BAGASSE FIBER FOR PRODUCTION OF NONWOVEN MATERIALS

A Dissertation
Submitted to the Graduate Faculty of the
Louisiana State University and
Agricultural and Mechanical College
In partial fulfillment of the
requirements for the degree
of
Doctor of Philosophy

in

The School of Human Ecology

by
Ovidiu Iulius Chiparus
B.S., Technical University "Gh. Asachi", Iasi, Romania, June 1993
May, 2004

Acknowledgements

I would like to express my gratitude to my major advisor, Dr. Yan Chen for his sincere supervision and guidance throughout the course of this study. I also thank to Dr. Billie J. Collier and Dr. John R. Collier for their help and support. It was an honor to know and to work with them.

I would also like to extend my appreciation and gratitude to the person that I consider it to be my "second father" and mentor, Dr. Ioan I. Negulescu. His word of advice and support meant a lot for me.

Furthermore, I am grateful to all the members of my committee: Dr. Betsy Garrison, Dr. Pam Monroe, and Dr. Kristy Reynolds for their cooperation and time.

I also acknowledge the financial support received by the School of Human Ecology. Special thanks to my colleagues and friends from our interdisciplinary research groups.

I deeply acknowledge the encouragement, support, guidance and patience of my friends Christian and Horia Negulescu.

Special appreciation goes to Olena Nesteruk for her permanent moral and physical support, for her patience and love.

I would like to express my gratitude and love to my parents, for whom their children's happiness is their meaning in life.

Table of Contents

List of Tables

List of Figures

Abstract

Raw materials used in nonwoven products vary greatly, covering the entire spectrum from synthetic to natural fibers. The limitation of use for industrial applications of nonwoven products has long been surpassed, today nonwovens being found in diverse applications ranging from intimate apparel to geotextiles.

The present work has as its ultimate goal to develop a commercial method for characterizing some of the physical properties of bagasse or other unconventional fibers obtained through a new atmospheric extraction method, and also to create and analyze different nonwoven structures based on bagasse, kenaf and other annual plants.

Bagasse fibers were extracted from sugar cane rind in two different steps: mechanical separation and chemical extraction. Several factors were considered such as solutions of sodium hydroxide with different concentrations and time of reaction. A similar process was used for kenaf. The kenaf rind containing outer bast fiber was mechanically separated (using a Tilby separator) and chemically treated with an alkali solution.

Even though underrated as a potential fiber, bagasse draws more and more attention because of the increasing concern for disposal of agricultural residues and the need for enhancing the sugar cane industry's profitability. However, there is a lack of an instrumental method to evaluate bagasse fiber length and fineness. This paper presents a study on measuring bagasse fiber fineness using image analysis method. Cross-section images of bagasse fibers were visualized using Scanning Electronic Microscopy (SEM). The relationship between fiber fineness and cross-sectional area was analyzed using the statistical method of regression. The model used in this method can be extended for

evaluating convesely the cross-sectional area when the finess is known, and/or for evaluating other unconventional fibers.

Different structures of nonwoven materials were created through carding, needle-punching, and thermal-bonding. As bonding agents, different types of synthetic polymers have been used depending on the final product usage.

The final products were subjected to testing procedures (according with their usage) such as mechanical determinations, thermal analysis, dynamo-mechanical analysis, biodegradability. The results provided information regarding the possibility to use the nonwoven structurescreated in different applications.

Chapter 1 Review of Literature and General Procedures

1.1 Introduction

In tropical regions of the world sugar cane represents a major crop. Because of the increasing demand for sugar in the last century, large areas in the tropical and subtropical countries all around the world were allotted for sugar cane crops. Low level of maintenance and good productivity made sugar cane an attractive crop for farmers in these regions.

In the US the sugar cane industry started to develop in the second half of the 19th century once steam power was available for agriculture machinery. Today, Louisiana is the second largest producer of sugar cane in the US, only behind Florida. Besides the main product, sugar juice, several by-products are available in the sugar cane extraction process. The most important is considered to be bagasse (Paturau, 1989).

As it can be seen in Figure 1-1, cane is crushed in a series of mills, each consisting of at least three heavy rollers. Due to the crushing, the cane stalk will break in small pieces, and subsequent milling will squeeze the juice out. The juice is collected and processed for production of sugar. The resulting crushed and squeezed cane stalk, named bagasse, is considered to be a by-product of the milling process (Elsunni, 1993). Bagasse is essentially a waste product that causes mills to incur additional disposal costs.

Current research in the US agricultural and forestry industry is concerned with the development of new uses and added value to farm and forestry products for greater economic benefits. Processing and recycling of the natural products is done in an environmentally responsible manner, using these resources efficiently. Utilization of the agricultural crops as alternate raw materials for many industries is more than an option.

Sugar cane bagasse is established as the future fiber in tropical and subtropical regions for pulp and paper making.

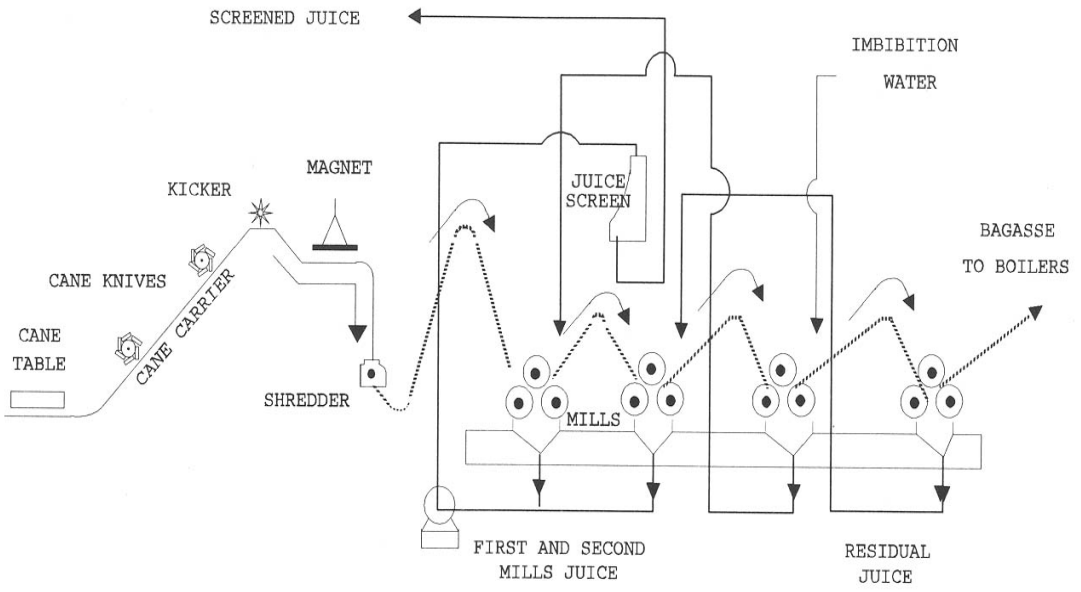


Figure 1-1 Current technological process for extraction of sugar juice from cane in a sugar cane mill (Elsunni, 1993).

According to Atchinson (1995), bagasse will be ahead of other crops as a source for the pulp in paper industry. It is estimated that the amount of bagasse produced annually is about 80,000,000 metric tons (MT), from which 25,000,000 metric tons will be used for pulping (equal to 13% of the total paper-making pulp capacity). Also, kenaf is a very promising material for paper pulps and textile applications (KP Products Inc., 1993).

Structurally sugar cane (Saccharum Officinarum) stalk is composed of an outer rind and an inner pith. According to Paturau (1989) the majority of sucrose together with bundles of small fibers is found in the inner pith. The outer rind contains longer and finer fibers, in a random arrangement throughout the stem and bound together by lignin and

hemicellulose. Previous studies on the extraction of the fibers from sugar cane rind demonstrated that controlled amounts of lignin and hemicellulose could be removed through alkaline and mechanical treatments, resulting in bundles of relatively thin fibers (Collier et al., 1992).

Kenaf (Hibiscus Cannabinus), native to south eastern Asia and parts of Africa, has been used as a natural fiber for hundreds of years. It came into the United States on a commercial scale at the beginning of World War II. It is considered to be an alternative annual crop, used primarily for industrial textile applications as cordage, rope, and burlap cloth (Moreau et al, 1995). Kenaf is similar to jute, but if the technological process is conducted properly, kenaf is more lustrous, has greater tensile strength, and has greater resistance to rot than jute. The price for kenaf fibers became reasonable with the latest improvements in the growing, harvesting, and mechanical separation of fiber and core. Even the leaves and stems of the kenaf cultivars have potential as livestock feed (Webber, 1993). Dried leaves contain 30% crude protein, compared with alfalfa's 16-21% (USDA, 1993). The kenaf plant has two fiber types: the outer bark or the bast portion (40% of the plant) and the inner woody core material (60%) (Sellers et al., 1993). One area that offers opportunity is the incorporation of kenaf fiber into nonwoven composites. Kenaf also found ground for application in pulp and paper industries.

Besides sugar cane bagasse and kenaf, other bast fibers like jute, ramie, and flax were considered by the scientists in the nonwoven industry. Due to their physical and mechanical characteristics these fibers can bring improvement into the overall properties of nonwoven composites. Also, they could be a replacement for the synthetic fibers that

dominate nonwoven industry. Their biodegradation properties make them more and more appealing in the context of new regulations for environment protection.

Further development of value added agricultural products will require not only optimization of the extraction process and determination of the effect of process parameters on fiber properties, but also the development of a valid sampling and measuring technique for the fiber's length and linear density (Romanoschi, 1998). New methods involving image analysis will allow determining physical parameters for unconventional fiber such as bagasse and kenaf in an easy and inexpensive way.

Extracting sugar cane and kenaf fibers from the plant stalks was considered to be a difficult and costly task. The extraction process should be optimized in terms of chemical use and time to maximize the potential for commercial use (Romanoschi, 1998). The primary goal is to remove controlled amounts of lignin and hemicellulose, so as to obtain fibers with uniform length, weight, and fineness. Another goal set by this work was to develop an extraction processthat was as environmentally friendly as possible.

In the last decades nonwoven products conquered several segments of the textile market making the nonwoven industry a success story in a crumbling world of textiles. Nowadays, nonwovens are everywhere from child diapers to car interiors. In this context new design and characterization of nonwoven products represent a need for this new industry. A particular area of interest is the use of cheap alternative crops in making nonwoven composites. Biodegradation, as one of the important characteristics for nonwoven materials, is investigated. The desire for use of nonwoven composites in diverse new applications also requires further testing for different specific conditions. Thermal analysis and dynamic mechanical analysis are tools used by the scientists

nowadays in order to give comprehensive information regarding material behavior under thermal and mechanical factors.

This research will have an impact on economic development in Louisiana and other southern states by providing agriculturists with alternatives in crop choices and adding value to the sugar cane crop. Also, the sugar cane mills can extend their work period beyond the traditional fall season. Converting agricultural by-products to value added products will benefit the economy of Louisiana, and new markets will be developed for agricultural crops (Romanoschi, 1998).

1.2 Research Objectives

1.2.1 To Use the Atmospheric Extraction Process to Obtain Bagasse Fibers

From previous work, it was determined that the most influential factors for the extraction process were alkaline concentration, time of the reaction, mixing, and presence or absence of steam explosion (Romanoschi, 1998). In this study, the extraction process took place at atmospheric pressure. The goal was to design an extraction process as affordable and as environmentally friendly as possible.

1.2.2 To Create Nonwovens Based on Bagasse and Other Annual Plants

Different methodologies can be used to create a variety of structures of nonwoven materials. Depending on the final sought product, a specific technique can be employed. By comparing the final products according to the physical and thermo-mechanical characteristics, a certain path for the technological process of creating nonwovens can be designed. The web formation follows a dry laid carded method. The web bonding according to the raw materials used is accomplished through a mechanical needle-punching method followed by a thermal bonding. The desire is to design a simple,

reliable, and economical technological process that will create nonwovens with characteristics in accordance with the final product properties.

1.2.3 To Determine Physical Properties, Length and Fineness, Through a New Method for Bagasse and Other Unconventional Fibers

Instruments used to determine cotton's length and fineness effectively cannot be employed for the coarse fibers with uneven structure as can be found in unconventional fibers like bagasse and kenaf. A combination of image analysis and statistical regression analysis provides a convenient approach for measurements of some of bagasse's physical characteristics. This method can be extended to other unconventional fibers.

1.2.4 To Establish Testing Procedures and Methods

One of the research goals was to identify testing methods and techniques that would allow assessment of performance of the materials used. In industrial application nonwoven-based materials must satisfy certain behavioral expectations that can be quantified through mechanical or thermal properties. Standard tensile strength testing evaluated the static mechanical properties for different nonwoven composites. Several parameters were registered - the most important being modulus, strain, and stress. These will allow assessment of performance of a certain type of nonwoven composite according to the specifications for the final product and in comparison with other structure-composition combinations. The use of nonwovens in products under the effect of variable stress and temperature recommended the thermal analysis and dynamic mechanical testing. Conductivity and thermal transmittance were determined in order to analyze the heat transfer for different structure-composition combinations of nonwoven as applications for heat insulation. Biodegradation is another important property and was used to asses the capacity of nonwoven materials to decompose in a specific amount of

time. The results were analyzed to determine the most critical tests and to eliminate any unnecessary or repetitive tests.

1.3 Arrangement of This Research Work

This dissertation is organized in three parts. The first part deals with experimental aspects of developing nonwoven composites used in industrial applications. The literature review subchapter presents different applications of sugar cane and kenaf, an extended overview of the previously used bagasse extraction process, the employment of image analysis and thermal analysis in characterizing textiles components, and an overview of biodegradation. In the experimental methods subchapter, all the processing and testing procedures are discussed.

In the second part three proposed articles are presented. The first one in Chapter 2, "An Image Method to Evaluate Bagasse Fiber Dimensions," proposes a new method in determining some of the physical characteristics for bagasse fibers. The second one in Chapter 3, "Manufacture and Analysis of Nonwoven Composites Based on Annual Plant Fibers and Polymers," describes the technological process of making nonwovens using a blend of natural and synthetic fibers, and analyzes the final mechanical and thermal properties. The third one in Chapter 3, "Biodegradable Nonwovens Materials," is an extension of the biodegradability study in the previous chapter. This goal is accomplished by using as a bonding agent a biodegradable polymer. Again, a battery of tests is conducted in order to assess the physical, mechanical, thermal, and biodegradation properties. The third part, Chapter 5, presents conclusions and recommendations.

1.4 Review of Literature

1.4.1 Sugar Cane – Bagasse

Bast fibers represent fibers that are obtained from the stem or stalk of the plants. Grasses such as sugar cane have stems which contain bundles of fibers, but they are not classified as bast fibers (Romanoschi, 1998). The difference comes from the arrangement pattern of the fiber bundles; in regular bast fibers the bundles are in a definite ring pattern, while in sugar cane they are more randomly dispersed. Nowadays several varieties of sugar cane are used in agriculture. The sugar cane plants are known to grow best in tropical and subtropical regions.

Sugar cane stalk characteristics vary considerably depending on variety. Typical commercial varieties grown under normal field conditions have a height of 1.5 to 3 meters and are 1.8 to 5 cm in diameter. The stalk surface can be greenish, yellowish or reddish in color and is covered with a thin waxy layer (van Dillewijn, 1952).

The cane stalk is made up of shorter segments and joints. These joints vary in length from 5 to 25 cm. The lower joints are longer, larger in diameter, and older than the upper joints. Each joint has two distinctive parts, the node and the internode (Elsunni, 1993). At the node and immediately around it are the important stalk structures: the root band, the bud, the growth ring, and the shoulder where the leaf attaches to the stalk (Clements, 1980). In a cross section in the internode we can observe two distinctive areas. The first one, known as the rind, is the outer dense hard layer. The inside layer, known as the pith, is the soft light colored region where the fibro vascular bundles are embedded. The rind varies in thickness and texture along the stalk length.

The fibro vascular bundles are rather widely spaced in the central part of the stalk, but towards the periphery their numbers increase while their sizes decrease. The bundles are composed of smaller ultimate fiber cells which are bound together by lignin and hemicellulose. As the cane ages, there is increased deposition of lignin-like compounds in and around fibro vascular tissues (van Dillewijn, 1952). This phenomenon results in what is called "fiber hardening" up to the time of flowering. After that, as the cane age advances, the process is reversed and the lignin is removed from the bundles causing them to become soft and weak. The softer rind is primarily cellulosic in nature (Elsunni, 1993). The fibro vascular bundles are fibrous strands which extend for long distances in the stem. At the internode they are separated from each other and each is surrounded by pith tissue. Within the internode these bundles run parallel to the axis of the stalk and do not branch. Although the fibro vascular bundles are scattered throughout the interior of the stalk, they are more abundant in the rind region than they are nearer to the center of the stalk. This bundle arrangement increases the strength and the rigidity of the stalk.

The hardness of the cane stalk is a property of considerable concern both in the mill and in the field. Hard cane varieties have been responsible for many mechanical problems (van Dillewijn, 1952). In the field, the sugar cane varieties with hard rinds are known to be difficult for manual cane cutters, and are direct cause of excessive breakdowns of mechanical harvesting units, resulting in loss of parts and valuable crushing time (Barnes, 1964). At the same time, varieties of hard rind have some advantages over softer cane varieties. Rind hardness is closely associated with resistance to attacks by animals such as rats, pigs, and mongooses.

The goal of sugar cane harvesting is to produce sugar cane stalks of high quality. Quality is reduced by damaged cane, increased trash in delivered cane, and delay in cane delivery (Meade, 1977). Most of the high sucrose varieties are fully ripened and ready for harvest when they are 10 to 15 months old. However, in some parts of the world there are varieties which grow for 22 to 26 months before they are ready for harvesting. The longer the growing season, the more likely it is that the crop may lodge and become entangled, making harvesting difficult (Barnes, 1964). For more than 60 years the entire sugar cane crop in Louisiana has been harvested mechanically. However, much of the world's sugar cane is still harvested and loaded manually, and in some parts of the world traditional transport of sugar cane by domestic animals is still in use (Meade, 1977).

