrophobic and is insoluble in water. This characteristic helps to conduct water to the remainder of the cell wall, akin to our vascular systems. Lignin is one of the most slowly decomposing components in dead vegetation. Despite its insolvility in water it is soluble in weak alkaline solutions such as sodium hydroxide, caustic soda, or supermarket lye. These are all commonly used to separate lignin from cellulose. Other alkalis are given in order from the most to the least powerful for dissolving lignin: trisodium phosphate (cleaner for walls and woodwork known as TSP), soda ash/sodium carbonate, or potassium carbonate (made by mixing wood ashes with water) [16].

Lignin is used as a water reducer in concrete admixtures, an additive in oil drilling, road dust control, as an animal feed additive, etc. [17]. For the purposes of bio-composites, lignin is the enemy because it rejects water and resists bonding. This may seem counterintuitive because moisture absorption can lead to failure in composite components. However, water absorption is a must for making KCSM, the matrix serves to encapsulate the fibers to
help protect them from moisture, and a better bond between the fiber and matrix can be achieved with the lignin removed.

**Pectin**

Pectin is a structural heteropolysaccharide found in the primary cell walls of plants. Etymologically pectin is derived from the Greek word pektikos which means congealed or curdled. In the plant it functions as a means for the plant to expand or grow and binds cells together. It consists of a long chain of pectic acid and pectinic acid molecules, of which both are sugars (polysaccharides). It is highly hydrophilic in nature [18]. Its solubility is dependent on temperature and composition. We are all familiar with pectin as it is a natural part of the human diet. It is found in the fruits and vegetables we eat, but it is also a common food additive acting as a gelling agent and/or a food stabilizer. It is what gives jelly, the jelly-like consistency. Pectin also keeps our gastrointestinal tracts in check which is why it is a key ingredient in medications for both constipation and diarrhea. In spite of these worthy uses, pectin is another component of the natural fiber that should be taken out for bio-composite uses. In general, pectin may be removed by increasing temperature, as this increases the solubility of pectin [19].

### 2.2 Reported Properties of Kenaf

As stated before, it is imperative to know the mechanical properties of a material when designing a component from that material. Natural fibers have varying crystalline and amorphous components that complicate matters at the level of the fiber. There are methods to improve the fiber at the molecular level for various purposes, like increased strength or improved wettability. Besides the factors unique to natural fibers, there still remains the challenges that all composite parts face like orthotropy, fiber volume fraction, etc.

There are some tests that isolate the fiber itself, like a single fiber tested in tension, while other tests attempt to incorporate interactions with the matrix like the single-fibre pull-out test. Table 2.3 shows some of the published mechanical properties of kenaf fibers
and kenaf-based composites.

Table 2.3: Sample of published mechanical properties for kenaf and kenaf-based composites.

<table>
<thead>
<tr>
<th>Ultimate Strength MPa</th>
<th>Young’s Modulus GPa</th>
<th>Storage Modulus GPa</th>
<th>Test Method</th>
<th>Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>930</td>
<td>53</td>
<td>-</td>
<td>-</td>
<td>[11]</td>
</tr>
<tr>
<td>275-450</td>
<td>-</td>
<td>-</td>
<td>D3039</td>
<td>[20]</td>
</tr>
<tr>
<td>101.3</td>
<td>5.5</td>
<td>-</td>
<td>-</td>
<td>[21]</td>
</tr>
<tr>
<td>65</td>
<td>8.3</td>
<td>7.3</td>
<td>-</td>
<td>[13]</td>
</tr>
<tr>
<td>350-600</td>
<td>40</td>
<td>9-10</td>
<td>D3039/DMA</td>
<td>[22]</td>
</tr>
<tr>
<td>-</td>
<td>-</td>
<td>3-7</td>
<td>DMA</td>
<td>[23]</td>
</tr>
</tbody>
</table>
Chapter 3
Paper and Chopped Strand Mats

The objective of this research is to provide a process for making kenaf-fiber test specimens and structures. In general, composite materials can be difficult to work with due to the preparation of the tooling, surface treatments to the tooling, application of resin, and fiber placement. Besides that, composite lay-ups are always messy! There are many different methods to make composite parts:

- Pre-pregnated lay-ups
- Wet lay-ups
- Spray ups (spray dry fibers onto resin then spray resin, repeat)
- Resin transfer molding and same quality resin transfer molding (RTM and SQRTM)
- Filament winding
- Pultrusion
- Automated fiber placement (AFP)

Some of these methods require vacuum pumps and/or ovens/autoclaves. At the very least, all of these methods require that the composite fiber be in a fabric, mat, or tow. Currently, none of the these are options for the kenaf fibers. In fact, making flat plates with a consistent fiber/matrix volume fraction for sample testing at Utah State University (USU) has proven to be a challenge. Even if the methods employed for making flat plates were adequate, they would be difficult to mimic on a more complex structure. The solution is to first make the fibers into a fabric, mat, or tow which can then be used in some of the aforementioned methods.
Chopped strand mats are a feasible method for the kenaf fibers and the mats prepared for this research exhibit the following characteristics:

- Readily absorbs resin (quickly and thoroughly wets out)
- Cuts as easy as paper without special scissors or cutting equipment
- Edges of the material do not fray
- Drapability is similar to fiberglass mat
- Size of the mat is small (12” x 14”)

3.1 Paper Physics

Paper is made by dewatering a suspension of fibers. There are statistical mechanics and growth models that help to understand how paper is formed through the process of aggregation, sedimentation, and clustering [24]. Regardless of the model used, the fibers rest in random positions and orientations with curls, kinks and undulations forming a planar network structure. Besides the fiber network, the other main ingredient, or lack thereof, is the pore space formed by and in between the fibers and fibrils. These two “ingredients” of paper are reviewed in regards to processing paper mats.

3.1.1 The Inter-Fiber Network

In a regular sheet of paper it is obvious that the fibers are somehow bonded to the other fibers and so on creating a fiber network. If it were not so, the alternative would be a mess of loose fibers. In fact, the fibers are connected to each other at the molecular level and the fraction of the surface area that takes part in bonding one fiber to the others is called the relative bond area (RBA). In a pure pulp sheet of paper there are between 10-40 inter-fiber bonds per fiber. This is referred to as the coordination number. The higher the coordination number, the more the in-plane mechanical properties resemble the axial mechanical properties of a single fiber [24]. To increase the coordination number, pulp and hand-chopped fibers are used to make the KCSM.
The inter-fibre bonds form due to molecular contact between adjacent fibers through hydrogen bonds. In the process of making paper, water breaks hydrogen bonds and allows the fibers to slide past one another. This is why wet paper has lower strength and stiffness than dry paper. When the paper is formed and dried the hydrogen bonds reform. Since this is the case of the KCSM, the stress is not just transferred between the fiber and the matrix as is assumed for normal composite analysis, but some stress is transferred from one fiber directly to another as well as through the matrix. Furthermore, there are internal stresses in the fiber network caused by anisotropic shrinking of the fibers. One researcher states that cellulose fibrils expand from 20-40\% dilitationally with a mere 1\% longitudinal expansion [24].

The mechanical properties of paper are dependent on fiber properties. The elastic modulus of any paper is less than that of the fiber used to make the paper. This is due to the fact that not all of the randomly oriented fibers will help carry the load and the pore space between the fibers cannot help to carry the load. Paper, by itself, is considered a viscoelastic-plastic material. The amorphous components control the viscoelastic properties of the paper while plastic deformation occurs when the fibers slide relative to one another.

3.1.2 The Pore Space

The typical distance between adjacent fibers is equal to the fiber’s width. The pore size can be partly dependent on the stiffness of the fiber. One reason fibers undergo mechanical and/or chemo-mechanical pulping is to fibrillate the fiber and soften it to decrease the amount of pore space. It has also been noted that fragmented fibers (fibrils) that are highly hydrophilic can fill the voids between fiber surfaces, not only by taking up the space themselves, but also by pulling adjacent fibers closer to each other when the fibrils contract as they dry. Recall that wet cellulose fibrils expand 20-40\% in the transverse direction. This means they have that potential to pull adjacent fibers closer upon drying. Conversely, it has been noted that hydrophobic constituents like lignin and other trace minerals act in the opposite manner, creating more pore space in dry paper [24].
3.2 Chemical Treatment of Kenaf Fibers

From Chapter 2 and the reasons discussed in the sections on paper physics as well as other research has shown, chemical treatments are necessary to improve the mechanical properties of natural fibers. Most methods only focus on the use of sodium hydroxide to remove the lignin and hemicellulose. These treatments are quite successful but they can leave large percentages of hemicellulose and do nothing to strengthen the remaining cellulose chains. One method that seems worthy of reproducing is a sodium hydroxide-anthraquinone (NaOH-AQ) pulping treatment followed by a 3-stage bleaching procedure. This has been shown to reduce the rate of degradation and causes less damage to the cellulose chains (the AQ helps to stabilize the cellulose) [12]. The bleaching procedure further removes lignin and hemicellulose.