Baggasse is a fibrous residue that remains after crushing the stalks, and contains short fibers (see Figure 1-2). Basically, it is a waste product that causes mills to incur additional disposal costs. It consists of water, fibers, and small amounts of soluble solids. Percent contribution of each of these components varies according to the variety, maturity, method of harvesting, and the efficiency of the crushing plant. In Table 1-1 (Elsunni, 1993) a typical bagasse composition is presented.

Figure 1-2. Bagasse

Table 1-1 Average Bagasse Composition

ITEM	*BAGASSE (%)*
Moisture	49.0
Fiber	48.7
Soluble Solids	2.3

Fibers in bagasse consist mainly of cellulose, pentosans, and lignin. Cellulose is a natural linear polymer and has polymer chains of 2000 to 3000 units (Paturau, 1989) and a specific gravity about 1.55 (Elsunni, 1993). Cellulose is highly crystalline regardless of the source. The ordered chains are tightly packed and have strong intermolecular hydrogen bonding because of the preponderance of hydroxyl groups (Romanoschi, 1998). The cellulose is present in three types: α, β, and γ. The α cellulose is known as pure cellulose, whereas β and γ cellulose combined are called hemicellulose (Marthur, 1975). The hemicelluloses are chemically linked with cellulose molecules. The other main compound in sugar cane fiber bundles is lignin which is a high molecular weight substance. Because it is not possible to isolate lignin quantitatively from plant materials without chemical or mechanical degradation, its true molecular weight is not known. The amount of lignin that naturally occurs in sugar cane depends to a great extent on the variety and age of the cane. The amounts of sugar, lignin, and lignin-like compounds increase as the plant advances in age until the flowering time, when the plant is considered to be fully mature. Beyond the flowering time, the sugar cane plant tends to consume its stock of sucrose and lignin as a result of physiological changes due to

flowering. The depletion of the organic compounds makes the rind and the fiber bundles softer and spongy (van Dillewijn, 1952).

For a fiber to be suitable for textile purposes certain qualities are essential and others are desirable. The length of the fiber should be several hundred times the width, which enables fibers to be twisted together to form a yarn. The actual length of the fiber is also important. It can be infinitely long, but should not be shorter than 6 to 12 mm, or it may not hold together after sinning (Batra, 1983). The apparent limitation on length of the sugar cane rind is the internode length, and this varies from 5 to 25 cm. The length of the ultimate fiber cells is from 2 to 4 mm (Paturau, 1989). The length of extracted fiber bundles depends on extraction conditions and the extraction process. The width of the fiber bundles can vary between considerable limits, and eventually determines the fineness. The resultant sugar cane fiber bundles consist of several to hundreds of ultimate fiber cells, with the width of the fiber bundles being dependent on extraction conditions and extraction processes. The amount of lignin removed from the rind affects the size of the fiber bundles as well as their tensile and bending properties (Collier et al, 1992).

Fibers must be strong to withstand spinning and weaving processes. Fiber strength is typically normalized by reporting tensile strength as "tenacity." Tenacity is the breaking load in grams divided by the linear density. Linear density, the mass or weight of a unit length of fiber, is given as grams per 1000m and called "tex," or as grams per 9000m and called "denier." The tenacity of the sugar cane fibers extracted under different conditions is variable according to extraction conditions.

Bending properties of the fiber are of great interest in the spinning process. Fiber bending resistance and hysteresis can be measured on the Kawabata Pure Bending Tester

(Collier, 1991). The results of a previous study showed that the bending properties of sugar cane fiber are significantly affected by extraction variables (Collier et al, 1992). Those variables include pretreatment time, temperature, concentration of alkali solution, and presence of steam explosion.

Nowadays bagasse is mainly used as a burning raw material in the sugar cane mill furnaces. The low caloric power of bagasse makes this a low efficiency process. Also, the sugar cane mill management encounters problems regarding regulations of "clean air" from the Environmental Protection Agency, due to the quality of the smoke released in the atmosphere. Presently 85% of bagasse production is burnt. Even so, there is an excess of bagasse (Figure 1-3). Usually this excess is deposited on empty fields altering the landscape.

Approximately 9% of bagasse is used in alcohol (ethanol) production. Ethanol is not just a good replacement for the fossil fuels, but it is also an environmentally friendly fuel. Apart from this, ethanol is a very versatile chemical raw material from which a variety of chemicals can be produced (Sharma, 1989). But again, due to the low level of sucrose left in bagasse, the efficiency of the ethanol production is quite low.

Figure 1-3. Land-filling with bagasse.

Bagasse pulps are used for all grades of paper: writing, toilet tissue, toweling, glassine, and others. Bagasse newsprint paper is a low-grade and low-priced sheet appropriate for high-speed printing presses, with resistance to deformation, quick oil adsorption, high opacity, and smooth printing surfaces. Bagasse newsprint paper is a commercially successful product in India, Mexico, and Indonesia (Romanoschi, 1998). Research has shown that some key factors should be considered in the production of bagasse newsprint. These relate to the use of a high content of mechanical pulp (all or part of which can be produced from bagasse), the use of highly efficient depithing systems, and the use of a storage method that assures excellent preservation of the bagasse properties, including color and brightness.

Research at Louisiana State University (LSU) has been conducted to determine the feasibility of sugar cane rind fibers for textile and geotextile applications (Elsunni & Collier, 1996). One product is a nonwoven mat formed by suspending the fiber bundles on a screen in water, then dewatering and drying. The mats have been tested as geotextiles for soil erosion control in civil engineering applications (Romanoschi, 1998). A suitable nonwoven mat for geotextiles should sustain, or at least, prevent erosion. At the same time it should be penetrable by growing plants, be capable of permitting interaction between air and soil, and allow rain to penetrate the soil and drain excess water (Collier et al., 1995). Thus, a low-cost, biodegradable geotextile can be produced in local sugar cane mills, providing an economic benefit to both the transportation industry and the sugar cane industry.

1.4.2 Other Plant Fibers – Kenaf, Ramie, Jute and Flax

1.4.2.1 Kenaf

Kenaf bast fibers are derived from the bark of the Hibiscus Cannabinus L plant. Kenaf is an annual plant grown in areas with temperate or tropical climate. At present it is grown in the Sunbelt region of the US, southern Russia, China, Thailand, Indonesia, Bangladesh, India, and in some Latin American countries. It can also be potentially grown throughout south and southeast Asia, Australia, and Africa, as well as in southern Europe, or wherever there are not sufficient or suitable forest resources (Kalgren et al., 1989). There are very low requirements for growing kenaf. The annual production can be up to six to ten tons of raw fibers per acre. USDA field trials showed that kenaf could yield three to five times more fiber per acre per year than southern pine (KP Products Inc., 1993).

When harvested the plant stems are decorticated to remove the inner part of the stalk and then retted to obtain the fiber bundles (Collier, 2001). The bast fibers are constructed of thick-and-thin walled cells (length of: 2 to 7 mm, and diameter of: 10 to 30 μm) that are overlapped and glued together by noncellulosic materials (lignin, pectins, and hemicellulose) to form continuous ribbons (Figure 1-4). The ribbons may run the entire length of 3 to 4.5m (10 to 14 ft) of the plant stem. (Parikh et al, 2002). The refined outer bast fibers measure 3.6 mm and are similar to the best softwood fibers in strength and burst tests. The refined inner core fibers measure 0.6 mm (Kaldor, 1992). The plant's composition is well suited for making newsprint. As was determined, for kenaf to be a viable source for chemical pulping, it would be necessary to separate and to process the

bast and core fibers independently (Jeyasingam, 1990). The two kinds of fibers can be blended in different proportions to produce nearly any grade of paper.

Figure 1-4. Cross-sectional view of kenaf plant (Parikh, 2002)

The process of separating the long and short fibers depends upon the method of harvesting. In frost-free regions, the kenaf stalk is cut while green with special equipment. In cooler regions, the plant is typically frost-killed and a natural drying of the stalk occurs, making harvesting with conventional farm equipment possible. The separation equipment is designed to accommodate the raw material in either whole stalk or chopped (KP Products, 1993).

From an economic point of view, a kenaf pulp mill would require a higher investment for raw material storage and fiber separation than the regular wood pulp mill; however, this is offset by lower costs in raw material preparation (Kalgren, 1989). Because of the lower lignin content than wood (7.7%), fewer chemicals are required for pulping, and, in addition, the fibers require less bleaching. Thus, the wastewater contamination is also reduced, and fewer chemical by-products are produced in the paper-making process. Another factor that distinguishes kenaf bast material from wood materials is the orientation of the fibrils. In bast fibers, the fibrils lie parallel to the plant axis, whereas in wood the fibrils are spirally wound. Thus, the kenaf, as well as other bast

fibers, can be split lengthwise by mechanical action, to yield fine and long fibrous bundles. The high hot-water solubility and the 1% NaOH solubility of kenaf explain also the loss of yield after any thermal or chemi-thermo-mechanical pulping process, as compared with wood. The fungal treatment of kenaf followed by pressurized mechanical pulping results in enhancement of strength proprieties. The controlled fibrillation that restricts the formation of fines is responsible for this behavior (Sabharwal, 1994).

Kenaf plants can grow to a height of 3 to 4.5m (10 to 14 ft) in 5 to 6 months, which make them an abundant, renewable resource (Figure 1-5). Kenaf can compete in cost, quality, and availability, if suitable conditions and land areas exist. In the US, kenaf is also a complementary crop for soybeans, cotton, and sugar cane. In Australia it can replace wheat or rice, depending on location. In Thailand kenaf is an alternative to plantation eucalyptus and cassava. Also there is land availability in most countries that rely on the import of long fiber pulps for which they need foreign currency (Romanoschi, 1998).

Figure 1-5. Harvesting kenaf crop (Nimmo, 2002)

As was stated earlier, for kenaf to become a viable source for pulp paper, it is necessary to separate the bast and core fractions. As a result, the core fibers generated have been investigated as sources for low-density composites (Sellers et al., 1993). Panels of core material were constructed and tested for strength proprieties, dimensional stability, water absorbance properties, and acoustical properties. Phenol-formaldehyde, urea-formaldehyde, and polymeric diphenylmethane diisocyanate resins were used as binders. The results showed that the panels would be suitable for sound absorption-type products. Also, kenaf core panels were produced for ceiling tiles, decorative panel substrates, floor tile substrates, and certain structural components (Sellers et al., 1995). Kenaf can be used as reinforcing fibers in the formation of synergistic property enhanced fiber/thermoplastic composite materials. A blend of kenaf/polypropylene has good tensile properties.

Research has determined also that kenaf fibers are excellent oil absorbent materials and prevent the oil from leaking after absorption. All these properties will be beneficial in minimizing industrial waste (Goforth, 1994). Kenaf can play a significant role in fluid/particle separation operations such as oil adsorption, coalescence, deep-bed filtration, and as filter aids for decreasing the resistance of filter cakes. Fibers can be used to improve filtering characteristics of municipal wastewater (Tiller & Zhon, 1995).

Because of the coarseness and stiffness of the bast fiber bundles, they can be carded only through a coarse wool system or through a modified cotton card. For the fibers to be carded on a cotton card, it is necessary to remove controlled amounts of the binding lignin and make the fibers sufficiently fine and pliable. By controlling the lignin

that is removed it should be possible to obtain fibers that are suitable to the cotton carding system (Parikh et al, 2002).

Kenaf fibers have been incorporated into various types of nonwoven textiles, like kenaf/PP or kenaf/cotton/PP blends, which may be used for products such as fabric softener sheets, furniture underlays, cover stocks, and barrier textiles for medical and agricultural protective clothing (Ramaswami & Boyd, 1994). Kenaf fibers were also bleached to a good white with hydrogen peroxide, and then dyed with direct and basic dyes (Romanoschi, 1997). Kenaf fibers treated with sodium hydroxide have been carded and needle punched into 100% kenaf and kenaf/cotton blended mats. These mats are also biodegradable and have potential in the prevention of soil erosion, the control of weeds, and cleanup of waste liquids (Tao & Moreau, 1994). Kenaf fibers, cleaned of core fibers, have been mixed with refined wood, synthetic and other natural fibers to make various nonwoven needle-punched products: lightweight seeded grass mats, wild flower mats, vegetable strips, erosion control mats, oil absorption mats, pads and pillows, substrates for molded automobile parts, and composites (Fisher, 1994).

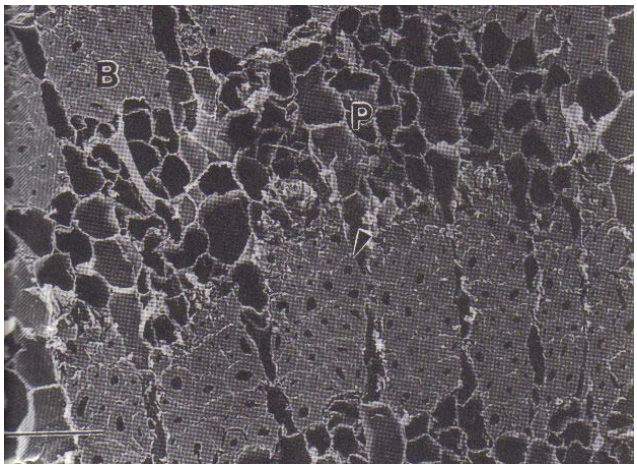


Figure 1-6. SEM of kenaf bast ribbon – cross section showing fibers contained in bundles (Akin et al., 1999)

The feasibility of kenaf for yarns and fabrics was also evaluated. Since jute and pineapple fibers have been used to make coarser fabrics, it was expected that kenaf's good tensile properties and its resistance to mildew and rot might make kenaf an alternative crop for industrial textiles. The fibers were retted both chemically and bacterially, followed by a degumming process. Then the fibers were blended with cotton, successfully spun into yarns, and knitted (Ramaswami & Boyd, 1994).

1.4.2.2 Ramie

Rhea, ramie or 'China Grass' has been grown in the far east for many centuries and its fiber used for making cloth even before the introduction of cotton. Ramie has the look and feel of linen but is higher in tenacity and lower in price. It is stronger wet than dry, does not shrink, and is mildew and rot resistant. Ramie fiber is exceptionally long and lustrous like silk.

Recent research was focused on controlling fiber quality. By controlling the growth and the processing, fibers with similar physical characteristics were obtained. Despite its many excellent properties and diverse uses, ramie failed to become a highly traded textile because of labor and other production costs associated with the processing of the fiber. Until the chemical retting process was developed, only hand methods could be employed to remove the fiber from the stem (Collier & Tortora, 2001). The development of new technologies reduced the cost of production and increased the attractiveness of this fiber. Lowering production cost will extend its end-uses not only in high-value final products, but also in low-value products like nonwovens. Good properties of the ramie fiber may enhance end-use performance of cotton-based diverse industrial applications.

1.4.2.3 Flax

Flax is a bast fiber derived from the straw of an annual plant of Linaceae family of the Linum genus (Smith et al, 2001). The flax plant is aggressively cultivated in several areas of the world (Russia, Canada, Belgium, Ireland, New Zealand, and others), as it is the source of useful components: the stem gives rise to the flax fiber, which is the basis for linen textiles; the seed is the source for linseed oil and linseed cake, an animal feed; the remaining leaves and core of the stem are generally referred to as bract, which is also a product of commerce (de Jong, 1999).

It is interesting to note that flax was the major source of cloth fiber for centuries, until the rapid growth of the cotton industry, which occurred after 1800. Prior to that time, flax was the king of fibers. Fine and coarse linen materials were preserved in the dry climate of Egypt, dating back to 4000 B.C.

Flax plants grow up to 0.6 to 1.5m (2 to 4 ft) in height. Stalks are dried enough so that they can be threshed, combed, or beaten to remove the flax seeds (Collier & Tortora, 2001). The first step is to loosen the bark from the stem and is called retting. The retting process can be done naturally or chemically (Figure 1-7). After retting the flax straw is passed over fluted rollers or crushed between slatted frames. This will break up the brittle stems but does not harm the fiber (Collier & Tortora, 2001).

Flax fiber can be compared with cotton in several respects (Figure 1-8). It has the same specific gravity (1.54 g/d) as cotton, but because of a higher degree of polymerization and a highly oriented structure, it is stronger than cotton. Linen products can be found almost everywhere in the textile complex from extremely fine products to

coarse industrial applications. Linen products are present in household textiles such as bed sheets, table cloths, and wearing apparel.

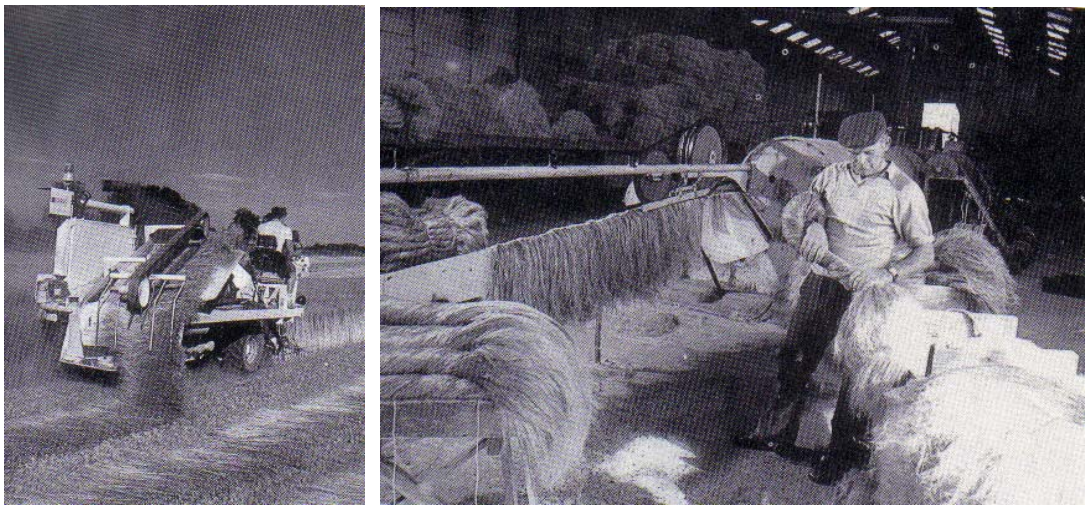


Figure 1-7. Flax harvesting and processing (Collier and Tortora, 2001)

Figure 1-8. Hanks of flax

The use of flax fibers for industrial applications is somewhat limited by the production cost (18 to 35 cents per pound). With new technologies employed to process flax, its price can drop making flax affordable for selected nonwoven composites. An interesting application for flax nonwoven composites is in aqua-culture. This involves growing vegetables and plants in a nutrient solution without soil. The traditional support

used in this method involves mineral fibers that cannot be used for more than five years, and their disposal is a major problem. Flax, a biodegradable fiber, is an alternative solution.