The following procedure for chemically pulping the kenaf was carried out on 400 grams of Hollander processed pulp and a separate 400 grams of 1/4 - 1/2" hand-chopped kenaf fibers. Table 3.1 shows the conditions of the NaOH-AQ pulping process. For this process, 420 grams of NaOH (CAS NO: 1310-73-2), 2.8 grams of AQ (CAS NO: 84-65-1), and 2377.2 grams of distilled water were combined and added to each batch of pulp and fibers in a well ventilated area. The literature suggests using a digester to elevate the temperature. The mixture is brought to 160 °C over a 60 minute time frame and held at that temperature for 45 minutes [12].

<table>
<thead>
<tr>
<th>Cooking Process</th>
<th>NaOH-AQ</th>
</tr>
</thead>
<tbody>
<tr>
<td>NaOH</td>
<td>15 %</td>
</tr>
<tr>
<td>AQ (anthraquinone)</td>
<td>0.1 %</td>
</tr>
<tr>
<td>Liquid-to-fiber ratio</td>
<td>7:1</td>
</tr>
<tr>
<td>Maximum temperature</td>
<td>160 °C</td>
</tr>
<tr>
<td>Time to maximum temperature</td>
<td>60 min</td>
</tr>
<tr>
<td>Time at maximum temperature</td>
<td>45 min</td>
</tr>
</tbody>
</table>

1This is a unique numerical identifier assigned by Chemical Abstracts Service to every chemical substance. It offers a reliable way to reproduce a procedure.
After this pulping process the pulp and the fibers are rinsed thoroughly by hand for 2 hours. In order to remove the lignin and hemicellulose that the NaOH-AQ treatment leaves behind a three stage bleaching process was followed as the literature recommends following the conditions outlined in Table 3.2. For treatment $D_1$, 70 grams of $O_2ClNa$ (CAS NO: 7758-19-2), 84 grams of Acetic Acid (CAS NO: 64-19-7), and 2646 grams of distilled water are combined and added to each batch of pulp and fibers in a well ventilated area. It is then heated to 70 °C for 180 minutes. The fibers are again hand rinsed for 1 hour before proceeding to the next stage. The $Ep$ stage removes any residual chlorite from the pulp and fibers and makes the removal of lignin more efficient. For the $Ep$ treatment, 42 grams of NaOH (CAS NO: 1310-73-2), 93.5 grams of $H_2O_2$ (CAS NO: 7722-84-1), and 2664.5 grams of distilled water are combined and added to each batch of pulp and fibers in a well ventilated area. Then it is heated to 70 °C for 90 minutes. Once again, the fibers are hand rinsed for 2 hours before proceeding to the final step. To further remove lignin and hemicellulose the $D_2$ treatment is done as follows: 43.75 grams of $O_2ClNa$ (CAS NO: 7758-19-2), 84 grams of Acetic Acid (CAS NO: 64-19-7), and 2672.25 grams of distilled water are combined and added to each batch of pulp and fibers in a well ventilated area and heated to 70 °C for 90 minutes. A final hand rinse of the pulp and fibers is done for 2 hours [12].

<table>
<thead>
<tr>
<th>Stage</th>
<th>$D_1$</th>
<th>$Ep$</th>
<th>$D_2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical charge</td>
<td>Sodium chlorite (2%)</td>
<td>NaOH (1.5%)</td>
<td>Sodium chlorite (1.25%)</td>
</tr>
<tr>
<td>Chemical charge</td>
<td>Acetic acid (3%)</td>
<td>$H_2O_2$ (1%)</td>
<td>Acetic acid (3%)</td>
</tr>
<tr>
<td>Liquid-to-pulp ratio</td>
<td>7:1</td>
<td>7:1</td>
<td>7:1</td>
</tr>
<tr>
<td>Temperature</td>
<td>70 °C</td>
<td>70 °C</td>
<td>70 °C</td>
</tr>
<tr>
<td>Time at temperature</td>
<td>180 min</td>
<td>90 min</td>
<td>90 min</td>
</tr>
</tbody>
</table>

The objective of this process is to attempt a chemical treatment that had not yet been attempted at USU and to see how chemically treated fibers faired in the paper-making process compared to untreated fibers that are only mechanically pulped.
3.3 Paper-Making Process

The process of making the KCSM for this research is adapted from a common paper-making process. Making paper is neither a new process nor a difficult one. Jules Heller, the author of *Papermaking* [25], wrote:

A great American papermaker is said to have said that “any damn fool can make paper.” I take his word for it, because here I am having made some myself.

The author of this paper echoes the sentiment and uses it as an argument that this process may be easily followed by anybody. Just because anybody can make paper does not imply that it is made well. Like many things that are simple, there is always room for improvement and experience cannot be taught but rather gained. So, do not give up if you attempt to make paper and are not satisfied with the results.

3.3.1 The Raw Fiber

There are several suppliers of kenaf fibers. Although kenaf is grown within the United States, most dealers, domestic and foreign, will have the fibers shipped from outside of the United States. It is possible to specify the fiber length (1/2" is the shortest most suppliers can provide), whether the fiber is cut or uncut, and whether the fibers are carded or not. Typically a finer fiber is obtained by carding before cutting. Some suppliers offer wet treatments that can enhance the performance of the fibers. These treatments include: bleaching, anti-rot, anti-mildew, wetability treatments (for better bonding), hydrophobic treatments, and even treatments to enhance strength and impact resistance. Other factors to consider when ordering the fiber are the purity, strength, fineness, color, and price.

The fibers used in this research arrived from Bast Fibers LLC in a bale consisting of “patches” that are roughly 3 inches long, see Figure 3.1, and are untreated. These patches have the fibers predominantly in the same orientation, though the patch itself may have been folded or twisted while being compressed tightly into the bale.
3.3.2 The Pulp and the Hollander

By definition, paper is a thin sheet of randomly oriented fibers that have undergone a maceration process. This is referred to as the pulping stage which involves hydrolyzing the fibers and beating them. The objective is to rid the fiber of as much hemicellulose as possible and hydrate the cellulose. Hydrating the cellulose is crucial to making good paper for reasons discussed in the section on paper physics.

First, the unaltered fibers are soaked in water for at least 24 hours. Due to the fact that the fibers are capable of holding nearly 10 times their mass in water, it is wise to check on the fibers after filling the container to ensure that all of the fibers are being soaked equally. It is also wise to remove trapped air bubbles so the fibers will not float out of the water. Roughly, 600 grams or 7 liters of fiber were soaked per batch for processing in the Hollander.

The Hollander, Figure 3.2 is a machine used to make paper pulp. This particular machine belongs to Western State Colorado University (WSCU). The first Hollander was developed by the Dutch in the late 1600s, which is how it received its’ name. Though there are many ways to create pulp, the Hollander makes a pulp with longer and more fibrillated fibers than other methods that involve grinding or just cutting. Fibrillated fibers are beaten
Fig. 3.2: A HYDRA®Hollander built by Lee S McDonald of Charlestown MA. so that part of the fiber is broken off and the fibrils are more exposed as seen in Figure 3.3. This creates more surface area and increases the potential for hydrogen bonding.

Fig. 3.3: Scanning electron microscopy (SEM) is used to inspect the fibers processed in the Hollander. Fibrillation is observed.

The Hollander itself consists of an oval-shaped tub with a mid-feather that form a raceway. The mid-feather is the wall in the center of the oval that helps to direct the flow. The roll and bed-plate are usually made from a hard aluminum alloy and have interlocking teeth which may be sharp or blunt. The motor turns the roll which pulls the fibrous
slurry in-between the roll and bed-plate and pushes it down the back-fall. The clearance adjustment gives the user control over the spacing between the roll and bed-plate. A roll hood helps to keep the slurry in the machine while keeping fingers, hair, etc. out of the machine. These components are labeled in Figures 3.4-3.7.

Fig. 3.4: The tub with a mid-feather and the drain.

Fig. 3.5: The roll is lifted up to show detail.

Fig. 3.6: The bed-plate and the back-fall. The slurry moves from right to left.

Before proceeding, the Hollander was used to make the kenaf pulp by Harry Heil, professor emeritus of the fine arts at WSCU. He has over 40 years of experience in making
his own paper and teaching paper-making for use in watercolors and printmaking. Some of his basic guidelines for using the Hollander beater are outlined below.

While the fibers are soaking for 24 hours in water, the Hollander may be prepared. First, the “scratch mark” is located and marked on the clearance adjustment. The scratch mark is the point at which the roll and the bed-plate just barely come into contact with one another. This must be done every time as the beater roller and bed-plate wear down and there are different types of rolls that may be exchanged. Next the “brushing position” is marked on the clearance adjustment. This mark indicates the fully raised position. Depending on the height of the roll, the qualities of the final product may be altered. Next, the tub is filled with approximately 10 gallons of fresh water to which the soaked fibers are added. The motion of the beater roll circulates the water/fiber mixture.

Periodically, the roll must be lowered until the desired consistency of pulp is reached. For the experienced user this is done by feel and by the freeness test. The freeness test is done by taking a small amount of pulp and placing it into a clear jar with clean water, shaking it vigorously, and holding it against the light. There should be no visible fibers or flocks of fibers and the pulp should make the water appear milky as it is free from the other pulped fibers. Some Hollander beaters have a display that indicates the amperes being drawn by the motor which can be used to help the operator know when to lower the roll. For this research, the clearance adjustments were made to keep the motor drawing between
12 and 14 amperes. When this point is reached, the pulp may be collected and transferred into a larger tub for the paper making process or the paper may be made directly from the Hollander if there is sufficient room. For the purpose of this research, the pulp was collected and shipped to USU where it was promptly set out to dry (see Figure 3.8) so that it could be re-hydrated as needed to make paper without rotting or mildewing.

Fig. 3.8: The pulp drying out after being processed in the Hollander.

At the beginning of the process the fibers’ appearance in the roll hood is unchanged from the soaked fibers. A short time after the Hollander has been running, a foam appears. Near the completion of the process the fibers seen through the roll hood are noticeably different and the foam has run over the side of the tub. The foam is an indication of the amount of starch\(^2\) that is being released from the cellulose and the hemicellulose. Figures 3.9-3.10 show the fibers near the start of the process and near the end of the process.

3.3.3 Making Paper and Making Kenaf Chopped Strand Mats

There are several ways to make paper. This section only covers the methods that are used for this research. Paper made by this method is referred to as *laid paper*.

To make the KCSM the pulp that has been processed in the Hollander machine is used as well as hand-chopped fibers. The pulp could be considered a binder for the hand-chopped fibers. Starch is a carbohydrate or polysaccharide that stores energy for the plants.

\(^2\)Starch is a carbohydrate or polysaccharide that stores energy for the plants.
fibers, though it does more than this. The hand-chopped fibers were cut by expanding a small patch of raw kenaf (to cut easier) and using a regular paper-cutter as shown in Figure 3.11. The final length was chosen through experimentation. Fiber lengths ranging from roughly 1/4 to 3” are used (or attempts were made) to make paper. The longer the fiber, the more clumping and variations there are in the thickness of the mat produced. The objective of making KCSM is defeated if the thickness of the mat has too much variation. The longest fiber to produce a mat with a consistent thickness is approximately 1/2”.

Fig. 3.9: (a) Fibers in the roll hood at start, (b) Amount of foam accumulated near the start.

Fig. 3.10: (a) Pulp in the roll hood at the end, (b) Amount of foam accumulated at the end.
Preparing the Pulp/Fiber Slurry

Before making KCSM the pulp and the hand-chopped fibers must be re-hydrated by soaking them separately for 24 hours. They are soaked independently because the pulp is held together in small enough clumps or flocks that they will not be adequately dispersed by the drill mixer in the tub of water. A blender is used to break these small clumps up by adding about 1 part pulp to 8 parts water and blending until the slurry is free as in the freeness test (Figure 3.12). The contents of the blender are emptied into a tub of water and this step is repeated until there is sufficient pulp to mimic the consistency of the freeness test in the tub. Next the hand-chopped fibers are added slowly to the water while a drill with a mixing attachment stirs the contents as shown in Figure 3.13. The two parts are added nearly 50/50 by weight, though it is difficult to measure and certainly hard to maintain throughout the process of making multiple sheets of paper.

The Mold and Deckle

The mold and deckle are the essential tools for making laid paper and examples of these are seen in Figure 3.14. The mold consists of a frame with many airfoil-shaped ribs
Fig. 3.12: (a) Pulp needing to be dispersed, (b) The *freeness test* is passed.

Fig. 3.13: (a) Stirring the pulp and fibers, (b) Mixing paddle traps small amounts of fibers.

that usually span the short dimension of the frame. The airfoil-shaped ribs are oriented so that the leading edge is facing away from the screen. These ribs act as a support for the screen that covers the entire frame. The screen (often referred to as the wire) is typically made of brass wire that is stretched across the longer dimension of the frame and secured to each rib with a finer brass wire. The deckle is a separate frame that fits around the perimeter of the mold and can be pressed neatly against the brass wire. A detailed cross section is shown in Figure 3.15.

The mold and deckle are dipped vertically into the tub of suspended fibers and turned to lay flat on the bottom of the tub, Figure 3.16. While holding the mold and deckle on the
bottom with one hand, the tub is stirred again by the other hand. The mold and deckle are then raised slowly towards the surface so that the suspended fibers and pulp become deposited on the wire. As the sedimentation builds on the screen, any areas with less accumulation allow water to pass through more than areas with more accumulation. This causes more sediment to be deposited in those areas and the end result is a fairly uniform layer of water, pulp, and fiber. Near the surface, the upward motion is slowed until the wire passes through the surface of the water, Figure 3.17. This helps to pack the fibers together and facilitates the removal of the newly formed sheet. The ribs are airfoil shaped with the leading edge pointing down so that they do not create voids in the paper and to lessen the
amount of exertion needed to pull the mold out of the water. The deckle acts as a dam to keep the sediment from washing off the sides. A taller deckle means a potentially thicker sheet can be formed.

![Fig. 3.16: Dipping the mold and deckle.](image1)

![Fig. 3.17: Slowly pulling the mold and deckle out.](image2)

**Kissing Off and Couching**

At this point the sheet may be examined for voids, lumps, or even possibly foreign
materials. If needed, the sheet may be discarded for another try by kissing off the material into the tub, Figure 3.18. This is accomplished by simply touching the inverted mold onto the surface of the water which will need to be stirred again.

![Fig. 3.18: Kissing off a sheet of paper that was not uniform.](image)

When a sheet is acceptable the deckle is removed, Figure 3.19, and the paper is couched onto a felt. To remove the paper while maintaining its form a piece of felt is placed on the couching table that has a convex surface. When the mold is rocked across the felt, the convex surface applies a counterforce along one line at a time while the felt receives the paper. To make multiple sheets, another piece of felt may be placed directly atop the previous sheet of paper. This creates a pile. This process is demonstrated in Figures 3.20-3.21.

![Fig. 3.19: Deckle removed. A uniform sheet of paper.](image)
The Printing Press

Conventionally, laid paper and other hand-made papers are completed by placing a pile of paper and felts into a press and squeezing out as much water as possible and then separating the sheets to dry. This method leaves small surface imperfections from the paper taking on the shape of the felt. In the composite samples made from paper manufactured in this method, the fiber volume fraction reached roughly 30%. A printing press is used to improve the surface finish of the paper to solve this issue.

While the paper is still wet from the previous process, it and its piece of felt are
sandwiched between two pieces of Plexiglas and run through a printing press. This squeezes out much of the excess water as seen in Figure 3.22. After two passes, back and forth, the water is nearly gone from the paper, but it seeps back in along the edges. Because this only smooths one side of the paper, the felt is removed and the paper is run through between the two sheets of Plexiglas. The final product is more uniform and when used to make samples the fiber volume fraction increased to between 45-50%.\footnote{It should be noted that the printing press likely creates different mechanical properties in the direction of the rollers (machine direction) compared to the direction transverse to the rollers. This is analogous to an isotropic metal being cold rolled.}

Fig. 3.22: (a) The paper going through the printing press. Note the amount of water being pushed out and the paper on the back side appears dry (lighter areas are more dry). (b) The water seeps back into the paper around the edges.
Chapter 4
Methods of Testing Kenaf Chopped Strand Mats

The focus of this research is processing kenaf chopped strand mats and for manufacturing test specimens and kenaf-based structures as opposed to attempting to obtain the mechanical properties of kenaf or a kenaf-based composite. However, the process would be incomplete without making samples and testing them. These results are not meant to be authoritative as other researchers have focused on using the same equipment to obtain material properties of kenaf-based composites. The goal is to be within the range of the published properties listed in Table 2.3.