1.4.2.4 Jute

Jute is the name of the fiber extracted from the stems of the plants belonging to the genus Corchorus (Cook, 1994). Jute is another bast plant mainly grown in southern parts of Asia . The plant requires a fertile soil, and a hot and humid climate. The process of getting the fibers out of the stem follows basically the same technological process used for the other bast fibers. First the stalks are retted using a natural or chemical process, and after that the stems are broken and the fibers are removed (Collier & Tortora, 2001).

Figure 1-9. Jute plants

Based on the fiber structure and composition these fibers are most similar with those extracted from sugar cane rind. Due to its physical and mechanical characteristics, jute never found grounds for application in apparel. More than that, after exposure to air the jute fibers become brittle. For a long time jute has been used for industrial textiles like bags, ropes, and cordage. Cheap polypropylene replaced jute in several applications, primarily carpet backing. Recent focus on biodegradability of the industrial textile made jute fibers again of interest. Possible uses are for geotextiles in erosion control.

Developing new technologies for fiber extraction would make jute a more competitive plant in the textile complex.

Tables 1-2 & 1-3 provide an comparative view of the composition and some of the physical properties for the main bast and grass fibers that are the subject of this work.

Table 1-2 Chemical compositions of vegetable fibers (Batra, 1993)

Fiber	*Cellulose (%)*	*Hemicellulose (%)*	*Lignin (%)*
Sugar Cane	50.0	30.0	18.0
Kenaf	65.7	13.2	21.6
Ramie	68.6	13.1	0.6
Jute	64.4	12.1	18.8
Flax (unretted)	56.5	15.4	2.5
Flax (retted)	64.1	16.7	2.0

Table 1-3 Length and linear density of vegetable fibers

Fiber	*Length (cm)*	*Linear density (tex)*	*Cell length (mm)*
Cotton	1.5 – 5.6	0.11 – 0.37	15 - 56
Sugar cane	2.5 - 20	6.50 – 14.00	2 - 4
Kenaf	7 - 15	1.50 – 4.50	1 - 7
Flax	20 – 140	0.19 – 1.98	4 - 77
Ramie	40 – 100	0.51 – 0.71	40 - 250
Jute	150 – 360	1.44 – 3.00	0.8 - 6

1.4.3 Extraction and Evaluation of Fibers

An alkaline technological process was successfully used to extract lignin (Collier et al., 1992). Extraction conditions, such as water pretreatment, alkaline treatment, appropriate mechanical action, presence of steam explosion, and time, influenced the fiber bundle properties (Collier et al., 1992). The first step was the mechanical separation of the rind containing the long fibers from the pith. In conventional processes the entire stalk is crushed and the resulting fibers are short and normally cannot be used in textile processes. The Tilby machine introduced in 1970s was found to be very efficient because it splits the stalks longitudinally and removes the pith (Bourzutschky, 1985). Therefore, longer fibers can be obtained. Originally the machines operated horizontally, but most now work vertically.

Figure 1-10 illustrates the operating principles of both full scale and pilot machines. Billets of cane stalks approximately 30 cm long (see Figure 1-11) are guided onto the splitter blade, which cuts the cane into half shells. Each piece passes to a pith-removing station, which has two rollers. The inner roller is equipped with blades, while the outer roller has spikes in order to guide the piece through this station. The removed pith falls through a chute, is fed onto conveyor, and, in commercial processes, the rind piece enters the next station, which removes the outer wax layer.

To extract the fibers from the rind, sodium hydroxide (NaOH) solutions were used (0.1 N and 1.0 N) at both high (2 atm, 121ºC) and low atmospheric pressures (Collier et al., 1992). Size, tenacity, and bending proprieties of the fibers were evaluated. The results showed that the length of the ultimate cells was not affected, but the

extraction conditions influenced the linear density of the fiber bundles, with alkaline concentration being the most significant factor.

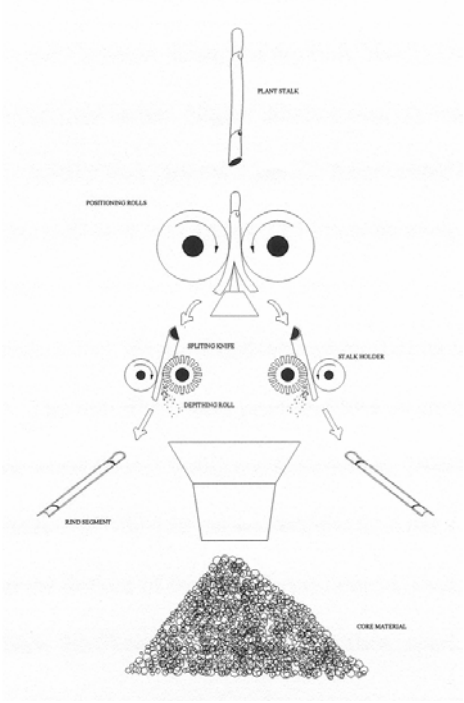


Figure 1-10. Schematic drawing for the Tilby Machine (Tilby et al., 1976)

Figure 1-11. Billets of Sugar Cane Stalks

Higher alkaline concentration yielded fibers with lower tex values. Also, it was observed that the most severe conditions yielded fibers with lower tenacity and toughness values, and lower bending rigidity and hysteresis (Collier et al., 1992).

Steam explosion is a technique developed for separation of fibers from each other in lignocellulosic composites. Application of this technique enables a simple nonreactive solvent extraction of the fiber bonding compounds while retaining the structural integrity of the fiber bundles (DeLong, 1990). The purpose of the steam explosion process was to replace the conventional chemical and mechanical pulping for producing fibers with certain characteristics. The main chemical components of the fiber are lignin, xylan, and cellulose (DeLong, 1990). Lignin is an easily hydrolyzed polymer with a melting point of about 125°C and a degradation point of about 195°C. Lignin and xylan are heavily cross-linked within the fiber bundle. Above 166°C the strength of the cross-link is considerably weakened. At this temperature level, lignin becomes highly soluble in alcohol and mild caustic soda. Meanwhile, the cellulose has a transition or softening temperature of 234°C and a degradation temperature of 260°C. These two temperature limits are well above those of lignin and xylan, allowing cellulose to retain its full structural strength (DeLong, 1990). Steam explosion can either be performed batch-wise or in a continuous manner.

In later work a 20-L reactor fitted with an oscillating agitator was used (Elsunni & Collier, 1996). The best fibers were produced by a steam explosion process modified from one that was used for wood fibers (Wolfgang & Sarkanen, 1989). Steam was injected in the reactor until a pressure of 100-150 psi was achieved. After a few seconds the discharge valve located at the bottom of the reactor was opened, creating a sudden release of pressure, which blew the fibers out, and also blew them apart. Dry, separated fibers were obtained. Fibers with the best proprieties for spinning into yarns were obtained with steam explosion and lower NaOH concentration (Elsunni & Collier, 1996).

Due to the safety concerns in operating a steam explosion reactor, a technological extraction process that would take place at atmospheric pressure was developed. The extraction parameters in this case were alkaline concentration, tumbling speed, and time. In Figure 1-12 is presented the schematic drawing of a continous atmospheric reactor. The extraction process can be continuous, improving the productivity. Similar results with the steam explosion process regarding fiber quality (see Figure 1-13) can be achieved by increasing the alkaline concentration of the solution and the time of reaction. Highly concentrated solutions of sodium hydroxide though create disposal problems.

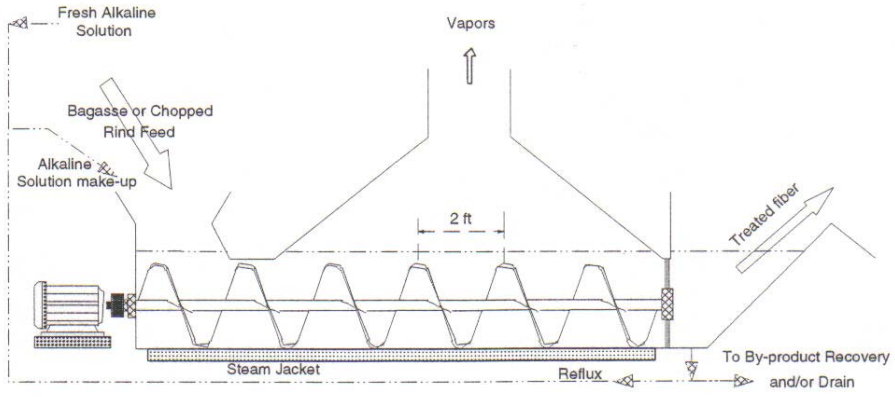


Figure 1-12 Schematic drawing of a pilot scale continuous atmospheric reactor for bagasse.

The physical characteristics of the sugar cane fibers extracted were examined using an environmental scanning electron microscope (Tao & Collier, 1994). The fibers were cut to 5-10mm length, mounted on a stub, and viewed without drying and coating.

Figure 1-13. Delignified Bagasse

The micrographs obtained showed the longitudinal and cross-sectional morphology of the sugar cane fibers after high temperature water pretreatment. The amount of encrusting materials between the ultimate fibers, as well as the size of the fibers, was observed. The SEM examination allowed understanding of the effect of high temperature water pretreatment on the presence of encrusting materials. Tensile and bending properties were also evaluated. It was observed that the most severe conditions yielded the finest fibers but with lower tenacity and toughness values and lower bending rigidity and hysteresis (Collier et al., 1992).

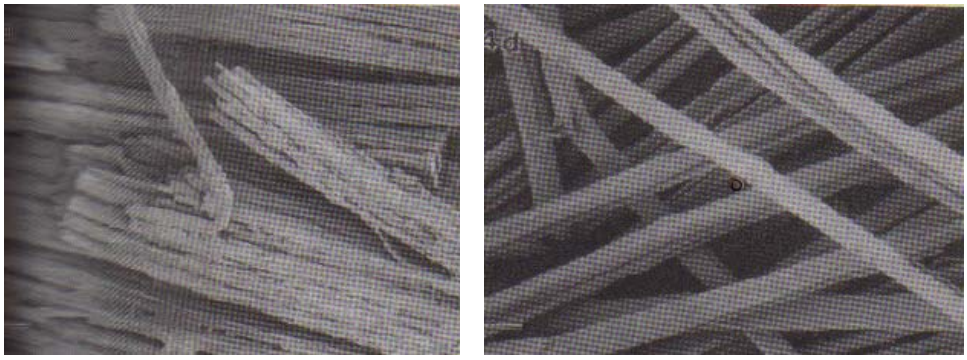
Delignification of jute is often carried out by retting (Figure 1-14). The action of retting involves microorganisms and enzymes, and takes several days to complete - the actual time being dependent on the temperature of the water (Batra, 1983).

The same process, used for the extraction of the fiber bundles from the rind of sugar cane, was adapted to kenaf (Collier et al., 1994). Different kenaf rind sources were used: green whole stalk, dry whole stalk, and peeled, stripped and two-year-stored kenaf.

Figure 1-14. Traditional jute fibers extraction

The process consisted of four distinct steps: mechanical separation, chemical extraction, steam explosion, and product formation. Using this process it is possible to obtain fiber bundles reduced in cross section and with sufficient length for textile applications. By changing the extraction parameters, the final properties of the fiber bundle can be controlled.

The first step for mechanical separation is accomplished with the Tilby cane separator. Next a Rando Cleaner machine can be used in order to further separate the fiber bundles and to dispose of the very short fibers and other impurities. The second process step can be a bacterial or chemical retting. Bacterial retting is a simple process based on the natural action of anaerobic bacteria or aerobic fungi. Chemical retting is a low-concentration alkaline solution, high temperature and pressure process. This process is different from the usual pulping actions since the intent is to extract controlled length fiber bundles from rind strips with minimal reduction of the fiber bundle length while reducing its cross section (Figure 1-15). A supplemental mechanical action, that can be an oscillatory agitation and/or a tumbling motion, is responsible for a preferential reduction of the cross-section rather than the length of the fiber bundles.



(a) (b)

Figure 1-15. Kenaf bast fibers from plant harvested 180 days after planting. (a) With no treatment. (b) Treated with sodium acetate (Akin et al., 1999)

The chemical retting is faster than the bacterial retting, but it has some harmful consequences for the final fiber characteristics: loss in tenacity, color, and luster. Both chemical and bacterial retting encounters some environmental problems regarding air quality and chemical disposal. In the optional third step the kenaf fiber bundles are steam exploded to further reduce the cross sections. In this process, as in the sugar cane fiber case, live steam is injected into the reactor and then the pressure is quickly released. The moisture in the fibers evaporates suddenly, blowing them apart into dry separated fiber bundles. Results showed similar reaction conditions to those applied to the cane rind could also be employed to obtain kenaf fiber bundles that can be used for nonwoven mats or yarn applications (Romanoschi, 1998). Also, as in the case of bagasse case, the extraction can take place in an atmospheric reactor.

1.4.4 Nonwoven Composites

Nonwoven as an industry -especially compared with its traditional textile and paper relatives -has proven to be an interesting and all-encompassing business with hundreds of end uses and product niches. For those new to the industry, the many facets of nonwovens are what make it the interesting and growing business that it is.

The major areas in nonwovens can generally be split into disposable, or "short-life," and durable, or "long-life," end uses. The U.S. market is divided, 60% for disposable markets and 40% for durables, while in Europe, sales lean more toward durables. According to consultant John R. Starr Inc., Naples, FL, disposable applications account for 53% of worldwide roll goods sales while durables represent 47%. The developed markets of the U.S., Canada, Western Europe and Japan accounted for 73% of nonwovens sales last year. Sales in these markets are expected to increase by 7.4% each year for the next five years, according to Mr. Starr. Meanwhile, sales in the developing areas beyond these regions are expected to grow 9.6% per year during the next five years. Paper processes and polymer extrusion technology have become important contributors to nonwovens manufacturing. The electronics industry's development of microprocessors and the subsequent development of affordable, high-speed computing are also contributing to the growing sophistication of nonwoven processes and products (Mansfield, 2001).

In the U.S., the overwhelming volume of nonwovens is utilized in the absorbent product markets of disposable baby diapers, feminine hygiene items (sanitary napkins and tampons), adult incontinence products and medical fabrics. The most influential market for nonwovens is baby diapers, a more than $4 billion market with about 16 billion diapers produced annually (Bitz, 2001). Recent product innovations, such as upstanding leg cuffs and cloth-like backsheets, have dramatically increased the use of nonwoven fabric per diaper, while line extensions into training pants and disposable swimwear products are broadening the category for both branded and private label suppliers. Related to the baby diaper market are the feminine hygiene and adult

incontinence industries, where complementary products and machinery mean similar companies' involvement in the business. Likewise, the market for baby wipes is also related to the baby diaper market in that wipes are sometimes line extensions of baby diaper products. Wipe products are also finding applications in the household cleaning, personal care and pharmaceutical/cleanroom categories. Another disposable nonwovens end use market, one that has withstood the ups and downs of economic hard times and concerns over "disposing of disposables," is medical nonwovens, where the life-threatening impact of AIDS and other contagious diseases have put worker protection above economic and environmental concerns. In the U.S., the medical nonwoven roll goods segment has application in a range of products for use in the operating room on medical personnel, patients, and in myriad other uses throughout the hospital (Bitz, 2001).

Protective apparel is another growing disposables market which, in addition to medical applications, also includes markets in industrial and cleanroom apparel, hazardous waste and asbestos clean-up applications, and agricultural protective apparel.

On the durables side of the nonwovens industry, major markets are geotextiles, which obviously are the highest volume segments for nonwovens. The North American geotextile market is famous for its vast opportunities. Years ago, there were extensive battles between woven and nonwoven fabric manufacturers. Woven manufacturers touted the highest strength per weight and high modulus. Nonwoven manufacturers proclaimed the advantage of higher permeability, better friction, better conformability, and construction survivability. The largest segment of the geotextile market is separation/stabilization geotextiles. The use of separation/stabilization geotextiles keeps

the subgrade soil from pumping up into aggregate that supports the pavement or unpaved aggregate surface. Recent developments in the separation/stabilization market have included studies on geotextile installation and construction survivability and long term survivability (Marienfield, 1995). The next largest market is the paving fabric. Paving fabric is the generic term for nonwoven geotextiles combined with asphalt cement in the field. Placed between layers of asphalt concrete, this interlayer system waterproofs and retards reflective and fatigue cracking in pavements. The third largest and fastest growing segment of the geotextile market is the liner fabrics. Here geotextiles are used in layered geosynthetic and natural liner and drainage system. The purpose of the geotextiles can be geomembrane protection, drainage and filtration, or simple separation of materials. Very heavy nonwovens up to 16 oz/square yard are necessary where protection is the primary function (Marienfield, 1995). The next segment includes applications in trench drainage, drain boards, drain pipe wraps, and highway edge drains. The drainage market segment will continue to grow. There is an increasing awareness by the pavement designers and maintainers that water in pavements causes the most damage and shortens the pavement life. The last significant geotextile market segment which uses predominantly nonwovens is the erosion control and slope protection application. The geotextiles act as filters to allow water to pass while retaining soil. This retards erosion on slopes, shorelines, and stream banks beneath rip-rap stone or another armor system. The market will continue to grow in this area due to increasingly strict laws on pollution and environmental protection (Marienfield, 1995).