4.1 Dynamic Mechanical Analysis

The DMA measures stiffness and damping of a material through non-destructive means. In a three-point bend test, the DMA applies a sinusoidal deformation to a small specimen of known geometry. The frequency of the input is defined by the user and can be set between 0.01-100 Hz. The sample’s modulus is expressed by an in-phase component and an out of phase component due to phase shift of the sinusoidal force being applied to the sample and the displacement. The in-phase component is the storage modulus and the out of phase component represents the loss modulus. The ratio of loss and storage moduli provides a measure of the energy dissipated, referred to as damping. The storage modulus is a measurement of elastic behavior. For all testing performed for this research a frequency of 1 Hz is used to obtain the storage modulus. While Young’s modulus is similar to the storage modulus, they are not the same. For a more complete understanding of how the DMA functions refer to Mechanical Properties of Kenaf Composites Using Dynamic Mechanical Analysis by Thomas Loveless [23].
The data reduction equation (DRE) used to obtain the storage modulus, $E'$, is

$$E' = \frac{F'}{A'} \left( \frac{l^3}{4wh^3} \right)$$  \hspace{1cm} (4.1)

where $F'$ and $A'$ are the real portions of the force and the displacement amplitude, respectively. The quantity in parenthesis is a geometric factor based on the length, width and height of each specimen.

For the three-point bend test the span is fixed at 40 mm. The sample size must be no greater than 5 mm thick by 12 mm wide. It is also recommended that the sample be at least 48 mm long so that 10% of the sample length passes the support on each side.

### 4.1.1 DMA Sample Preparation: First Trial

Ten samples are prepared from each batch of pulp processed in the Hollander beater. They are prepared by forming a sheet of KCSM thick enough that only one layer is needed to achieve an appropriate sample thickness. The paper was then cut to approximately 50 mm x 11 mm and placed directly onto a pool of resin to wet out as seen in Figure 4.1. When

![Image of DMA samples being prepared](image)

**Fig. 4.1**: The first set of DMA samples being prepared individually. Note how the resin is absorbed.
the material is saturated with resin it is said to be wet out. The resin system is PTM&W
Industries, Inc’s PT2050 A resin and the PT2050 B1 hardener with a mixing ratio of 100
resin to 27 hardener by weight. The samples and a high density rubber mat treated with car
wax as a release agent are pressed between two caul plates\(^1\) in order to make them uniform
in thickness and squeeze out excess resin. After the resin is set the samples are trimmed
to size (excess resin pools around each sample) using a belt sander. Subsequent sanding is
done with 320-grit to 600-grit wet sandpapers. The samples are then post cured at 70 °C
for 8 hours to ensure adequate cross-linking of the polymer.

The rubber mat functioned as planned but created a slightly uneven surface that either
causes some samples to lay unevenly on the supports or the probe to only come into contact
with a fraction of the specimen’s width as opposed to the width used to calculate the
geometric factor. Furthermore, the variations caused by the rubber mat has a negative
impact on determining the geometric factor for each sample. The results from the various
Hollander batches are presented in Table 4.1. The objective of this test is to help determine
the ideal amount of processing in the Hollander.

<table>
<thead>
<tr>
<th>Batch</th>
<th>Time in Hollander</th>
<th>E’ [GPa]</th>
<th>Std Dev [GPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>90 min</td>
<td>4.84</td>
<td>0.44</td>
</tr>
<tr>
<td>F</td>
<td>15 min</td>
<td>4.42</td>
<td>0.84</td>
</tr>
<tr>
<td>D</td>
<td>30 min</td>
<td>4.03</td>
<td>0.60</td>
</tr>
<tr>
<td>E</td>
<td>45 min</td>
<td>3.59</td>
<td>0.42</td>
</tr>
<tr>
<td>C</td>
<td>30 min</td>
<td>3.53</td>
<td>0.26</td>
</tr>
<tr>
<td>B</td>
<td>45 min</td>
<td>3.35</td>
<td>0.34</td>
</tr>
</tbody>
</table>

Batch A produced the best results. It consisted of the finest fibers of all the batches
having been run in the Hollander the longest. This batch is considered “paper quality.”
The test samples made from Batch A have the most consistent thickness which likely plays
a factor in their superior performance. Batch F faired second best having been processed

\(^1\)A caul plate is smooth metal plate that is usually the size and shape of the composite. It is used to
create a normal pressure to hold the fiber and matrix in place, to aid in debulking resin, and provide a
smooth surface to the laminate.
in the Hollander least amount of time. This batch contains fibers that are still up to an inch long, though noticeably damaged and softer than unprocessed fibers.

Combining the principles learned in the literature review on paper physics and the results obtained from the DMA testing it was decided to have more fibers processed in the Hollander beater as done for batch A. This pulp is used in combination with hand-chopped fibers. This takes advantage of the mechanical properties of the unprocessed fiber and provides a way to bind them together. Also, the fibrillation caused in the pulping process by the Hollander decreases the pore space and strengthens the inter-fiber network by increasing the RBA and coordination number (more bonds per fiber).

4.1.2 DMA Sample Preparation: Second Trial

A different method is employed to improve the fiber volume fraction and the geometry of the test samples. Instead of cutting out each sample individually, a large plate is made from which all the samples are cut. Also, in lieu of forming a thick sheet of paper, which is difficult to do with a consistent thickness, 10 thin sheets are laminated together. After oven drying all of the papers to remove moisture absorbed from the air the paper is wet out a layer at a time using a plastic spreader to squeegee away the excess resin. The layers are combined and clamped together between two caul plates made from 1” thick aluminum plates with several clamps as shown in Figure 4.2.

Fig. 4.2: There are many ways to apply pressure, 21 clamps answer the call.
Once the resin system is set up, the laminate is removed and post-cured in an oven for 8 hours at 70 °C to ensure that all of the polymer chains are adequately cross-linked. The plate is ready to be trimmed. The first straight edge is cut using a wet tile saw as shown in Figure 4.3. Subsequent cuts are made using a Proxxon FET table saw with a 3.5” diamond blade to minimize damage to the laminated edge. The edges of each samples are then wet sanded smooth using 400 grit and then 600 grit sandpaper. The specimen are once again dried in the oven as shown in Figure 4.4 for 1 hour at 70 °C.

Fig. 4.3: A wet tile saw cuts composites easily and keeps harmful dust to a minimum.

Fig. 4.4: The samples are drying after the cut edges have been wet sanded. DMA samples are the small samples in the back while the larger samples are for ASTM D3039 tensile testing. Light samples are chemically treated.

Ten samples of both untreated and chemically treated KCSM are tested in the DMA
to obtain the storage modulus, $E'$. The results for each sample appear in Figures 4.5-4.6. To remove the outliers from the sample set Chauvenet’s criterion is used [26]. The average values are reported in Table 4.2.

Fig. 4.5: The storage modulus for chemically treated samples.

Fig. 4.6: The storage modulus for untreated samples.
Table 4.2: Storage Modulus, in GPa, of KCSM after applying Chauvenet’s criterion.

<table>
<thead>
<tr>
<th></th>
<th>E'</th>
<th>Std Dev</th>
<th>Max E'</th>
<th>Min E'</th>
</tr>
</thead>
<tbody>
<tr>
<td>Untreated KCSM</td>
<td>5.56</td>
<td>0.90</td>
<td>7.00</td>
<td>4.26</td>
</tr>
<tr>
<td>Treated KCSM</td>
<td>3.88</td>
<td>0.32</td>
<td>4.30</td>
<td>3.45</td>
</tr>
</tbody>
</table>

4.2 Tensile Testing

The tensile test chosen for testing the KCSM/polymer matrix composite is the ASTM D3039 testing standard. This test is designed to obtain the in-plane tensile properties of a polymer matrix composite material reinforced by high-modulus fibers, usually > 20 GPa. The kenaf fibers do not exhibit such a high modulus. The coupons required for this test are a thin flat strip of material having a constant rectangular cross section. When mounted in the machine, the coupon is loaded in tension while recording the load. The ultimate strength of the material can be determined from the maximum load carried before failure. A strain gage is also mounted axially on the coupon in order to monitor the stress-strain response of the material. Young’s modulus is usually obtained through this test by using the linear region of the stress-strain curve. However, because there is no true linear region due to the viscoelastic-plastic behavior the KCSM demonstrates, the secant modulus is also included. The secant modulus is defined by the ASTM standards to be the ratio of stress (nominal) to corresponding strain at any specified point on the stress-strain curve. It is expressed in force per unit area and reported together with the specified stress or strain.

4.2.1 Sample Preparation

Both untreated and chemically treated samples are made for testing. Tensile coupons for this test are prepared in the same fashion as the samples in the “DMA Sample Preparation: Second Trial” section. The specimen geometry chosen follows the recommendation provided in the ASTM D3039 guidelines for random-discontinuous fibers. This is 250 mm in overall length and 25 mm in the width. As there is no recommendation for a specimen thickness, the same thickness required by the DMA is used. The strain gages are Micro-Measurements brand gages. They are type WK-06-125BT-350 and from lot number
DU-K47FD08. They have the following reported data: 350.0±0.3% resistance in ohms, 2.06±1.0% gage factor, and -1.8±0.2% transverse sensitivity, all at 24°C. The gages are applied to the coupon with M-Bond 200 Adhesive and M-Bond Adhesive 200 Catalyst-C. The instructions for mounting the gages are followed as prescribed in the instructions included with the adhesive and catalyst (one exception is the use of an alignment template as the gage installation tape was not available). These steps are itemized as follows:

- Degrease gaging area with solvent
- Abrade area with 320-grit silicon carbide paper and clean with conditioner
- Apply M-Prep Neutralizer and scrub with cotton-tipped applicator
- Place strain gage on coupon with tweezers and align using alignment template
- Place clear tape onto the coupon above the gage
- While holding the gage in place bring the tape down to pick up the gage
- Apply small amount of M-Bond 200 catalyst to the gage and wait 1 minute
- Apply a drop of adhesive and quickly wipe the tape with gage firmly into place
- Maintain a firm pressure with a thumb for at least 2 minutes

At this point the tape is left in place until testing to protect the gage. Wire leads are soldered to each strain gage. The samples are now ready to be loaded into the machine for testing. Each coupon is loaded into the grips following the same procedure as follows: The coupon is placed between the top and bottom grips until the back side of the coupon is seated against the back wall of the grips. While holding the coupon in place the bottom grip is tightened completely before tightening the top grip. Because all coupons are 250 mm in length, care was taken to position the grips so that each coupon would barely pass

\footnote{Grip tabs were determined to not be necessary for the testing as previous kenaf samples reached ultimate strength around 2000 N, which did not cause the samples to fail near the grips frequently. However, grip tabs should have been included to prevent premature failure in these specimen as a high percentage of them broke at the grips from loads ranging between 6200-8700 N.}
into the grips. This helps to ensure the grips have more area to grip as well as give a more consistent initial length. The head-speed is set at a constant displacement of 5 mm/min. All other portions of the test, including calibration and grip alignment were set up by fellow researcher Manjunath Prasad and a more extensive look into testing kenaf samples can be found in his work, “Effect of NaOH Concentration on Kenaf Short Fiber Composite Young’s Modulus and Poisson’s Ratio” [3].

4.2.2 Results of Tensile Testing

The properties sought after are the ultimate strength in the test direction ($F_{tu}$), Young’s Modulus ($E$), and the secant modulus. From the LABView VI that interfaces with the tensile machine a large output file is generated for each test. A Matlab code imports roughly 2 million data points per sample for each the force and the strain, which is $\frac{\Delta L}{L_0}$. Another code is used to locate the maximum force ($P_{max}$) for computing the ultimate strength, the strain at 3000 N, and the force at 1,000 and 6,000 $\mu\epsilon$, which is used to calculate the secant modulus.

The ultimate strength in the test direction is calculated by Equation 4.2 as,

$$F_{tu} = \frac{P_{max}}{wh}$$

where $P_{max}$ is the load at failure and the product $wh$ is the cross-sectional area of the coupon.

Figure 4.7 shows an example of the behavior of the material. The stress-strain curve is nearly linear. The secant modulus offers a more conservative value than Young’s modulus.

Tables 4.3-4.4 show the results obtained for each of the coupons tested. Columns are included that indicate the maximum load and if the coupon failed in the center or near the grips.
Fig. 4.7: This is a typical shape for the stress-strain curve for the untreated coupon.

Table 4.3: Tensile properties obtained from the chemically treated samples.

<table>
<thead>
<tr>
<th>Sample No</th>
<th>$p_{max}$ [N]</th>
<th>$F_{ut}$ [Mpa]</th>
<th>$E$ [GPa] at 3 kN</th>
<th>Secant Mod. [GPa]</th>
<th>Fail in center?</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>6483</td>
<td>74.71</td>
<td>6.17</td>
<td>6.04</td>
<td>Yes</td>
</tr>
<tr>
<td>2</td>
<td>7262</td>
<td>83.23</td>
<td>6.99</td>
<td>6.82</td>
<td>Yes</td>
</tr>
<tr>
<td>3</td>
<td>6159</td>
<td>70.34</td>
<td>0.00</td>
<td>0.00</td>
<td>No</td>
</tr>
<tr>
<td>4</td>
<td>6405</td>
<td>75.94</td>
<td>0.00</td>
<td>0.00</td>
<td>No</td>
</tr>
<tr>
<td>5</td>
<td>7349</td>
<td>83.52</td>
<td>7.18</td>
<td>6.92</td>
<td>Yes</td>
</tr>
<tr>
<td>6</td>
<td>6223</td>
<td>67.13</td>
<td>6.62</td>
<td>6.45</td>
<td>No</td>
</tr>
<tr>
<td>7</td>
<td>7796</td>
<td>84.06</td>
<td>6.49</td>
<td>6.30</td>
<td>No</td>
</tr>
<tr>
<td>8</td>
<td>7513</td>
<td>87.88</td>
<td>6.93</td>
<td>6.78</td>
<td>Yes</td>
</tr>
<tr>
<td>9</td>
<td>6817</td>
<td>79.48</td>
<td>6.80</td>
<td>6.58</td>
<td>Yes</td>
</tr>
<tr>
<td>10</td>
<td>6701</td>
<td>78.27</td>
<td>6.94</td>
<td>6.67</td>
<td>Yes</td>
</tr>
</tbody>
</table>

Averages 6871 78.46 5.41 5.26 40% Failed
Std Dev 575 6.53 2.87 2.78 Near Grips
Table 4.4: Tensile properties obtained from the untreated samples.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>$p_{max}$ [N]</th>
<th>$F_{ut}$ [Mpa]</th>
<th>$E$ [GPa] at 3 kN</th>
<th>Secant Mod. [GPa]</th>
<th>Fail in center?</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>8253</td>
<td>106.43</td>
<td>8.55</td>
<td>7.98</td>
<td>No</td>
</tr>
<tr>
<td>2</td>
<td>8668</td>
<td>109.25</td>
<td>8.48</td>
<td>8.04</td>
<td>No</td>
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<tr>
<td>3</td>
<td>8008</td>
<td>97.97</td>
<td>7.55</td>
<td>7.29</td>
<td>Yes</td>
</tr>
<tr>
<td>4</td>
<td>7436</td>
<td>90.40</td>
<td>7.13</td>
<td>6.88</td>
<td>Yes</td>
</tr>
<tr>
<td>5</td>
<td>8355</td>
<td>105.67</td>
<td>7.90</td>
<td>7.64</td>
<td>No</td>
</tr>
<tr>
<td>6</td>
<td>6538</td>
<td>84.29</td>
<td>7.69</td>
<td>7.41</td>
<td>Yes</td>
</tr>
<tr>
<td>7</td>
<td>7751</td>
<td>95.64</td>
<td>8.41</td>
<td>8.00</td>
<td>No</td>
</tr>
<tr>
<td>8</td>
<td>7113</td>
<td>87.49</td>
<td>7.21</td>
<td>7.06</td>
<td>No</td>
</tr>
<tr>
<td>9</td>
<td>6479</td>
<td>83.92</td>
<td>8.22</td>
<td>7.96</td>
<td>No</td>
</tr>
<tr>
<td>10</td>
<td>7174</td>
<td>85.83</td>
<td>7.39</td>
<td>7.11</td>
<td>No</td>
</tr>
<tr>
<td>Average</td>
<td>7577</td>
<td><strong>94.69</strong></td>
<td><strong>7.85</strong></td>
<td><strong>7.54</strong></td>
<td>70% Failed</td>
</tr>
<tr>
<td>Std Dev</td>
<td>757</td>
<td>9.75</td>
<td>0.54</td>
<td>0.44</td>
<td>Near Grips</td>
</tr>
</tbody>
</table>

4.3 Uncertainty Analysis and Discussion

4.3.1 Uncertainty Analysis

The Taylor Series Method (TSM) is used to consider how the systematic and precision uncertainties in the measured variables propagate through the data reduction equation (Equation 4.1) used to obtain the storage modulus. The goal of any uncertainty analysis is to find the range ($X_{best} \pm u_X$) within which the true value falls, where $X_{best}$ is not the true value, but the best value we have for it and $u_X$ is the standard uncertainty [26].