Besides geotextiles, nonwovens can be found in automotive applications, which encompass anything from trunkliners and package shelves to door panels and headliner

materials and filtration. Composites reinforced with natural fiber nonwovens have successfully proven their qualities in this field because of their excellent properties, e.g., high strength and stiffness, low weight, etc. One of their most important advantages, however, is the possibility of designing the material itself by arranging long fibers in the direction of the applied forces in order to create lightweight structures with anisotropic properties optimally tailored to each specific requirement (Mueller, 2002).

Also included on the durable side are apparel interlinings, although here more wovens than nonwovens are used. Interlining segments include women's dresses, blouses, sportswear, shoes, hats and handbags and men's tailored clothing, tuxedos, waistbands and lapels, collars and pocket flaps.

Despite continuing consolidation, intense competition and several economic trouble spots around the globe, nonwovens companies continue to expand and grow through capital investments, capacity expansions, joint ventures and acquisitions. Geographic and end use market expansion also continues, with companies claiming new global markets almost daily.

1.4.5 Mechanical Properties

Strength is a mechanical property that we are able to relate to, but we might not know exactly what we mean by the word "strong". There is more than one kind of strength depending on the type of deformation a material undergo. There is *tensile* strength. Tensile strength is important for a material that is going to be stretched or under tension. Fibers need good tensile strength. Then there is *compressional* strength. Concrete is an example of a material with good compressional strength. Anything that has to support weight from underneath has to have good compressional strength. There is also

flexural strength, *torsional* strength, and *impact* strength. A sample has torsional strength if it is strong when one tries to twist it. A sample has impact strength if it is strong when one hits hit sharply and suddenly, as with a hammer.

There is a precise definition for strength. To measure the tensile strength of a material, a sample must be stretched. The stretching is performed by a tensile testing machine. This machine clamps each end of the sample, then, stretches the sample measuring the amount of force *(F)* needed to resist the extension. Dividing this forceby the cross-sectional area *(A)* yields the sample *stress*. If the sample is stretched to the breaking point, the breaking stress is obtained.

$$\frac{F}{A} = \text{stress}$$

.

Likewise, one can imagine similar tests for compressional or flexural strength. In all cases, the breaking strength is the stress needed to break the sample. Since tensile stress is the force placed on the sample divided by the cross-sectional area of the sample, tensile stress, and tensile strength as well, are both measured in units of force divided by units of area, usually N/cm^2. Stress and strength can also be measured in English units, commonly used are pounds per square inch.

All strength tells us is how much stress is needed to break something. It does not tell us anything about what happens to our sample during deformation. That is why it is useful to study the *elongation* behavior of the material. Elongation is the deformation that occurs when a tensile force is applied. Under stress, the sample deforms by stretching, becoming longer. Usually elongation is measured in percentage, which is the length of

the stretched sample (L), divided by the original length of the sample (L_0), multiplied by 100.

$$\frac{L}{L_0} \quad \mathbf{x} \quad 100 \quad = \% \text{ elongation}$$

There are a number of things we measure related to elongation. Which is most important depends on the type of material one is studying. There are two important measures of elongation: *ultimate or breaking elongation* and *elastic elongation*. Ultimate elongation is important for any kind of material. It is the amount one can stretch the sample before it breaks. Elastic elongation is the percent elongation one can reach without permanently deforming the sample; that is, how much can one stretch it, and still have the sample returns to its original length once the stress is released.

If the goal is to know how well a material resists deformation, we measure its *modulus*. To measure tensile modulus, the same thing is done as it was to measure strength and ultimate elongation. The stress is measured, just as it was done for tensile strength. The amount of stress is increased at a constant rate, and the elongation the sample undergoes at each stress level is measured. The process continues until the sample breaks. A plot of stress versus elongation, is shown in Figure 1-16:

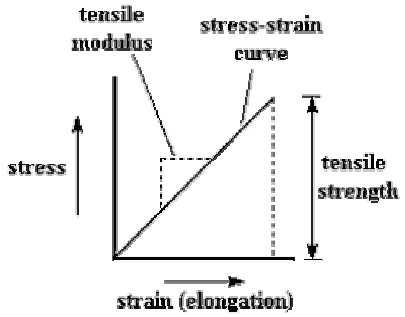


Figure1-16. Stress-strain plot

This plot is called a stress-strain curve. Strain is any kind of deformation, including elongation. The height of the curve when the sample breaks is the tensile strength, and the tensile modulus is the slope of this plot. If the slope is steep, the sample has a high tensile modulus, which means it resists deformation. If the slope is gentle, then the sample has a low tensile modulus, which means it is easily deformed.

There are times when the stress-strain curve is not linear, such as the plot in Figure 1-17. For some materials, especially flexible plastics, we get odd curves that look like this:

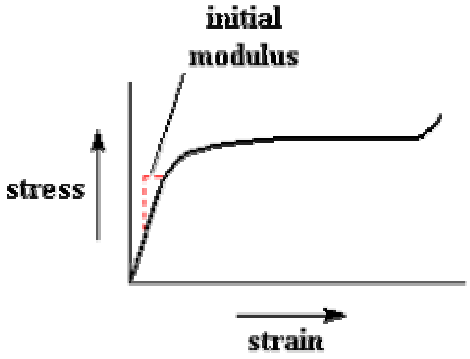


Figure 1-17. Tensile strength plot

The slope is not constant as stress increases. The slope, that is the modulus, is changing with stress. In a case like this the initial linear portion of the curve is used to determine the modulus. In general, fibers have the highest tensile moduli, elastomers have the lowest, and plastics have tensile moduli somewhere in between fibers and elastomers. Modulus is measured in units of stress divided by units of elongation. Since elongation is dimensionless, however it has no units. So modulus is expressed in the same units as strength, such as N/cm^2.

Tensile strength assessment is done according to an established test method or standard. The term standard can refer to the actual method of assessment, to minimum

level safety, or to performance requirements. A problem often occurring in standard textile test methods is how to simulate real-life end use. In the laboratory, usually it can be tested only one property at a time. However in actual use a textile product is subjected to many forces at the same time. It is hard to simulate this combination of effects in one standard laboratory test.

There are several organizations involved in textile testing.

1. American Society for Testing and Materials (ASTM). The purpose of this organization is to develop standards on characteristics and performance of materials, products, systems, and services. The standards developed by ASTM include test methods, specifications, and definitions, and usually deal with physical properties of materials.

2. American Association of Textile Chemists and Colorists (AATCC). This organization was formed to promote the increase of knowledge of textile dyes and chemicals, and therefore it is concerned specifically with textile products. In addition to development of test methods, AATCC sponsors scientific meetings and promotes textile education. All of the activities are concerned with the chemical properties of textiles, in contrast to ASTM's physical emphasis.

3. American National Standards Institute (ANSI). The purpose of ANSI is to coordinate voluntary standards development and use in the U.S. It also serves as liaison between standards organizations in the U.S. and other countries. ANSI is concerned with physical and chemical properties for many different products.

1.4.6 Image Analysis

"Seeing is believing." Sight is fundamental to our understanding of the world. This is as true in science as it is in everyday life. The collection of much statistical data is

dependent upon human vision. For example, the examination of samples under a microscope, observing animal behavior, and the identification and counting of plant species in a field are all forms of image analysis. Humans do a great job in projecting images in our retinas, using a third of our brains for vision. However, computers are being used more and more to automate and extend the potential of image analysis. Computers are better at extracting quantitative information from images than human observers - they can be more accurate and more consistent from day to day. Furthermore, computers may spare us from tedious image interpretation. Progress was expected to be rapid when research commenced in the 1960s on computer-based image analysis. The task, however, has proved to be far more difficult. In part, this is because scientists were not conscious of the processes humans go through in seeing. Biological objects present an even greater challenge to computer interpretation than man-made ones, because they tend to be more irregular and variable in shape.

Manufacturing products that adhere to strict industry and corporate quality standards is no easy task. With competition getting more fierce, smarter, and increasingly global every day, organizations must deal with thinning margins and potentially severe repercussions if inefficiencies and poor productivity are not addressed.

In the quality assurance lab, the situation is no different. Manual laboratory procedures are being slowly replaced with innovative automated solutions that provide consistently accurate, objective and reproducible results. One such technology being implemented into leading labs around the world is image analysis.

A typical PC-based image analysis system consists of a camera mounted on a microscope and attached to a frame grabber. With the use of sophisticated image analysis

software, laboratory technicians are able to easily manipulate images into their accurate binary components of interest, and then analyze their binary structures from an analytical, statistical point of view (Glasby et al., 1998).

With the advent of the computer revolution, today's image analysis systems have come a long way since their early beginnings. These systems used integrated light pen technology to digitize and quantify single objects of interest when pointed at a screen. Significant advancements in hardware coupled with new developments in mathematical algorithms applied to image processing have contributed to the growing acceptance of image analysis technology as an effective tool for image quantification. Image analysis is not a tool used exclusively in quality control laboratories. The use of image analysis in different fields of science created new domains of research and applications: biomedical imaging, industrial vision, remote sensing, scientific visualization, and virtual reality.

Image analysis has been versatile in measuring various properties in the textile world. Quality control specifications for yarn and fabric density, yarn configuration and the knit geometry were checked by image analysis (Zhang, 1996). The composites industry used image analysis to study the fiber breakage in some injection molded plastics (Shortall and Pennington, 1982). In nonwovens, pore sizes as well as structural parameters of individual fibers and bundles have been successfully measured with this technology (Xu, 1996).

There is a need in the textile industry for objective detection of nonuniform coloration in dyed blended textiles. An image analysis system can be used to follow the effects of varying certain conditions for the pretreating of textiles. Dyed fabrics were imaged and the image data digitized. Data analysis gave values that were used to obtain

relative rankings of fabrics that had been dyed under various conditions. This method was also applied to evaluate the possibility of successively reusing a pretreatment bath. Textile mills can use the method for measuring the quality of union dyeing as a blended fabric passes through the processing range (Valiente et al., 2001).

Nowadays several software used in image analysis are in the public domain. The software available can acquire, display, edit, enhance, analyze and animate images. It reads and writes TIFF, PICT, PICS and MacPaint files, providing compatibility with many other applications, including programs for scanning, processing, editing, publishing and analyzing images. It supports many standard image processing functions, including contrast enhancement, density profiling, smoothing, sharpening, edge detection, median filtering, and spatial convolution with user defined kernels (www.scionimage.com). It can be used to measure area, mean, centroid, perimeter, and other features of user-defined regions of interest. It also performs automated particle analysis and provides tools for measuring path lengths and angles. Spatial calibration is supported to provide real world area and length measurements. Density calibration can be done against radiation or optical density standards using user-specified units. Results can be printed, or exported to text files.

1.4.7 Thermal Analysis

The term thermal analysis (TA) is frequently used to describe analytical experimental techniques which investigate the behavior of a sample as a function of temperature. This definition is too broad to be of practical use. TA refers to conventional TA techniques such as differential scanning calorimetry (DSC), differential thermal analysis (DTA), thermo-gravimetry (TG), thermomechanical analysis (TMA) and

dynamic mechanical analysis (DMA). TA is widely employed in both scientific and industrial domains. The ability of these techniques to characterize, quantitatively and qualitatively, a large variety of materials over a considerable temperature range has been pivotal in their acceptance as analytical techniques. Under normal conditions only limited training of personnel is required to operate a TA instrument. This, coupled with the fact that results can be obtained relatively quickly and are accurate and reproducible, means that TA is employed in an ever-increasing range of applications (Hassel, 1991). However, the operational simplicity of TA instruments belies the subtlety of techniques which, if improperly practiced, can give rise to misleading or erroneous results. The abundance of results of dubious integrity in both the academic literature and industrial performance reports underlines the extent and seriousness of this problem (Hatakeyama, 1999).

The advantages of TA over other analytical methods can be summarized as follows (Hatakeyama, 1999):

(i) The sample can be studied over a wide temperature range using various temperature programs;

(ii) Almost any physical form of sample (solid, liquid or gel) can be accommodated using a variety of sample vessels or attachments;

(iii) A small amount of sample (0.1 μg-10 mg) is required;

(iv) The atmosphere in the vicinity of the sample can be standardized;

(v) The time required to complete an experiment ranges from several minutes to several hours;

(vi) TA instruments are reasonably priced.

In polymer science, preliminary investigation of the sample transition temperatures and decomposition characteristics is routinely performed using TA before spectroscopic analysis is begun. TA data are indirect and must be collated with results from spectroscopic measurements, before the molecular processes responsible for the observed behavior can be elucidated. Irrespective of the rate of temperature change, a sample studied using a TA instrument is measured under no equilibrium conditions, and the observed transition temperature is not the equilibrium transition temperature. The recorded data are influenced by experimental parameters, such as the sample dimensions and mass, the heating/cooling rate, the nature and composition of the atmosphere in the region of the sample and the thermal and mechanical history of the sample (Pierce, 1996).

The general conformation of TA apparatus (consisting of a physical property sensor, a controlled-atmosphere furnace, a temperature programmer and a recording device) is illustrated in Figure 1-18. Table 1-4 lists the most common forms of TA.

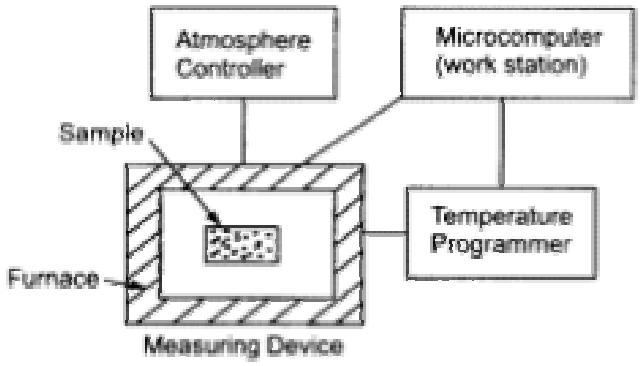


Figure 1-18. Block diagram of a TA instrument.

Modern TA apparatus is generally interfaced to a computer (work station) which oversees operation of the instrument controlling the temperature range, heating and cooling rate, flow of purge gas and data accumulation and storage.

Table 1-4 Conventional Forms of TA

Property	*TA Method*	*Abbreviation*
Mass	Thermogravimetry	TG
Difference of Temperature	Differential thermal analysis	DTA
Difference of Temperature	Alternating current calorimetry	ACC
Enthalpy	Differential scanning calorimetry	DSC
Length, Volume	Dilatometry	
Deformation	Thermo mechanical analysis	TMA
	Dynamic mechanical analysis	DMA
Electric Current	Thermo stimulated current	TSC
Luminescence	Thermo luminescence	TL

Various types of data analysis can be performed by the computer. A trend in modern TA is to use a single workstation to operate several instruments simultaneously.

Thermo-gravimetry (TG) is the branch of thermal analysis which examines the mass change of a sample as a function of temperature (scanning mode) or as a function of time (isothermal mode). Not all thermal events bring about a change in the mass of the sample. Melting, crystallization,and glass transition do not exhibitmass change whereas desorption, absorption, sublimation, vaporization, oxidation, reduction and decomposition do. TG is used to characterize the decomposition and thermal stability of materials under a variety of conditions and to examine the kinetics of the physicochemical processes occurring in the sample. The mass change characteristics of a material are strongly dependent on the experimental conditions employed. Factors such

as sample mass, volume and physical form, the shape and nature of the sample holder, the nature and pressure of the atmosphere in the sample chamber and the scanning rate all have important influences on the characteristics of the recorded TG curve.

Differential Scanning Calorimetry (DSC) is a branch of TA where the differential energy supplied between a sample and reference to maintain a minimum temperature difference between the sample and reference in response to a temperature program is used to investigate the nature of the sample. The DSC curve is a graphic representation of the data collected, where the differential energy supplied is plotted as a function of temperature (scanning mode) or time (isothermal mode). Both, sample and reference are subjected to continuous temperature increase (Negulescu, 2001).

Dynamic mechanical analysis (DMA) is a branch of thermal analysis where the behavior of a sample subjected to an oscillating stress in response to a temperature program is used to investigate the nature of the sample. Most DMA measurements are made using a single frequency and constant deformation amplitude while varying temperature (Foreman, 1997). The value of a dynamic test is most significant, in that a 30 to 60 minute experiment yields a tremendous amount of information on the sample in question. The modulus value below the glass transition will tell about levels of molecular orientation and crystallinity. Transitions occurring can be related to the polymer's structure and may be particularly useful where a multiple component blend is under investigation. Dynamic mechanical methods are the most sensitive way of measuring the glass transition itself, which is one of the key properties of a polymer from both the structural and processing viewpoint. Above the glass transition the rubbery behavior yields important factors such as the effective cross-link density and clues to

processability. Finally data can be obtained in shear mode after the melting point. This technique is complementary to infra-red or NMR results on chemical composition. These tests yield comprehensive detail on the molecular moieties present, whilst the mechanical data reveal how they are connected together (www.triton-technology.co.uk).

1.4.8 Biodegradation

Concerns for a clean environment have impacted not only textile manufacturers but also consumers in the choice of raw materials to final products. Public awareness increasingly demands biodegradable or environmentally friendly textiles, especially disposable nonwoven products. The possibility of composting disposable nonwoven products, such as diapers, incontinence products, surgical gowns, and landfills has attracted special attention in an effort to solve the solid waste crisis. Other niches of the nonwoven textile industry, such as geotextiles, deal with the same problem. Unfortunately, there are only a few biodegradable fibers available that can serve as raw materials in nonwoven production, and in most cases newly developed biodegradable fibers are expensive (Suh et al., 1996).

Biodegradation by definition is a biological process and requires microorganisms such as bacteria, fungi, algae, and acetinomyecetes (Suh et al., 1996). Biodegradation of textile materials is usually associated with the presence of natural fibers. Due to the relatively modest physical and mechanical properties of the natural fibers, their use in nonwovens is limited. Even so, presence of synthetic materials is required as a bonding agent, except for needlepunching bonding. Cotton, for centuries the most important of fibers, is now taking second place to synthetics. Viscose rayon staple fibers were, in

1966, the cheapest manufactured fiber. Now they are around twice the price of the main synthetic without the ability to be easily spun-laid or thermal bonded (Woodings, 2001).