For TSM we say that if an experimental result is a function of $J$ variables,

$$ r = r (X_1, X_2, \cdots, X_J) \quad (4.3) $$

the general uncertainty can be expressed by

$$ U_r = \left[ \left( \frac{\partial r}{\partial X_1} U_{X_1} \right)^2 + \left( \frac{\partial r}{\partial X_2} U_{X_2} \right)^2 \right]^{\frac{1}{2}} \cdots \left[ \left( \frac{\partial r}{\partial X_J} U_{X_J} \right)^2 \right]^{\frac{1}{2}} \quad (4.4) $$

where $U_{X_J}$ is determined by the sensitivity of the measurement taken or is the precision uncertainty present in each measurement. Looking at Equation 4.1, there is uncertainty
from the calipers used to measure \( l, w, \) and \( h \) for length, width, and thickness, respectively. There is also uncertainty in the force and displacement amplitude, \( F' \) and \( A' \) respectively, which arise from the DMA’s load cell and the DMA’s sensor that measures the displacement amplitude. The precision uncertainty is dependent upon the standard deviation, \( s_x \), and the number of samples considered, \( N \), as

\[
U_X = \frac{s_x}{\sqrt{N}}
\]  

(4.5)

Systematic \((b_r^2)\) and random uncertainties \((s_r^2)\) are combined using,

\[
U_r = t_{95} \sqrt{b_r^2 + s_r^2}
\]  

(4.6)

where \( t_{95} \) is a 95% confidence level for a given number of degrees of freedom.

Taking the partial derivatives of Equation 4.4 using the DRE in Equation 4.1 we obtain the following equations

\[
\frac{\partial E'}{\partial F'} = \frac{1}{A'} \frac{l^3}{4wh^3}
\]  

(4.7)

\[
\frac{\partial E'}{\partial A'} = -\frac{F'}{A'^2} \frac{l^3}{4wh^3}
\]  

(4.8)

\[
\frac{\partial E'}{\partial l} = \frac{F'}{A'} \frac{3l^2}{4wh^3}
\]  

(4.9)

\[
\frac{\partial E'}{\partial w} = -\frac{F'}{A'} \frac{l^3}{4w^2h^3}
\]  

(4.10)

\[
\frac{\partial E'}{\partial h} = -\frac{F'}{A'} \frac{3l^3}{4wh^4}
\]  

(4.11)

4.3.2 Discussion

The DMA results for the storage modulus are not trustworthy for the first set of tests. This is believed to be a result of the first method used to prepare the samples. In this method, the uneven thickness and varying width in each sample have a large impact on the storage modulus. The span of the supports is fixed and therefore does not play a role in the
amount of scatter in the data. To verify that it is the samples themselves, the same sample is run through the testing multiple times in slightly different positions and also differing orientations. For a single sample there is a large spread in the results. For example, Sample #2 from batch E was tested 4 times and the storage modulus reported as low as 1.2 GPa and as high as 4.2 GPa.

For the second set of samples made for the DMA, much more consistent results are obtained. Even when testing the same sample multiple times, the spread in the reported storage modulus is within 0.5 GPa.

It was hypothesized that the chemical treatment would increase the stiffness of the KCSM compared to the untreated KCSM by increasing the degree of crystallinity, stabilizing the cellulose chains and ridding the fiber of the weaker constituents. However, the untreated samples performed better in both the flexural tests and the tensile tests. By examining the failed region of the coupons, the treated KCSM shattered more than the untreated KCSM. This indicates a more brittle material and therefore, a higher degree of crystallinity in the treated samples. However, it is possible that in the chemical pulping process, the fibers are broken down too much. This is apparent when comparing the feel of both chopped strand mats before the resin is applied. The treated samples being softer and more pliable.

**Comparison of Results**

Mechanical properties of kenaf and kenaf-based composites are reported from Utah State University using different methods. These methods include the DMA, tensile testing, and the half-power vibration test (impact hammer). All research is done independently, but the kenaf fibers are obtained from the same source. The results are listed in Tables 4.5 and 4.6.

In Table 4.5 the neat resin is shown to have a Young’s modulus of roughly 3.8 GPa while it has a storage modulus of roughly 3.1 GPa. Some difficulties arise in testing the neat resin samples. The presence of air trapped in the resin when the hardener is added and stirred is one issue that will adversely effect these properties due to the air space not helping to carry the load. Another difficulty is that the neat resin is extremely brittle and shatters
Table 4.5: Results for kenaf obtained at Utah State University.

<table>
<thead>
<tr>
<th>$F^{ut}$</th>
<th>$E$</th>
<th>$E'$</th>
<th>Material</th>
<th>Fiber/Volume Fraction</th>
<th>Test Method</th>
<th>Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>MPa</td>
<td>GPa</td>
<td>GPa</td>
<td>State</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>78.46</td>
<td>5.41</td>
<td>-</td>
<td>15% NaOH KCSM</td>
<td>50%</td>
<td>ASTM D3039</td>
<td></td>
</tr>
<tr>
<td>94.69</td>
<td>7.85</td>
<td>-</td>
<td>KCSM</td>
<td>50%</td>
<td>ASTM D3040</td>
<td></td>
</tr>
<tr>
<td>-</td>
<td>-</td>
<td>3.88</td>
<td>15% NaOH KCSM</td>
<td>50%</td>
<td>DMA</td>
<td>4.3-4.4</td>
</tr>
<tr>
<td>-</td>
<td>-</td>
<td>5.56</td>
<td>KCSM</td>
<td>50%</td>
<td>DMA</td>
<td></td>
</tr>
<tr>
<td>-</td>
<td>3.76</td>
<td>-</td>
<td>Neat Resin</td>
<td>-</td>
<td>Half-Power</td>
<td></td>
</tr>
<tr>
<td>-</td>
<td>6.11</td>
<td>-</td>
<td>Raw Fibers</td>
<td>40%</td>
<td>Vibration Test [27]</td>
<td></td>
</tr>
<tr>
<td>-</td>
<td>-</td>
<td>3.05</td>
<td>Neat Resin</td>
<td>-</td>
<td></td>
<td></td>
</tr>
<tr>
<td>-</td>
<td>-</td>
<td>4.12</td>
<td>Raw Fibers</td>
<td>20%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>-</td>
<td>-</td>
<td>4.45</td>
<td>Raw Fibers</td>
<td>25%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>-</td>
<td>-</td>
<td>3.79</td>
<td>Raw Fibers</td>
<td>30%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>-</td>
<td>-</td>
<td>4.02</td>
<td>Raw Fibers</td>
<td>35%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>-</td>
<td>-</td>
<td>5.00</td>
<td>Raw Fibers</td>
<td>40%</td>
<td>DMA</td>
<td>[23]</td>
</tr>
</tbody>
</table>

Table 4.6: Young’s Modulus [GPa] for chemically treated kenaf fibers [3].

<table>
<thead>
<tr>
<th>Soak Time</th>
<th>2% NaOH</th>
<th>4% NaOH</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Hr</td>
<td>6.69</td>
<td>7.27</td>
</tr>
<tr>
<td>4 Hrs</td>
<td>6.97</td>
<td>7.42</td>
</tr>
<tr>
<td>8 Hrs</td>
<td>6.56</td>
<td>7.03</td>
</tr>
<tr>
<td>12 Hrs</td>
<td>6.51</td>
<td>7.67</td>
</tr>
</tbody>
</table>

while clamping a coupon in the test fixture for the half-power vibration test. Though a neat resin sample is not tested in the ASTM D3039 test, it would likely shatter in the clamps before a tensile load is imposed.

In spite of the difficulties there are trends with the different tests. The storage modulus increases as the fiber/volume fraction increases. The KCSM allows for a higher fiber/volume content than the raw fiber allows. For Young’s modulus, the half-power vibration test produces a result lower than the KCSM and the treated fibers shown in Table 4.6. This could be due to the test method itself or in how the samples are prepared. The fiber/volume fractions are not given for the data in Table 4.6, but the results are close to value obtained for the untreated KCSM.
Chapter 5

Manufacturing a Kenaf Structure

Kenaf bio-composites are already being used today in many non-structural applications but still have not found many uses in structural applications. Panasonic Electric Works has made a structural wall board from kenaf that could be used in place of plywood. Kenaf core fibers have also been made into a particleboard that is used in furniture components. This chapter presents a method that could be used to make a kenaf-based structure.