The biodegradation mechanism is generally explained by the enzymatic catalyzed process, where enzymes are produced by various microorganisms in the presence of degradable substrates. Requirements for microbial growth vary with temperature, pH, and oxygen availability (Suh et al., 1996). Usually the presence of moisture and nutrients is necessary. The biodegradation process involves a number of different mechanisms, including hydrolysis and oxidation, which result in polymer chain scission (Cooke, 1990). Intermediate products from the continuation of the chain breakages are water-soluble fragments. As total mineralization proceeds, further degradation products are carbon dioxide (CO_2), water (H_2O), methane (CH_4), and biomass (Cooke, 1990). The biodegradation of cellulose-based fibers has been intensively studied. Cellulose is believed to be readily biodegraded by many microorganisms due to the activity of cellulose enzymes catalyzing the hydrolysis and oxidation (Finch and Roberts, 1985). The cellulose enzymes are classified into three groups according to their catalyzed reactions: hydrolases, oxidases, and phosphorylases (Finch and Roberts, 1985). Enzymatic activities on cellulosics are influenced by many factors depending on their morphological and physical structures. These enzymes act by hydrolyzing or oxidizing the polymer, and can work at the end of the chains (exo-enzymes) or randomly along their length (endo-enzymes). The higher the degree of polymerization and the greater the degree of crystallinity and orientation, the less susceptibility to microbial attack due to limited accessibility. The most biodegradable fibers therefore tend to be hydrophilic, and made up of short, flexible chains with low levels of crystallization. They will often have

chain backbones with oxygen or nitrogen links. This description clearly fits most natural fibers and many natural polymers.

Biodegradation within a specified time frame in a solid compost medium is called composting. Composting is a managed process that controls the biological decomposition and transformation of biodegradable material into a humus-like substance called compost. A material is compostable if it is capable of undergoing biological decomposition in a compost site as a part of an available program such that the material is not visually distinguishable and breaks down into carbon dioxide, water, inorganic compounds, and biomass at a rate consistent with the known compostable materials (ASTM D 6002 – 96).

Population and social concerns, the Green movement and government mandates have provided an incentive to develop and use biodegradable polymers (Haille et al., 2001). Polymer scientists have facilitated a paradigm shift in the use of textiles, from the historical desire to improve durability, weatherability and other long life characteristics, to a triggered or reproducible breakdown under prescribed conditions.

Biodegradation in fibers, including synthetic ones, occurs when their constituent polymers are depolymerized. Biodegradation-resistant polymers have the opposite characteristics and unsurprisingly are used to make the stronger more durable fibers. Oxygen-free polymers such as polypropylene and polyethylene resist biodegradation totally. Polyester, despite its oxygen content is degradation-resistant probably because it has rigid, rod-like chains. The same is true for polyamides despite their nitrogen content. Unlike aromatics, aliphatic polyesters are generally biodegradable. Manufactured biodegradable aliphatic polyesters are, however, still based mainly on industrial

polymerization of monomers such as glycolic acid (PGA), lactic acid (PLA), butyric acid (PHB), valeric acid (PHV) and caprolactone (PCL) (Woodings, 2001). Despite these new achievements in biodegradable polymer development, the use of these materials is limited by the production costs. It is forecast that by the end of this decade, advancements in technology will allow manufacture of biodegradable polymers at a price level comparable with the regular polymers.

1.5 Experimental Methods

1.5.1 Processing Procedure

Bagasse fiber used in this study was provided by a local sugar mill, from the 2001 crop. The raw bagasse was already crushed at different lengths as an output from the sugar extraction process. To extract the bagasse fibers, a series of mechanical and chemical procedures was used. Waste bagasse was laid out on the ground of an open but roofed area in the LSU Audubon Sugar Factory for a period of two weeks. To assure a uniform drying process the layer of bagasse was turned over once a day. The moisture content was measured for randomly selected samples of fibers. The results indicated a consistent level of moisture less than 15%. Small fibers and impurities were removed through a sifting process using a 2 ft by 2 ft wooden frame sieve having a screen with 1/16 in eye dimension.

For the alkaline extraction, an atmospheric process was employed. An LSU-designed, previously built atmospheric reactor was used (Figure 1-19). From previous studies, it was determined that a 2.0 N NaOH soulution is required to remove a significant amount of lignin. The measured volume of the reactor was 200 liters. Certified A.C.S. NaOH pellets were used. The solution was heated up to the boiling point

(approximately 100°C). Sifted bagasse was fed into reactor gradually. The fiber/liquid ratio used was 1:10. In approximately 90 minutes the whole amount of bagasse, intended for delignification, was collected at the other end of the reactor. The extracted fibers were rinsed thoroughly with water and left to dry for two days in a controlled environment with a relative humidity of 65% and a temperature of 71°C.

Figure 1-19. Atmospheric Extraction Reactor

The same procedure for delignifying bagasse was replicated on a smaller scale in the laboratory. In laboratory experiment the atmospheric reactor was replaced with an 8 liter pot. For drying an oven (Blue M Electric Company) was used. It was determined that the necessary time for drying was 45 minutes at a preset temperature of 135°C.

For kenaf the procedure used was similar. The fiber/liquid ratio was 1:20. The fibers were boiled for 1 hour in a 2.0 N NaOH solution, neutralized in a 0.3% hydrochloric acid (HCl), and washed again. Kenaf fibers were dried at 135°C for 45 minutes in a Blue M Electric Company oven.

1.5.2. Nonwovens Formation

The extracted bagasse and kenaf fibers were first cleaned using a Cleaning McPherson Machine at USDA Southern Regional Research Center (SRRC) in New Orleans. The cleaned bagasse and kenaf were blended manually with cotton, ramie or polypropylene in different ratios by weight. For intimate blending and nonwoven web formation, a carding process was employed. The different samples were fed into a F105 D Universal Carding Machine (Figure 1-20). To enhance uniformity of the web, each combination was passed several times through the carding machine.

Figure 1-20. F105 D Universal Carding Machine

The web bonding was realized in two different ways. A Morrison Berkshire Needle-Punching Machine (see Figure 1-21) was used for mechanically bonding the bagasse/cotton/biopolyester, kenaf/cotton/biopolyester, bagasse/kenaf/polypropylene, kenaf/ramie/polypropylene, bagasse/ramie, kenaf/ramie, bagasse/polypropylene, kenaf/polypropylene, and ramie/polypropylene. The feeding speed was 5.4 ft./min. and the punching rate 228 strokes/minute. Each sample was punched twice faced up and twice faced down.

Figure 1-21. Morrison Berkshire Needle-Punching Machine

The carding and needle-punching processes for nonwoven fabrication were purposely chosen for the development of these structures because they have been used to make automotive interior mats that can be easily thermoformed into various shapes and molds for automotive interior trim parts (Figures 1-22 & 1-23).

Samples of nonwoven based on jute, kenaf, cotton, and polypropylene (PP) were provided by USDA Southern Regional Research Center in New Orleans.

Figure 1-22. Three Layer (sandwich type) Nonwoven Composite – Ramie/Kenaf/Polypropylene

Figure 1-23. Three Layer (sandwich type) Nonwoven Composite – Bagasse/Kenaf/Polypropylene

1.5.3 Thermal-Bonding

Thermal-bonding was done using a Carver Laboratory Press (see Figure 1-24) with heated plates. The thermal-bonding temperature was set to 150°C and the heating time was one minute. Thickness of the thermal-bonded nonwoven was set to 0.6cm (1/4") and 1.3cm (1/8"). The pressure applied was 8.62 Pa (12,500 psi). Due to the limitations of the press dimensions, the samples obtained measured 12.5 cm x 15 cm (5" x 6").

Figure 1-24. Carver Laboratory Press

1.5.4 Static Mechanical Testing

The tensile strength of the nonwoven composites was conducted according to ASTM D5035-95 on an Instron Tester Model 4301 (Figure 1-25). For data recording the Instron software of Series IX Automated Materials Testing System with an interface type 4200 was used. Sample sizes were 25.5 mm x 76.5 mm (1 in x 3 in). Before testing the samples were at 20°C (70°F) and 65% relative humidity for 24 hours. The samples were mounted between the machine jaws and pulled apart until breaking. Grip distance was set at 2 inches and the specimen gauge length was 1 inch. The crosshead speed was set at 0.5 in/min. For needle-punched nonwovens, a load cell of 1 kN (200 lb) was used, while for the needle-punched and thermal bonded nonwoven composites a 5 kN (1000 lb) load cell was used.Recorded tensile properties include: displacement at maximum load, load, stress, strain, maximum percent strain, and modulus. For each type of nonwoven structure, at least three samples were analyzed and the mean and the standard deviation were reported.

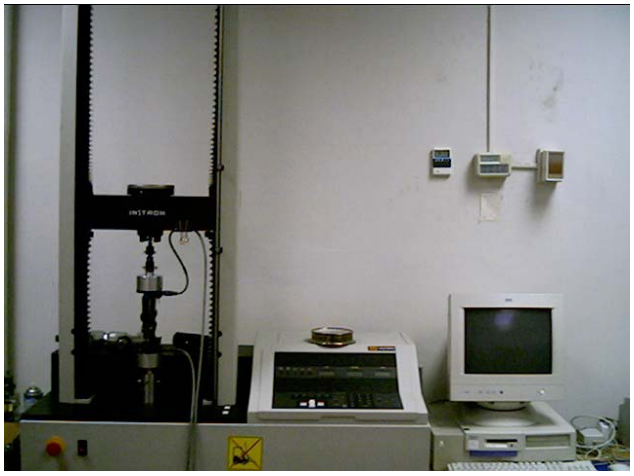


Figure 1-25. Instron Tester Model 4301

Flexibility of the thermal-bonded nonwoven composites was tested in accordance with the ASTM 790-97 for flexural properties of plastics (three-point bending method) on

the Instron Tester Model 4301. The samples were subjected to 24 hours at of 20°C (70°F) and of 65% relative humidity. The sample dimensions were 25.5 mm x 76.5 mm (1 in x 3 in). The load cell used was 1 kN (200 lb) with a cross-head speed of 1.0 in/min. The parameters of interest in this case were: displacement at yield, strain at yield, load at yield, stress at yield, and modulus. A minimum of three samples for each structural type of nonwoven composites were analyzed and the mean and standard deviation were recorded.

1.5.5 Image Analysis

In order to determine physical characteristics of bagasse fibers, image analysis was used. Due to the requirement regarding a statistical analysis, it was decided that 24 samples were needed for analysis. Each sample contained seven fiber randomly assigned.

For length measurements, a digital imprint for each was obtained using an AGFA Duoscan T1200 scanner. The images were collected as a JPEG (Joint Photographic Experts Group) file, which is a standardized image compression mechanism. JPEG is designed for compressing either full-color or gray-scale images of natural, real-world scenes. The length was measured directly using the Scion Image software for Windows 2000. Scion Image allowed us to measure the length for each individual fiber by following the fiber imprint contour. Scion Image may be used to capture, display, analyze, enhance, measure, annotate, and output images. It also has advanced capturing capabilities such as frame averaging and summation, frame sequence, and on-chip integration support.

To determine the cross-sectional area, the bagasse fibers were cut individually to a 1mm length and glued with the cross-section up on stubs using “Spot-o-gold” labels.

The cross-sections were coated with 25 nm gold palladium using a Hummer II Sputter Coater. A scanning electronic microscope (SEM) Cambridge 260 Stereoscan, with a magnification between 15 and 50,000 and a resolution of 10 nm-29 nm, was used to visualize the cross-section of the bagasse fibers. The pictures taken were saved as JPEG files (Figure 1-26). Again, the Scion Image software allowed direct determination of the cross-sectional area for each individual fiber. Multiplying by seven (the numbers of fibers in each sample) was how the corresponding area for each sample was determined.

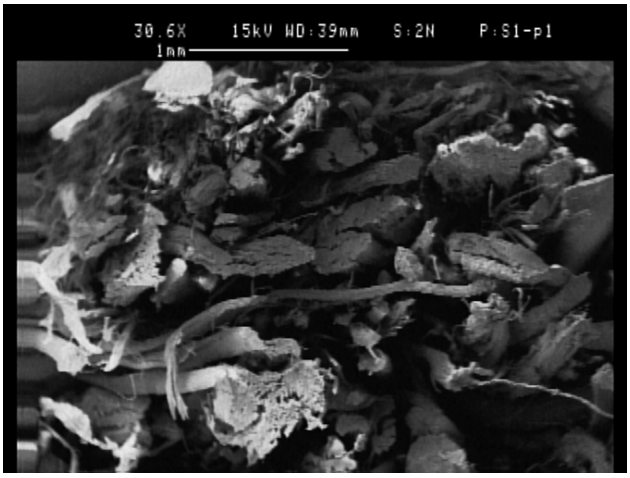


Figure 1-26. SEM Image for Bagasse Fiber Bundle Cross-Section

1.5.6 Thermo-gravimetric Analysis

Thermo-gravimetric analysis (TGA) was carried out in air and inert atmosphere of nitrogen. The range of temperature used was between 0°C and 600°C with a temperature increase rate of 5°C/minute. A TA thermobalance was used.

1.5.7 Dynamic Mechanical Analysis (DMA)

For the DMA tests, Seiko dynamo-mechanical spectrometers DMS 200 (Figure 1-27 and DMS 110 (Figure 1-28) were used. By utilizing DMS 200 the behavior of materials as a function of temperature to dynamic mechanical tensile stresses is

determined, while DMS 110 showed the materials behavior as a function of temperature to dynamic bending (flexural) stresses.

Nonwoven composite samples based on Eastar BioCopolymer (EBC) were analyzed in a large range of temperature from -100°C to 200°C. For tensile stresses five frequencies were used: 0.01 Hz, 0.1 Hz, 1.0 Hz, 10.0 Hz, and 100.0 Hz. The heating rate employed was 1°C/minute. For the bending mode, a train of frequencies from 0.01 Hz to 50.0 Hz was considered appropriate, with a heating rate of 0.5°C/minute.

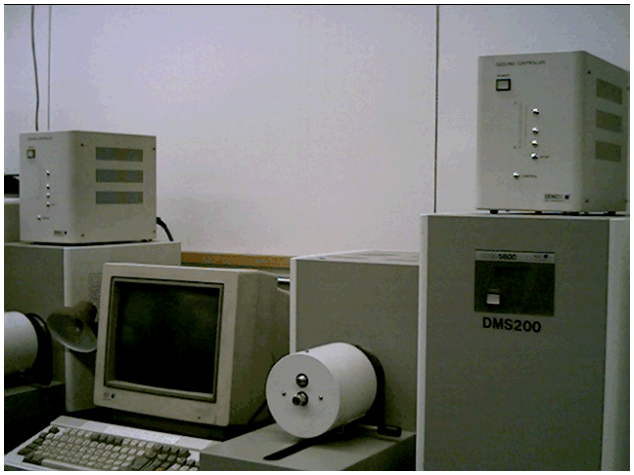


Figure 1-27. Seiko dynamo-mechanical spectrometers DMS 200

Figure 1-28. Seiko dynamo-mechanical spectrometers DMS 110

For Ramie/Kenaf/Polypropylene (RKPP) and Kenaf/Bagasse/Polypropylene (KBPP) for the tensile mode (DMS 200), four levels of frequencies were used: 0.01 Hz, 0.1 Hz, 1.0 Hz, and 10.0 Hz. A total of 274 steps were performed with the starting temperature of 26°C and the ending temperature of 200°C. In bending mode five levels of frequencies were used: 0.01 Hz, 0.1 Hz, 1.0 Hz, 10.0 Hz, and 50.0 Hz, with the same temperature range. Both tensile and bending analyses were carried out in no special gas environment.

The parameters of interest for both tensile and bending mode were the storage modulus E', the loss modulus E", and their ratio, the tangent of the loss angle denoted by $\tan\delta = E"/E'$.

1.5.8 Thermal Conductivity and Thermal Transmittance

Thermal conductivity and thermal transmittance were performed at USDA Southern Regional Research Center in New Orleans. The test method used was ASTM D1518-85. All samples were conditioned for 24 hours prior to testing at 21°C (70°F) and 65% relative humidity. Sample dimensions, according to machine requirements, were 150 mm x 150 mm (8" x 8"). Thickness for each sample was determined according to ASTM D1777-85. For testing a thermal conductivity meter, FOX 200, manufactured by LaserComp Corporation was used. Samples were placed in between two plates with different temperatures: for the cold plate the temperature was 21°C (69.9°F), and for the hot plate the temperature was 36.6°C (97.9°F). The parameters of interest were: coefficient of thermal conductivity (λ), and the heat transmittance. The average of three samples with triple measurement for each sample was used to calculate the mean values for each nonwoven specimen.

1.5.9 Composting

There is no unique method to evaluate the biodegradability of nonwoven composites. ASTM D6002-96 provides the standard guide for assessing the compostability of environmentally degradable plastics. According to this standard, the compostability of products can be determined using backyard composting environments. The composting process tends to be slower due to the relatively short or non-existent thermophilic composting phase. Biodegradation was assessed through loss of integrity and quantified by tensile strength determinations according to ASTM D5035-95. Material degradation was also determined based on the weight loss. Surface damage was evaluated by visual inspection.

Several samples of nonwoven composites were buried in rich composting soil in the backyard of a farm in Arkansas. After each week some of the samples were removed and analyzed. The length of the experiment was 6 weeks. Static mechanical parameters as modulus, strain, and stress were recorded using an Instron Tester Model 4301. The data recorded from each week were compared against the other weeks' results.

The soil burial method is not the only one used in biodegradation assessment. HS 2001- Biodegradability Test gives a guideline regarding biodegradability assessment of disposable plastic materials exposed to a controlled composting environment under laboratory conditions. The test method is designed to yield the percentage of conversions from carbon dioxide in the test materials as their rate of conversion.