5.1 SAMPE Bridge Contest

The SAMPE bridge contest has been held every year since 1998. It gives students the opportunity to design, analyze, and build composite bridges to compete against other students from around the world. In June of 2014, the overall winner was Tongji University from China. There are usually 7 categories to compete in which include: 3 I-beam and 3 box-beam categories for each fiber class: carbon fiber, fiberglass, and natural fibers. The 7th category is an open class category. Official contest rules are posted on the SAMPE webpage the November before the contest, which is normally held in the month of May. Sometimes changes are made to the contest. These have included changes to the loading fixture, size restrictions on the bridges, or a different design load.

To participate, students must join a SAMPE chapter and register for the competition. There are many large sponsors that donate materials that are evenly distributed and sent to participants at no extra cost. Students are also required to submit a design proposal to a governing committee for approval and create a poster to present with their entry to be eligible for awards. The poster presentation should highlight the materials, processes used, and pertinent design aspects of their bridge.
The Test Fixture

A test fixture was designed and built following the guidelines in the 2015 SAMPE bridge contest. It consists of 1” diameter supports whose span has been made adjustable from 5.75-29 inches. Two loading blocks were also built, one following the guidelines and a smaller one that is used to load the I-beams constructed for this research. The test fixture is shown in Figure 5.1.

![Fig. 5.1: The adjustable loading fixture for the SAMPE bridge contests.](image)

5.2 Making the Tooling for I-Beam Construction

Tooling is a very important part of manufacturing any composite. Tooling refers to the mold and mold inserts that hold the fibers and liquid matrix in place until the matrix cures. Tooling is generally made from invar, steel, aluminum, rubbers, nickel, or stiff composites like carbon Fiber. The quality of the final product is dependent on the quality of the tooling used to produce it. Things to consider when selecting the tooling material are the expected number of cycles, coefficient of thermal expansion, desired surface finish, temperature conditions for the cure stage, cost, etc. It would not be wise to use invar as tooling to make only a few parts because the price of invar is so high. Similarly, if elevated temperatures are required to cure the resin system the tooling needs to be able to withstand those temperatures without combustion or significant deformation.

Two different types of I-beam are constructed. One type of I-beam is prismatic. Two of these will be made to the same dimensions, one kenaf and one fiberglass for comparison.
The second type has an arched top flange with web stiffeners in the areas of the loading block and the supports. The later is not tested, but is made to show that complex structures can be simple to fabricate using the KCSM.

**Tooling for the Prismatic I-Beams**

To make the tooling for the two prismatic I-beams, two sections of steel C-channel are cut about 3 inches longer than the targeted length of the I-beam. The C-channel is re-purposed from a bed frame because it is the desired size. The first step is to make the two C-channels as symmetric as possible. This is accomplished using a surface grinder. After grinding, the C-channels will form the shape of the I-beam when placed back to back. Two caul plates are made using 3/16” steel plates. The caul plates are also ground flat on the surface grinder and further smoothed on a belt sander. These will help to form the top and bottom flanges. The tooling is shown in Figure 5.2 along with the KCSM I-beam. The detailed lay-up procedure for the arched-flange I-beam is the same basic procedure used to make the prismatic I-beams.

![Fig. 5.2: The two C-channels and caul plates next to the KCSM I-beam.](image-url)
Tooling for the Arched-Flange I-Beam

To make the more complex geometry of the arched-flange I-beam shown in Figure 5.3, a different method of making the tooling is needed. Note that this I-beam also includes web stiffeners at the supports and where the load is applied. Medium density fiber board (MDF) is used as the tooling because it is only needed for one cycle and the resin system cures at room temperature. The MDF is also cost effective, easy to machine (with machinery available), and requires little effort to create a sufficient surface finish. The drawbacks are that the MDF is weak and will likely sustain scratches or break with part removal and to obtain a smooth surface the MDF tooling is painted. This is unfortunate because many paints react negatively to many common release agents. Release agents should be tested to ensure that the part will not adhere to the tooling.

Fig. 5.3: The arched-flange I-beam with web stiffeners at the supports and loading block.

To make the tooling, six blanks of 1” thick MDF are cut to the approximate height and length of the I-beam. As this is a symmetric I-beam about the web and from end to end, a template is made for only one half of the I-beam which is then used to make a full template. The first template is made from a piece of 1/4” plywood and is used to trace a full template (trace one half, flip, trace opposite half) on a piece of 1” MDF. The outline of
the second template is cut on a band saw leaving all of the traced line. This is not meant to be exact yet! The 1/4” plywood template is temporarily screwed to the MDF template. Now, a flush trim router bit is used to make a copy of the first template, one half routed at a time. Once both sides have been routed, the MDF template can be used to trace the outline on the six blanks. To check that the template is symmetric, simply trace the pattern and flip it to see if there is agreement.

Similar to how the full template is made, after the MDF template is traced on the six blanks they are then cut on the band saw, making sure to leave the lines. The template is then temporarily screwed to each rough cut blank and passed around the flush trim router to create 6 identical blanks. At this point two of the blanks need further work.

To obtain the nice fillets where the web transitions to the flanges a 1/4” round over router bit is used on two of the six blanks. To create the areas for the web stiffeners, a table saw is used. First the height of the blade and the distance from the rip fence are tested on a piece of scrap material. Once satisfied with the depth and location, these channels are cut into the two blanks. The channels are then smoothed and rounded by hand using various files to remove any potential stress risers. At this point the blanks are ready to be combined into the tooling similar to what the C-channels are for the prismatic I-beam. For lack of a better description, they are now referred to as C-channels. They are bonded together using wood glue. After the glue sets, the surface is covered neatly with joint compound to create a smooth surface. Joint compound is used because it dries quickly and sands easily. When an acceptable surface is reached, the tooling is primed with spray paint. Any remaining surface imperfections are much more visible and can be filled with glazing putty and sanded. The next step is to apply a gloss spray paint because gloss paints have a very smooth finish and can be wet-sanded and buffed even smoother.

Now that the two “C-channels” are made, they can be used to make the top caul plate that has to match the curvature of the upper flange. This process is the same as for the C-channel portions except that less blanks are required and they are all glued together. A word of caution is that the caul plate cannot be traced directly from the C-channel. It must
be offset by the expected thickness of the upper-flange to create an even normal pressure while in use. To obtain the proper offset a No. 2 pencil is sanded on one side to adjust where the lead traces onto the material (there is likely a better way). The bottom caul plate is made from a steel plate as detailed for the prismatic I-beam’s tooling.

5.2.1 Fabrication of I-beams Using KCSM

Cutting the Layers

The fabrication process starts by cutting the KCSM to size for the web/flange layers and the top and bottom flange layers and forming the fillers for the web stiffeners. To cut the web/flange layers, two templates are needed. The first template provides the overall shape and is the bold outline in Figure 5.4. The second template is traced onto the previously cut out shape and is the shaded area in Figure 5.4. The 45° lines in this same figure are cut because the KCSM is not able to make the fold from the web to the arched top flange. These lines are not cut in the same place each time, but are cut parallel to each other. This will make is so the weak points are not all in the same place. All cuts are made easily and cleanly with a rotary cutter. To ensure that the I-beam is as symmetric as possible, two layers are cut from a single sheet of paper, which are then numbered and marked left or right. This accounts for any differences that one sheet of KCSM may have from another such as thickness. When the I-beam has a symmetric lay-up there will be no bend-twist coupling or extension-twist coupling.

![Fig. 5.4: Templates for cutting out the KCSM that will wrap around the C-channels forming part of the top and bottom flanges and the web.](image)
The web stiffeners are formed by hand and custom fit. After blending a small amount of kenaf pulp in water a cup of hand-chopped fibers is stirred into the mixture. A handful of the slurry is gathered and rolled to a size slightly larger than the channel for the web stiffener. Pressing the roll of fiber/pulp into the channel removes water while forming a custom stiffener which can be carefully removed and set aside to dry. An example of a web stiffener is shown in Figure 5.5.

![Fig. 5.5: A hand-formed web stiffener removed to dry.](image)

### The Wet Lay-up

First, the layers and the web stiffeners are placed in the oven to remove any moisture that they may have absorbed, Figure 5.6. While this takes place, the tooling is treated with a release agent that is compatible with the surface of the tooling and the resin system. In this case, car wax is a suitable choice.

The lay-up area is set up so that all tools are readily available. The layers are placed into four separate stacks: right web/flange, left web/flange, top flange, and bottom flange. A plastic sheet is placed onto the workstation to keep the area clean and helps to wet-out the material. All of the web stiffeners are dipped into the resin and set onto the plastic to
allow them enough time to become saturated with resin. A small amount of mixed resin is poured directly onto the plastic sheet. A layer of KCSM is placed onto the resin which is quickly absorbed into the material. This method helps to reduce the amount of air bubbles trapped in the material. Another layer is added to the first and then a small amount of resin is poured directly on top and spread across the surface with a plastic spreader. This process is repeated until all of the layers for each stack are wet-out. Next, each layer is inspected for dry areas and then squeegeed to remove excess resin. It is then flipped and placed into a new stack where the opposite side is inspected for dry spots and excess resin is squeegeed off.

With all of the material wet out, the saturated web stiffeners are pressed into their respective channels. A C-channel is oriented so that the web surface is facing up. Next, a web/flange stack is placed on top so that it is only contacting the web surface of the tooling. There is no need to attempt to wrap the material onto the flange surfaces. Two plastic spreaders are placed on the top of this stack on each side of the web/flange stack so that they are only on the portion that will be the flanges. The plastic spreaders will indicate the center of the I-beam before the opposing stack is added. This is more apparent
in Figure 5.7. Next, the opposing web/flange stack is added taking care to keep the stacking sequence exactly opposite of the first stack. The other tooling half may now be placed on top and clamped into place, see Figure 5.7.

![Image](image_url)

Fig. 5.7: The spreaders indicate the center of the web while the two halves are clamped together. A set of blocks facilitate clamping and protect the KCSM when placing the flange layers.

The next step is to pack thin strips of KCSM into the triangular void that is formed by the fillets. If sufficient force is applied on the clamps enough resin should be available to saturate these strips as well as prevent them from being packed in-between web layers. The portions of the web/flange stacks that are hanging off the sides are automatically pressed into place as a stack of flange layers is placed. A caul plate is added to hold the layers in place and protect them while the same procedure is done to the remaining flange. When both flanges are complete, the clamps on the web can be tightened once more and then clamps may be placed on the caul plates, shown in Figure 5.8.

**Release and Trimming**

After allowing to cure, the I-beam is released from the mold. Although the car wax was tested, the I-beam did not come free from the tooling as expected. This is likely due
to the areas between the web stiffeners creating more suction than expected. Some rules of thumb for draft angles are $1/2^\circ$ for parts with side or rib walls up to 1” and $1/4^\circ$ of draft for every additional inch of draw [28]. One of the reasons MDF was chosen as the tooling material was so that it could be destructively removed if necessary.

To trim the I-beam a wooden block wider than half of the flange is placed along the length of the web. The wooden block is parallel to the web and therefore creates a flange edge that is also parallel to the web when run through a table saw while the block runs against the rip fence. This is done for all four flange edges. The ends of the beam are trimmed using a wet tile saw. The final product is seen in Figure 5.3.

5.2.2 Comparing Fiberglass and Kenaf I-beams

For the sake of comparison a fiberglass and a KCSM I-beam are made to the exact same dimension. The fiberglass I-beam has a mass of 279.2 grams while the KCSM has a slightly lower mass of 266.1 grams. They are tested in the 3-point bend test with a 2” wide loading block at the center. The machine used for the loading is a Riehle Model P.H.-200. The load speed was set to approximately 1 inch/minute. The glass I-beam failed at 5,530
lbs due to delamination in the web directly under one edge of the loading block. The KCSM I-beam failed at 3,380 lbs and exhibited brittle failure. Figure 5.9 shows the failed beam.

Fig. 5.9: The kenaf I-beam exhibits brittle failure.

**Discussion**

The KCSM I-beam performed at about 65% of what the fiberglass I-beam demonstrated. The glass I-beam is constructed using a mat which is not an optimal material for fiberglass structures, but the fiber orientation is the most similar to the KCSM. The performance of the fiberglass beam could easily be improved by using a woven s-glass fabric or a uni-directional pre-pregnated material. This shows that fiberglass is still superior to kenaf for structural applications when only considering performance. The KCSM I-beam exhibited a brittle failure that is not desirable for structural members. Brittle failures tend to be less predictable and occur with little warning. This issue may be mitigated by using different chemical treatments on the fiber and/or by using a different matrix. However, concrete is an example of a brittle material that is used in structural applications. If used in a hybrid composite or in a strictly compressive load application the KCSM-composite may be an appropriate choice.
Chapter 6
Conclusions and Future Work

6.1 Conclusions

6.1.1 Manufacturing Kenaf Chopped Strand Mats

Raw kenaf fibers are difficult to manipulate into a composite part without preprocessing. Modified paper-making techniques provide a means to make kenaf chopped strand mats that are easy to work with in making test samples.

The KCSM consists of mechanically pulped fiber and hand-chopped fiber. Through the process investigated in this paper, the pulp and fibers form an inter-fiber network which can be modeled as a random planar beam network. Analysis of KCSM differs from conventional composite analysis as the fiber transmits the load to both the matrix and through fiber-to-fiber contact.

The techniques used to manufacture samples with KCSM result in superior fiber distribution and higher fiber/volume fractions when compared to other methods used to make kenaf-based composites samples at USU. These methods improve the surface finish of the samples and allow for variability in sample geometry. The increase in control over the fiber content and distribution result in a increased mechanical properties and less scatter in the data.

The overall properties for the untreated KCSM samples are a storage modulus of 5.56 GPa, an ultimate strength of 94.7 MPa, a Young’s modulus of 7.85 GPa, and a secant modulus of 7.54 GPa. These results are superior to those obtained by fellow researchers using the same source of kenaf and the same resin system as used in this research. This is more likely due to higher fiber/volume fractions and more consistent fiber distribution as opposed to the structure of the KCSM itself.
Lastly, a chemical pulping procedure was attempted to isolate and stabilize the cellulose portion of the fiber while increasing the overall degree of crystallinity. The treatment did not produce the expected results. In all cases, the treated fibers were outperformed by the untreated fibers. This may be due to the use of improper equipment which introduced contaminants or the treatment may have provided too much pulping [12]. In spite of the negative results, the process used to produce the KCSM functioned just as well for the chemically treated fibers. This is important because other chemical treatments have been found to increase the mechanical properties of kenaf fibers.

6.1.2 Manufacturing a Kenaf-Fiber Structure

The KCSM provided a material that made it possible to build a prismatic I-beam and an arched-flange I-beam with web stiffeners. For the sake of comparison a fiberglass I-beam and a KCSM I-beam were made and tested. The KCSM I-beam had a strength-to-weight ratio that is 65% of what the fiberglass I-beam had. The glass I-beam is constructed using a mat which is not an optimal material for fiberglass structures, but the fiber orientation is the most similar to the KCSM. The performance of the fiberglass beam could easily be improved by using a woven s-glass fabric or a uni-directional pre-pregnated material. This shows that fiberglass is still superior to kenaf for structural applications when only considering performance.

The KCSM I-beam exhibited a brittle failure that is not desirable for structural members. These failures tend to be less predictable and occur with little warning. One possible explanation for the brittle failure is that the fibers are so hydrophilic. As the KCSM absorbs the resin many things happen. The hydrogen bonds that hold the pulp and fibers in the inter-fiber network may be broken down which cause the relative bond area to just be fiber-to-fiber contact. This fiber-to-fiber contact is not efficient in transferring the load and results in decreased strength. Another explanation could be that the pulp and fibers swell with resin to the point that the mechanical properties of the fiber are diluted by the weaker resin. This issue may be mitigated by using different chemical treatments on the fiber and/or by using a different matrix.
6.2 Future Work

One of the objectives of this paper is to provide a material that can be used to make a bridge to compete in the annual SAMPE bridge competition. One area of future work could include a successful entry in this contest which would certainly help to gain notoriety for kenaf-based composites in structural applications. Of course, kenaf is not the only natural fiber, so the methods outlined in this research could also be applied to other types of natural fibers.

As for the KCSM, more research needs to be done to determine its transport properties. The amount of water it holds and how quickly it absorbs it is amazing! One quick test showed that it could hold up to 10 times its weight in water. This is why kenaf is used as an absorbent. Areas for improvement would be to quantify and optimize the relative bond area of the inter-fiber network.
References