Chapter 2 An Image Method to Evaluate Bagasse Fiber Dimensions*

2.1 Introduction

The U.S. sugar cane industry started to develop in the middle of the 19th century, and currently Louisiana is the second largest producer of sugarcane after Florida. Many by-products are available from sugarcane industry, but the most important is bagasse (Paturau, 1989). Recent research in the U.S. agricultural and forestry industry has as its objective investigating new uses of the agricultural by-products for developing value-added industrial products to enhance the sugar cane industry's profitability. Bagasse fiber can be extracted from bagasse residue for textile applications. In the current production process, cane is crushed in a series of mills. The crushing causes cane to break in small pieces. The crushed and squeezed cane stalks are known as bagasse. The sugar cane stalk is composed of an outer rind and inner pith. The pith contains small fiber and the majority of the sucrose, while the rind contains longer and finer fibers, arranged randomly throughout the stem and bound together by lignin and hemicellulose (Elsunni, 1996).

Waste bagasse is considered as a type of unconventional fiber because of its very limited applicability in textile industry. More processing steps are needed before it can be used as an alternative fiber for textile industry. Because the bagasse fiber is unusually coarse and stiff, it is suitable for making nonwoven products. Nonwoven materials are becoming important industrial materials with diverse end-use applications like insulation, sound deadening materials, sorbents, and geotextiles (Collier, 1995). Bagasse nonwoven

* Reprinted by permission of *Bioresource Technology Journal.*

materials are also biodegradable and environmentally friendly, and, therefore are more attractive to industries either in replacement for synthetic materials or in use as reinforcement materials for a polymer matrix.

One of the most important physical characteristics of textile fibers is the fineness. For the traditional fibers like cotton and wool, instrumental methods to determine fiber fineness are well developed. Basically, there are two types of measurements for fineness: a direct method by which fiber cross-section is measured, and an indirect method which measures fiber length per unit weight, or fiber weight per unit length (linear density). The direct method is not practical because the shape of the cross-section of many natural fibers is irregular. So, it is quite difficult to compute the area or to measure precisely the diameter.

Several methods were developed over time to determine the fineness for the most commonly used fibers, cotton and wool. By British Standard Method, a known amount of fibers is cut to a particular length and then weighed. The Arealometer method (ASTM D1448-97) uses a Wheatstone bridge in a tube with two channels. A known amount of cotton is dropped in one of the channels while the other one is left empty. The pressure drop is equalized in both sides, and the fineness is determined through a series of equations. The SDL Fineness and Maturity Tester (Montalvo, 2000) also measures the pressure drop across 4 grams of cotton at high and low air flow. In the Vibroscope method (ISO 2061-95), a tension is applied to the fiber till the fundamental frequency is found. As mentioned before, these methods were developed for the traditional fibers. Some of them are time-consuming or require specific training. As a type of coarse fiber, bagasse has limited access to the testing instruments used for cotton. Also, there is a lack

of methods and commercial instruments to determine the dimensions of bagasse and other unconventional fibers.

The present work focuses on finding an alternative method for measuring the length and evaluating the fineness of bagasse as well as other unconventional fibers. Image analysis becomes more and more popular as a simple and fast method of determination and prediction of fiber dimensions. Because of a direct relationship between diameter (for circular cross-section fibers) or cross-sectional area (for irregular cross-section fibers) and fineness, it is relatively easy to evaluate the fineness by measuring one of the above two cross-sectional parameters. The cross-section of bagasse fibers is irregular. Therefore, the fiber cross-section is measured in this study.

2.2 Experimental

2.2.1 Bagasse Extraction

Bagasse fiber used in this study was provided by a local sugar mill. The raw bagasse was already crushed at different lengths as an output from the sugar extraction process. Bagasse used in this study came from the 2001 crop. To extract the bagasse fibers, a series of mechanical and chemical procedures were used. Waste bagasse was first cleaned and treated with an alkaline solution. Then, the delignified bagasse was washed thoroughly and dried in an electric oven. The dried fiber was further mechanically cleaned using a cotton cleaner machine.

2.2.2 Visualizing and Measuring Procedures

To get a better resolution for the images, it was decided that each sample would contain seven fibers. The fibers were assigned randomly to the samples. To comply with the requirements for further statistical analysis, 24 samples were made. A Cambridge 260

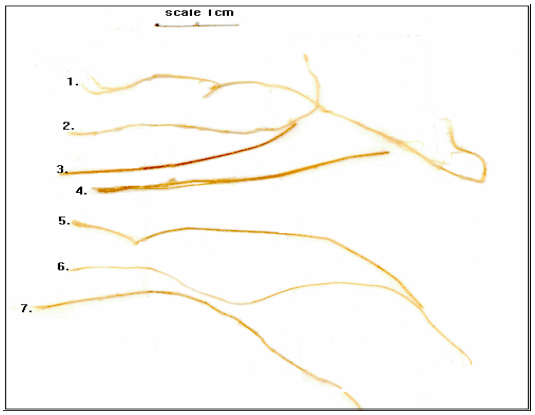
Stereoscan SEM, with a magnification in between 15 and 50,000 diameters, and a resolution of 10 nm - 29 nm, was used to visualize the cross-section of the bagasse fibers. The bagasse fibers were cut individually at 1mm length and glued with the cross-section up into stubs using "Spot-o-gold" labels. The cross-sections were coated with 25nm gold palladium using a Hummer II Sputter Coater.

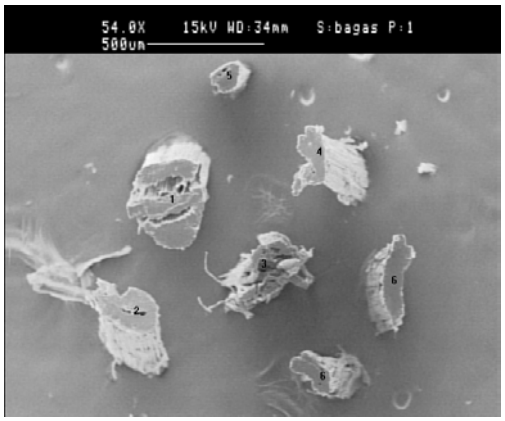
The measurements were actually performed using the Scion Image software for Windows 2000 that allows measuring the length, the selected area, the centroid, etc. (see Figure 2-1 b1-b3). Scion Image software is available free of charge from Scion Corporation. Scion Image may be used to capture, display, analyze, enhance, measure, annotate, and output images. Scion Image also has advanced capturing capabilities such as frame averaging and summation, frame sequence, and on-chip integration support. (www.scioncorp.com).

2.2.3 Determination of Fiber Length and Fineness

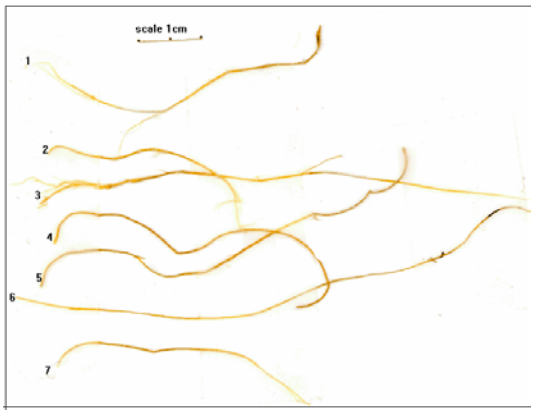
For visualizing the length aspect of the bagasse fibers, the digital imprint for each sample made out of the seven fibers was taken using an AGFA Duoscan T1200 scanner. (See Figure 2-1 a1-a3). The length was measured directly using the Scion Image software. The Scion Image allowed us to measure the length for each individual fiber by following the fiber imprint contour. For each sample composed of seven fibers, the software provided additive and statistical tools.

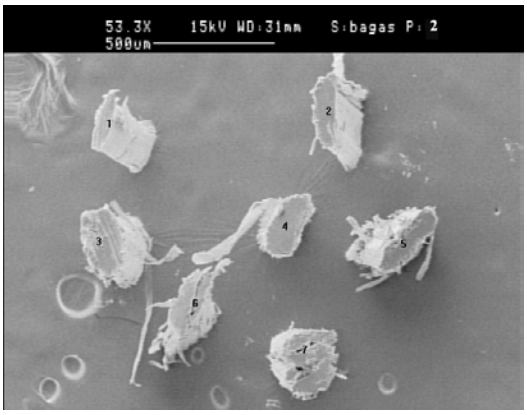
For determination of fiber fineness, a direct method was used. Each sample of seven fibers was weighed, and after that the ratio between the weight and the sum length for the seven fibers in each sample was recorded as the mean fineness (Table 2-1).



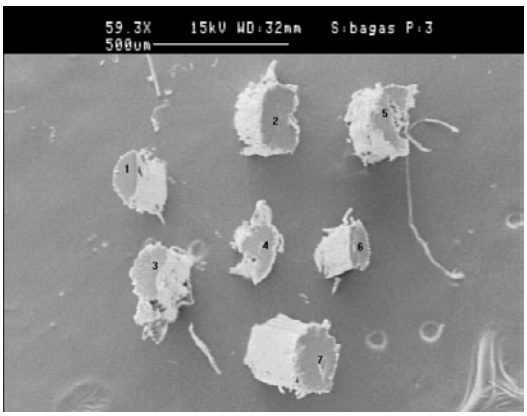


(a1) (b1)





(a2) (b2)



(a3) (b3)

Figure 2-1 Length (a1-a3) and cross-sectional (b1-b3) visualizations for 3 samples of bagasse fibers.

Table 2-1 Direct determinations for the Area and the corresponding Fineness.

Sample number	Mean Area (μm^2)	Mean Fineness (Tex)
1	21309.9	28.3
2	11629.9	18.7
3	30392.2	33.1
4	30404.7	34.1
5	27086.1	32.2
6	25423.9	26.8
7	17860.6	19.4
8	29559.6	31.6
9	22899.6	34.1
10	29512.1	36.4
11	22638.8	28.8
12	22473.5	35.7
13	20521.8	24.7
14	13465.6	21.0
15	14385.9	22.9
16	30173.8	38.8
17	30166.6	33.1
18	22083.6	33.9
19	31520.8	43.9
20	34285.7	47.8
21	28849.2	39.9
22	27701.1	35.7
23	23686.7	31.4
24	17315.7	28.6

2.2.4 Statistical Analysis

Because of the direct relationship between the cross-sectional area and the fineness, a method of regression analysis was considered to be appropriate. Each sample containing seven randomly selected fibers was weighed. Afterwards the length and the cross-sectional area were registered. Assuming that the density of the bagasse fibers is uniform, the fineness, as the dependent variable, can be determined and predicted from

the cross-sectional area, the independent variable. For conducting the statistical analysis, SAS 8.0 software was used.

2.3 Results and Discussion

Using a chemical and mechanical treatment, the bagasse fibers can be extracted from sugar cane crushed stalk. The delignification process helps break down the size of the bagasse fiber bundle. The length of the fibers is dictated by the parameters involved in sugar extraction and stalk crushing. The concentration of the sodium hydroxide (NaOH) used in the fiber extraction process can vary depending on the requirements for fineness and strength for the final applications of bagasse-based products.

Linear regression analysis determined the relationship between bagasse fiber fineness and cross-sectional area as an alternative measure for the diameter. Figure 2-2 shows the best-fit line that describes this correlationship. The coefficient of correlation (r^2) is 0.725. Considering the irregularity of cross-section for bagassse fibers, this coefficient of correlation indicates that a good dependency exists between the cross-sectional area and fineness. The regression model describing this dependency is:

Fineness (mTex) = 7507.3 + 0.992*Area (μm^2)

A "Lack of Fit" test proved that the linear model proposed above fits the data with the same power as a polynomial model. The prediction region was obtained using the formula: (Estimate) $\pm$ t*S$(1 + D)^{1/2}$ where S is the standard deviation (estimated by mean square error), t-value from a Student's t-distribution depending on the level of confidence and degrees of freedom, and D depending on each value of the regression line. As shown in Figure 2-2, a 95% prediction interval includes all the experimental data, giving a good confidence for prediction of bagasse fiber fineness.

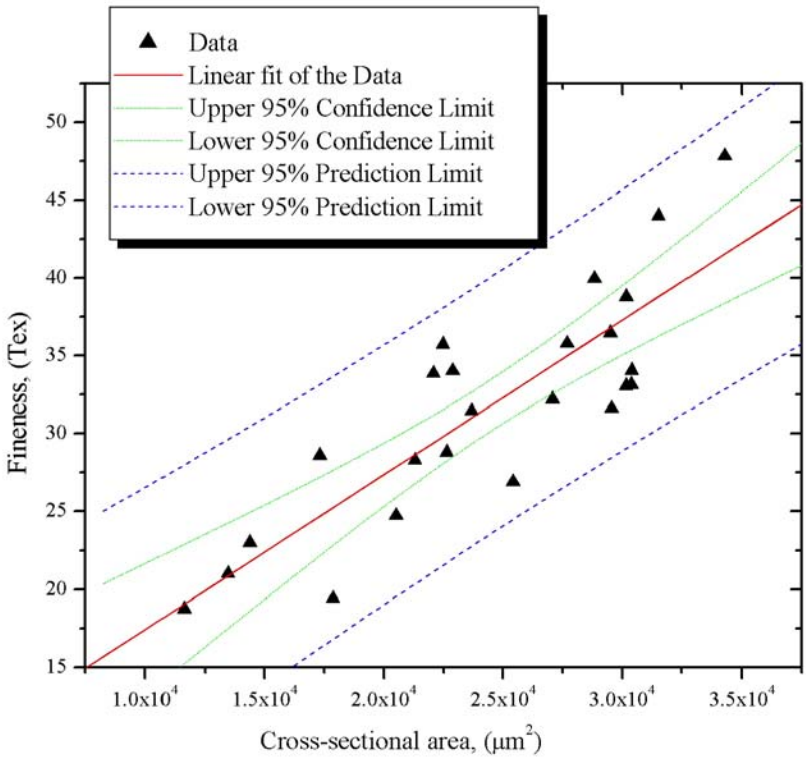


Figure 2-2. The dependency between the cross-sectional area and fineness.

The validity of the results of the statistical analysis requires fulfillment of certain assumptions about data. Specifically we assume that the linear model is appropriate, the residuals are independent and normally distributed with the same variance everywhere. A tool for detecting violations of assumptions is an analysis of the residuals. The plot of the residuals against the predictor variable is shown in Figure 2-3. The plot does not show any pattern which gives us confidence regarding the normal distribution of the data and therefore suggests that assumptions are fulfilled.

Image Analysis has proven to be a helpful and easy to use tool in determining the length and the cross-sectional area for those fibers without regular cross-section.

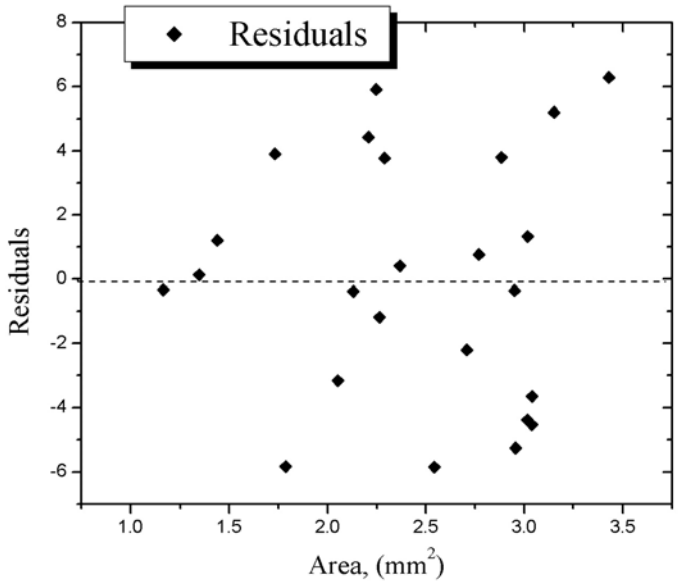


Figure 2-3. The residuals (error) distribution.

In particular, the equipment used for image acquisition is commonly used in academy and industry, and the image analysis software is free of charge. This assures the evaluating method for dimensions of unconventional fibers is cost-effective.

2.4 Conclusions

The extraction of bagasse fibers from sugar cane does not require a complicated technological process. Length of extracted bagasse fiber is imposed by the crushing process for the sugar cane stalk. Measurements of the bagasse fiber length can be accomplished directly using the Scion Image software. The magnification level used in order to capture a clear cross-section picture is around 50x. This suggests that a customary optical microscope can be used. The image method presented in this paper provides a simple approach for evaluation of bagasse fineness. Also, the model used in this method can be used conversely for determining the cross-sectional area when the fineness is known. This method can be extended for evaluating other non conventional

natural fibers, as well. The present work is still preliminary because of the limited number of bagasse fiber samples measured. Further research emphasis will be on development of a computerized image analysis system that can measure bagasse fiber length and cross-sectional area automatically.

Chapter 3 Manufacture and Analysis of Nonwoven Composites Based on Annual Plant Fibers and Polymers

3.1 Introduction

Since the introduction in the 1940s of nylon, the first manufactured synthetic fiber, synthetic fibers have had a significant impact on the quality of our lives and many consumer products (Paturau, 1989). However, the demand for natural fibers continues to increase because of their many outstanding properties, including aesthetics, comfort, and biodegradability. Farmers, fiber producers, and a diversity of scientists worldwide have been exploring the use of alternative fiber crops (kenaf, jute, and hemp), crop residues, and agricultural by-products, which are often underutilized and undervalued. For example, kenaf fibers are biodegradable, environmentally friendly, and inexpensive to grow; plus, they will grow almost anywhere and in any type of soil (Romanoschi et al, 1997). Jute also is a relatively cheap, easy-to-grow fiber having good mechanical properties. Flax and ramie, textile fibers used for long periods of time in different parts of the world, became cost-competitive because of new developments in the fiber extraction process.

Agricultural residues and by products from major U.S. Agricultural commodities (sugar cane, soybeans, wheat, corn, etc.) Potentially could be used to produce a multitude of value-added non-food products, ranging from fibers, films, plastics, and composites to resins, finishing agents, and auxiliaries (Collier & Arora, 1994). For example, sugar cane is an important agricultural crop in Louisiana, Texas and Florida. Florida is the largest producer of cane sugar in the u.s., with a crop value >50% of the total u.s. Crop value, followed by Louisiana with a crop value of approximate by 30% (Romanoschi et

al.,1997). The crushed stalks (bagasse), remaining from traditional sugar cane processing, are used mainly for low-value applications. For example, most of the bagasse is used in-house as a fuel in mill processes and for other low-value applications such as mulch and inexpensive ceiling tiles (Paturau, 1989). The development of value-added products from waste bagasse could allow mills to migrate to cleaner burning fuels and provide economic benefits as cane producers compete in a freer trade environment. To this aim, researchers in Louisiana have developed processes for converting sugar cane rind into nonwoven textile and geotextile products (Collier et al., 1992) .

The extent of nonwoven applications increased dramatically in the last decades. Because of the low production cost in manufacturing, explained by the fact that some of the regular steps in the textile process, like yarn spinning, can be skipped, nonwovens started to be widely used in almost all the aspects of the industry (Thames et al, 1994). Nonwovens can be found nowadays in automotive manufacturing, building construction, medical applications, petroleum industry (as sorbents), and civil engineering. There are two important steps in nonwovens manufacturing that influence the characteristics of the final product. First is the preparation of the fiber web and the second one is the bonding of the fibers in the web. In addition a main influence is given by the composition of the nonwoven. It is customary to blend two or more fibers in order to improve the final characteristics. In the web bonding step, the presence of fine fibers, as synthetic, will assure a better bonding, influencing positively the mechanical properties of tensile strength and flexural rigidity. Also, in the thermal bonding step the synthetic fibers will melt, gluing together the natural fibers, in the desired shape. This is specifically important in manufacturing boards and side panels for the automotive industry.

The physical and mechanical characteristics of the natural fibers will also influence the final product. Several natural fibers – bagasse, kenaf, ramie, jute and flax - have been considered in the present investigation, along with only one synthetic polymer – polypropylene - to be employed for manufacturing of composite nonwovens. The preparation of nonwovens, as well as their most significant thermal and mechanical properties will be presented in the following.

3.2 Experimental

3.2.1 Materials

The composite nonwovens, all having a sandwich structure, were designed with the following fibers and fiber-blending ratios:

a) 35/35/30 kenaf/recycled polyester [shoddy poly(ethylene terephthalate), PET]/off-quality polypropylene (denoted as PP)

b) 50/50 polypropylene/jute (denoted as PP/jute)

c) 50/50 polyplropylene/flax (denoted as PP/flax)

d) 100 % polypropylene (denoted as PP) - control.

Virgin polypropylene (denoted as P) has been used for another series of sandwich type samples containing three layers as follows:

e) The outer layers composed of 70/30 kenaf/polypropylene (denoted as KP) with the neutral layer made of 50/50 bagasse/polypropylene (denoted as BP). The whole nonwoven composite structure was denoted as KP-BP.

f) The outer layers composed of 70/30 ramie/polypropylene (denoted as RP) and the neutral layer made of 70/30 kenaf/polypropylene (denoted as KP). The whole nonwoven composite structure was denoted as RP-KP.

The Reason For Including Recycled Polyester (Shoddy) And Off-Quality Polypropylene (Pp) In The Various Blends Was To Lower The Fabrication Cost And To Impart Thermal Formability To The Ultimate Nonwoven Composition.

3.2.2 Processing Procedures

Jute, kenaf, and PP fibers were opened in an Uster Spinlab Fiber Opener/Blender and blended as required. The blended fibers were passed through the Fiber Opener/Blender again to improve intimacy of the blend, which then was used to produce a carded web for needle-punching. The webs were needle punched (twice and four times) on spun bonded polyester scrim to produce mid-to-heavy weight nonwoven fabrics of different weights (620 and 950 g/m^2, or 20 and 30 oz/yd^2). With the air-laid system, the fibers were used without passing them through the Fiber Opener/Blender. Approximately 3000 g of fibers of desired blend composition were tumbled in the hopper of a Rando Feeder-Webber for one hour. Air-laid batts (45 cm wide) were formed at a speed of 0.3 meter/minute on a Rando machine using the standard settings and speeds. The feeder fan damper was set open and the Webber fan was set 73 degrees towards the closed side. The batts produced were longitudinally cut in half, compressed by rolling under a tray, and run twice through a Morrison-Berkshire needle-punching machine with the following settings: width of 25 cm; needle board width of 1.8 needles/cm (Groz-Beckert needles, 15x18x40x3); 228 operating cycles/minute; and 46.5 penetrations/cm^2.

Waste bagasse was manually sifted and boiled in an alkaline solution (2.0N NaOH) in order to remove the lignin. Subsequent to this treatment the fibers were rinsed with water and dried in an air-ventilated electric oven. The extracted fibers were thereafter cleaned using a cleaning Mcpherson machine at USDA southern regional

center (SRRC) in New Orleans. The natural fibers were then blended with polypropylene fibers in the desired ratio and fed into a F015d universal carding machine to obtain a fiber web. To enhance the uniformity of the web, each fiber blend was carded twice. A Morrison Benkshire needle-punching machine was used also for the mechanical bonding of the bagasse/polypropylene, kenaf/polypropylene, and ramie/polypropylene webs using a feeding speed of 5.4 feet/min and a punching rate of 228 strokes/min. Thermal bonding was realized using a carver laboratory press with the temperature of plates set for 170°c. Pressing time was 5 minutes and the thickness controlled by spacers (1/8"). The KP-BP composite material had a post-pressing weight of 983 g/m^2. The post-pressing weight of the RP-KP nonwoven composite was 1099 g/m^2.

3.3 Testing Procedures

3.3.1 Mechanical Determinations

Tensile strength for the nonwoven composites was measured according to the ASTM D5035-95. The testing machine used was an Instron tester Model 4301. Flexibility of the nonwoven composites was tested in accordance with the ASTM 790-97 for flexural properties of plastics (three-point bending method).

3.3.2 Thermal Analysis

Thermo gravimetric analysis (TGA) of the composite nonwoven and its components was carried out both in air and inert atmosphere (nitrogen) with a rate of 5°C/min using a TA thermobalance. Glass transition (T_g), softening (T_{soft}) and melting (T_{melt}) of polyester were determined by differential scanning calorimetry (DSC) with a rate of 5°C/min using a TA DSC instrument.

3.3.3 Dynamic Mechanical Analysis

Dynamic mechanical properties were determined in bending mode using a Seiko Dynamo-Mechanical Spectrometer DMS 110. A train of frequencies (0.01, 0.1, 1.0, 10.0, 50.0 Hz.) was employed using a heating rate of 0.5°C/min.

3.4 Results and Discussion

3.4.1 Thermal Transitions by DSC

This technique allowed the determination of processes involving a heat exchange with the sample, such as melting and drying. Melting was an endothermic effect peculiar to synthetic materials, such as polypropylene and polyesters. Drying referred to cellulosic components because the water absorption of polymers is very small (<1.0%).

Figure 3-1 presents the results for kenaf, jute and flax nonwoven composites containing synthetic polymers. Drying of cellulosics is well represented as a large endothermic transition with a maximum at around 60ºC. Melting of PP in all samples has a peak at 163.6°C. PET is present only in the DSC thermogram of kenaf/PP/PET nonwoven with a melting peak at 256.6°C.

3.4.2 Thermo Gravimetric Analysis

The content of water in each nonwoven can be quantified from thermo gravimetric data presented in Figure 3-2. The weight loss levels after 150°C and corresponds to the weight of water lost by evaporation.

Drying and thermal degradation of kenaf/PP/PET nonwovens are shown in Figure 3-3. The overlapping of degradation plots for PET and PP components is resolved in Figures 3-4 and 3-5 by 1st and 2nd derivatives of the thermo gravimetric curve. Thermo-gravimetric curves corresponding to the weight loss by drying, as well as to thermal

decomposition of RP-KP and KP-BP nonwoven composites are plotted in Figure 3-6 and Figure 3-7, respectively.

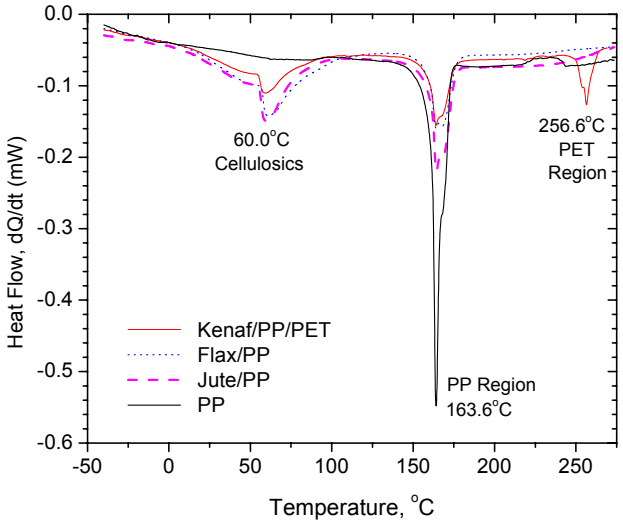


Figure 3-1. DSC thermograms of nonwoven composites and PP

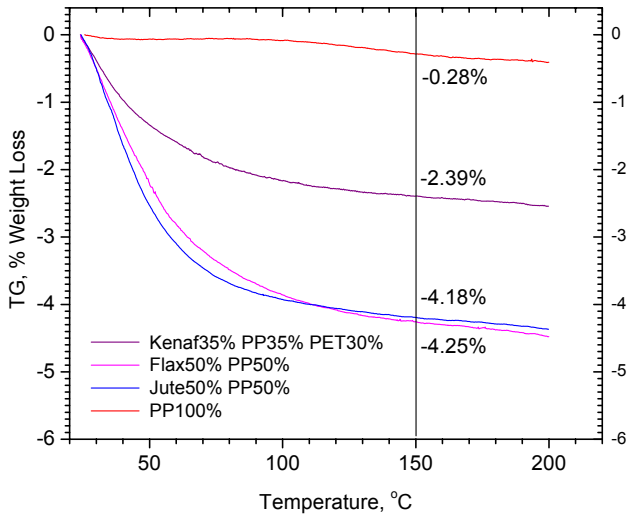


Figure 3-2. TG curves corresponding to weight loss by drying of nonwoven composites and PP

The rather significant content of absorbed water is revealed by the weight at 115°C of dried samples. A higher rate of degradation of cellulosics, as reflected by DTG curves, was recorded in the case of RP-KP sample. However, the degradation of synthetic polymer component is more visible in KP-BP sample. Thermal degradation in air occurred at much lower temperatures, with a total loss of mass at 480°C, as compared to the sample decomposed in inert atmosphere (Figure 3-8), which did not undergo a total decomposition even at 600°C.

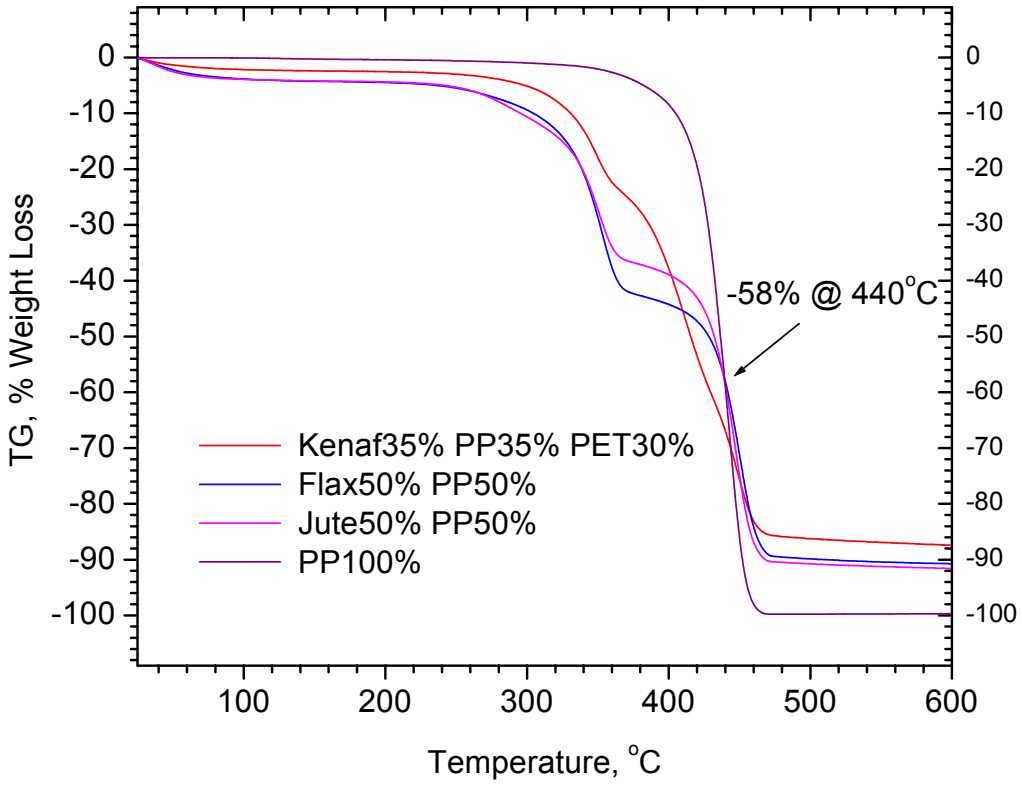


Figure 3-3. TG curves corresponding to weight loss by drying and thermal decomposition of kenaf, flax and jute nonwoven composites and PP.

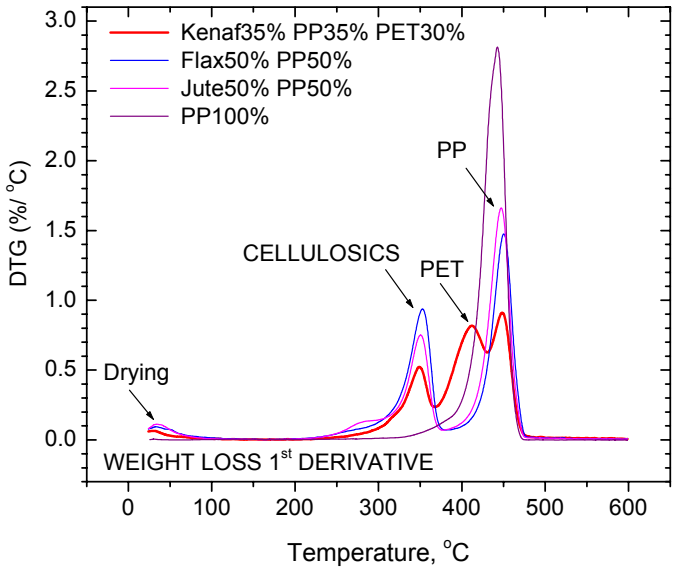


Figure 3-4. First derivative of thermo gravimetric (TG) curves of kenaf, flax and jute nonwoven composites showing the peaks corresponding to degradation of cellulosics and individual synthetic polymers

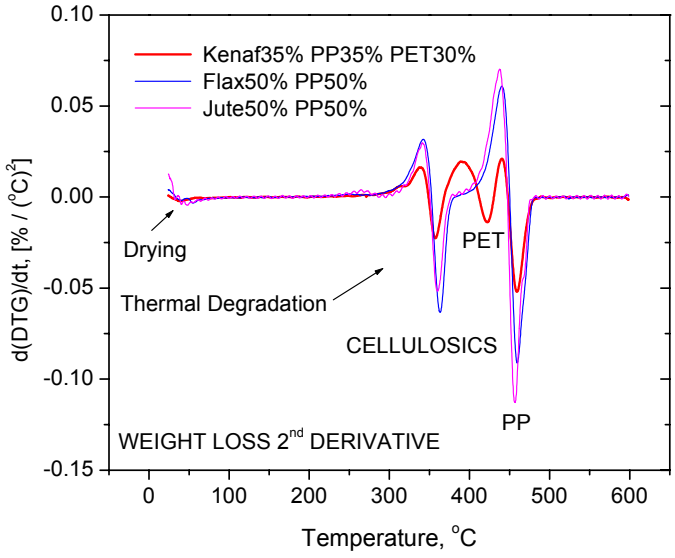


Figure 3-5. Second derivative of thermo gravimetric (TG) curves of kenaf, flax and jute nonwoven composites showing the peaks corresponding to degradation of cellulosics and individual synthetic polymers.

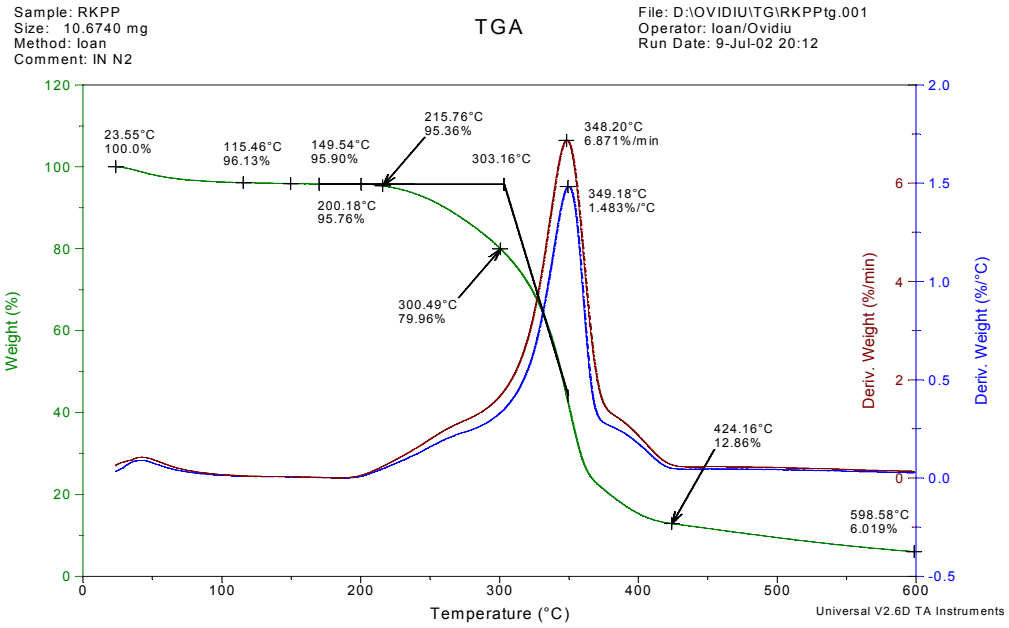


Figure 3-6. TG curves corresponding to weight loss by drying and thermal decomposition of ramie/kenaf/PP nonwoven composite

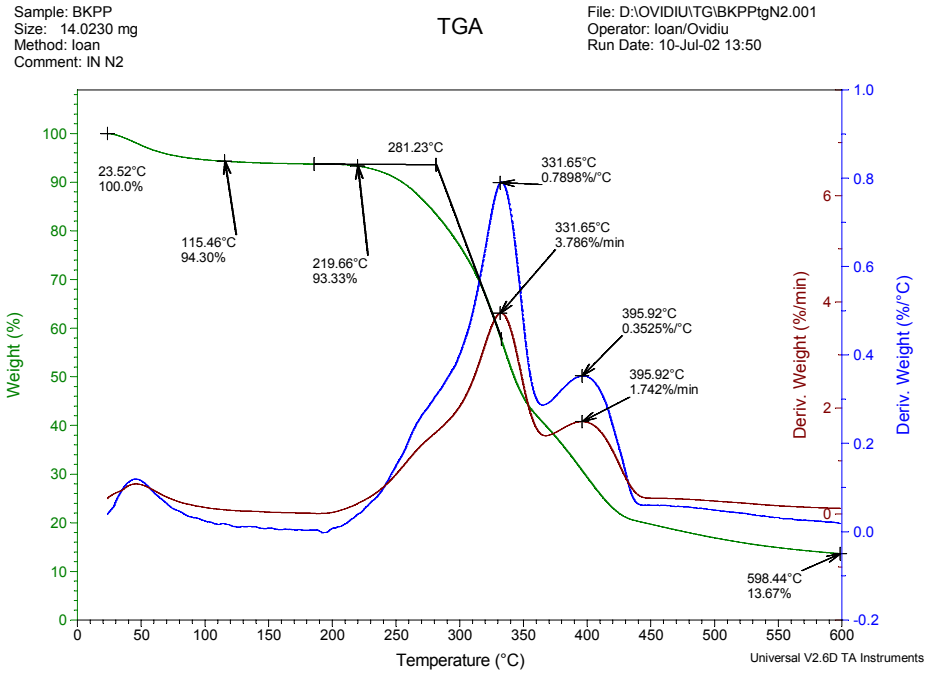


Figure 3-7. TG curves corresponding to weight loss by drying and thermal decomposition of Bagasse/Kenaf/PP nonwoven composite.

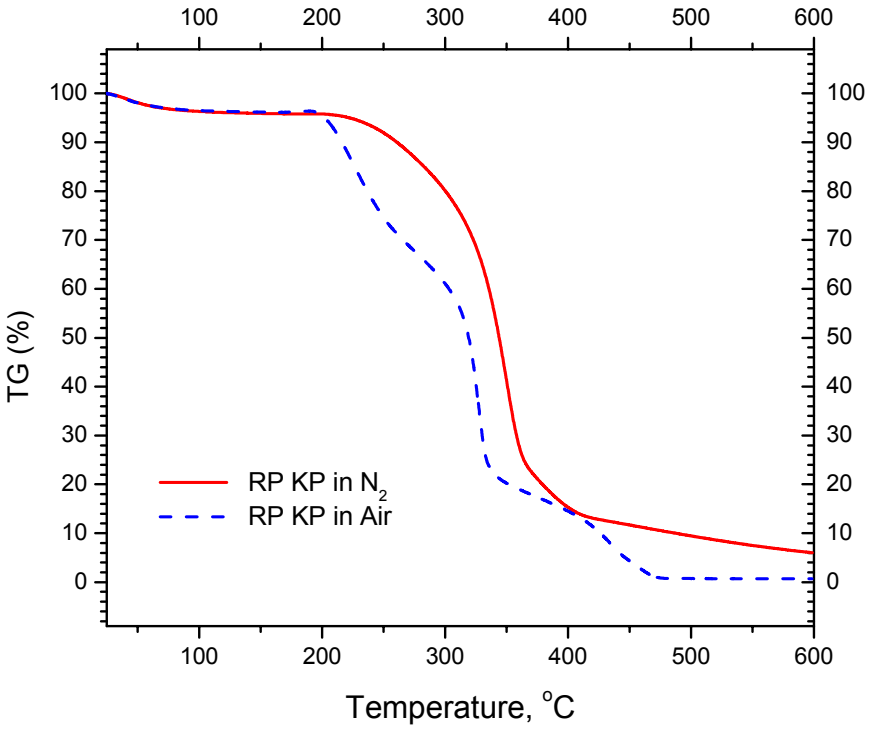


Figure 3-8. TG curves corresponding to weight loss by drying and thermal decomposition of Ramie/Kenaf/PP nonwoven composite samples in air and inert atmosphere (N_2)

3.4.3 Dynamo-Mechanical Analysis

Drying of cellulosic components affected in a visible way the visco elastic characteristics of samples. Both the loss modulus, E", and its ratio to the storage modulus, E', plotted as tanδ = E"/E', pointed to distinct drying (of cellulosics) and melting (of the synthetic polymer) regions (Figures 3-9, 3-10, and 3-11). The elastic component, E', decreased rapidly during drying, with a sudden drop after softening and melting of polypropylene (Figures 3-11 and 3-12). The higher the frequency used, the higher the temperature of transition (Figure 3-12).

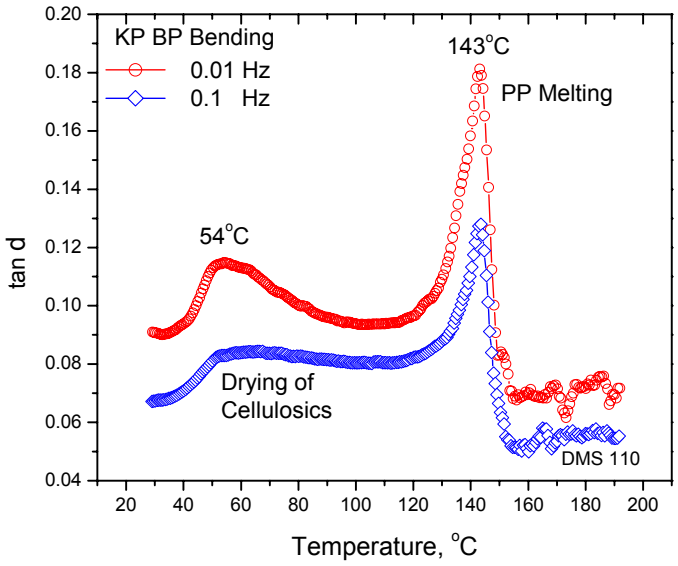


Figure 3-9. DMA of KP BP nonwoven in bending mode. Dependence of tanδ upon the temperature

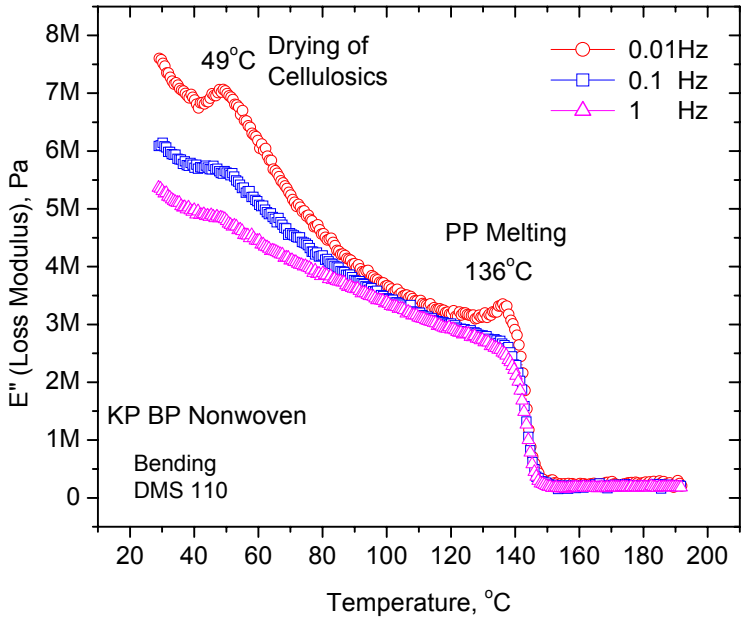


Figure 3-10. DMA of KP BP nonwoven in bending mode. Dependence of the viscous component E" upon the temperature

It seems that due to the rapid decay of elastic properties of nonwoven samples upon the softening of the polypropylene component, it is this temperature (around 125°C, depending upon the frequency considered) that limits the exploitation of these nonwoven composites at higher temperatures.

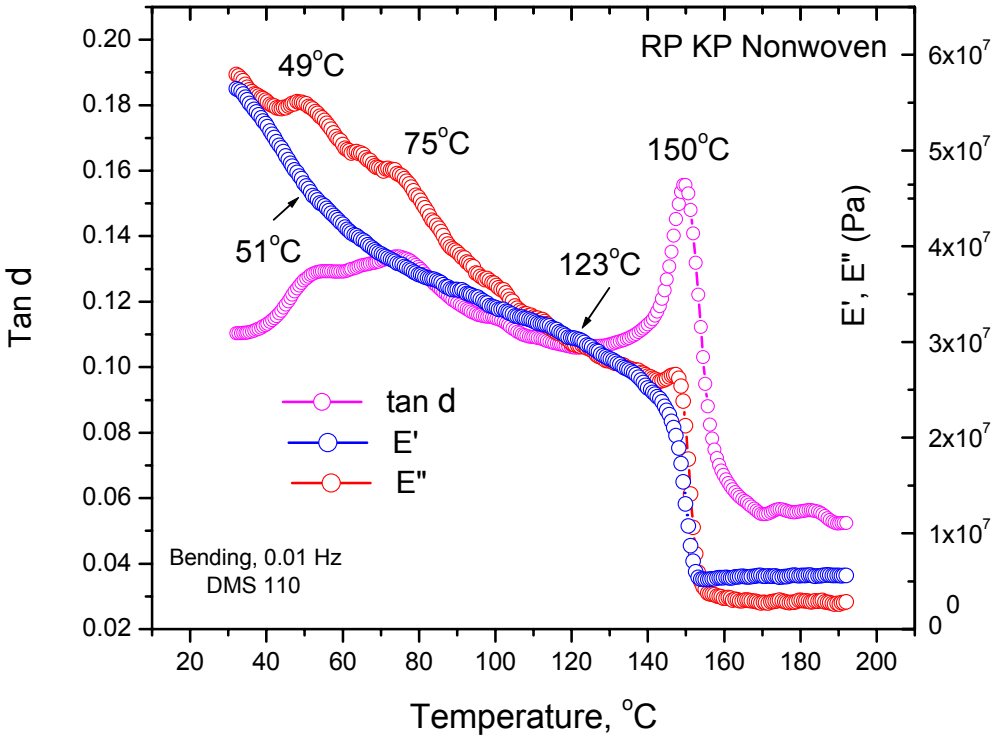


Figure 3-11. DMA of RP KP nonwoven in bending mode

3.4.4 Mechanical Properties

Table 3-1 lists the strength, the elongation at break and the modulus of elasticity of nonwoven composites samples prepared in this work. The sandwich structure did not show a significant improvement in the tensile strength. More important to having a higher tensile strength seemed to be the fiber composition, its length, and the technique used for the web preparation. Flax blended with polypropylene experienced the highest modulus while jute blended with polypropylene had the lowest modulus.

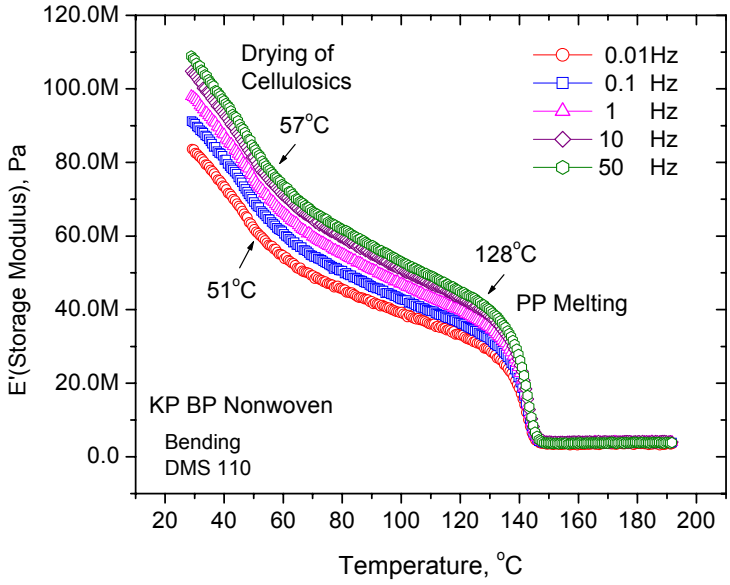


Figure 3-12. DMA of KP BP nonwoven in bending mode. Dependence of the elastic component E' upon the temperature.

Table 3-1. Tensile properties of nonwoven composite materials.

Materials	Max Stress (psi)	Max Strain (in/in)	Modulus (psi)
PP/Flax	313.4	0.58	1017.0
Kenaf/Polyester/Polypropylene	182.1	1.180	277.2
RP-KP	39.9	0.75	78.5
KP-BP	24.2	0.90	45.4
PP/Jute	42.1	2.11	23.9
PP	55.8	3.17	17.7

3.5 Conclusions

Viscoelastic nonwoven composites were successfully prepared from annual plant fibers and synthetic polymers. Thermal properties of nonwoven composites were related to drying at lower temperatures (T<100°C) and to softening and melting of polymers at T>120°C. Elastic properties of KP BP and RP KP decayed very rapidly after softening and melting of polypropylene. The tensile strength was related to the nonwoven composition and construction mode.

Chapter 4 Biodegradable Nonwoven Materials

4.1 Introduction

Cotton has been considered the principal fiber used in apparel and home fabrics all around the world. Despite the last years' stagnant productivity of cotton fields explained by bad weather and pests damage, the overall cotton production is expected to increase in the U.S. due to the larger areas cultivated. New markets are coming to the attention of cotton producers. There is a strong need to promote use of cotton in nonwoven manufacturing. Several research projects identified and evaluated the consumers' preferences and perceptions of fiber content in nonwoven products. A significantly higher percentage of respondents would prefer to buy nonwoven personal care products that are made from natural fibers (Messura, 2002). Although the size of the nonwoven cotton market is small, this industry is one of the few that has managed to hold its own. This may be due, in part, to the very favorable price cotton merchants have seen recently, and, in part, to new and potentially significant markets for cotton nonwovens - the market for cotton composites made from griege cotton fibers and synthetics is quite promising generally in the insulation market, and particularly in the automobile insulation market (Calamari, 2002).

In the same time recent research in the US agriculture and forestry industry has as its object of investigating new uses and adding value to farm and forestry products for greater economic benefits. From this point of view, sugar cane bagasse is established as the future fiber in tropic and subtropical regions for textile production for industrial applications. Many by-products are available from the sugar cane industry, but the most important is bagasse (Paturau, 1989). The sugar cane bagasse is the result of the

production process of the sugar in which the cane is crushed in small pieces and the juice is squeezed out. The sugar cane stalk is composed of an outer rind and an inner pith. The pith contains small fibers and the majority of the sucrose, while the rind contains longer and finer fibers arranged randomly throughout the stem and bound together by lignin and hemicellulose (Collier, 1995). The crushed and squeezed cane stalks are known as bagasse. Currently sugar cane is the most valuable agricultural crop produced in Louisiana. Last year the gross farm value of sugar cane production was nearly $378 million. An additional $242 million in value was added by processing and marketing sugar. Excluding forest products, sugar cane production accounted for 26% of the total value of all agricultural plant commodities sold in 2001 (*Louisiana Agricultural Summary* 2001). Sugar cane bagasse is still considered by many researchers an unconventional fiber with a very limited range of applicability in textile industry. Not only bagasse, but also other so-called unconventional fibers such as kenaf and coconut fiber found grounds for development in nonwoven textiles.

Almost all the industrial sectors benefit from using nonwovens. Nonwoven materials have properties that make them suitable for diverse end-use applications, e.g., insulating or sound-deadening materials, sorbents, and geotextiles. The yarn sequence in the fabrication process of nonwoven materials is eliminated, resulting in reducing the time of production, increasing the productivity, and implicitly lowering the cost. The steps that have to be taken in the nonwoven manufacturing process are lap formation (air laid, wet laid or spun laid), and web bonding that can be accomplished by chemical, mechanical or thermal means.

Thermal bonded nonwovens are now in "fashion" because of favorable process economics, the absence of chemical binders, the availability of new fibers and machinery, and process and product enhancement. The adhesive component, subjected to heat, melts on contact areas with more stable fibers to form potential bonding sites. The adhesive becomes attached to a network fiber, solidifies, and forms a bond, or a thermal fusion, at each fiber-binder contact (Bhat, 2002). Several requirements have to be met by the nonwoven composite materials in order to be used particularly for agriculture applications. These are: air permeability, heat transfer, flexibility (or flexural rigidity), and last but not least, biodegradability. In the past of cotton-based nonwoven research, synthetic fibers such as polypropylene, polyethylene, and low-melting polyester were employed for binding. However, the synthetic binder fibers were not biodegradable (Bhat, 2002).

Consequently, natural fibers are nowadays increasingly employed for making nonwovens, replacing the synthetic materials due to economic and/or environmental considerations. One of the limitations of the extensive use of natural fibers in nonwovens is their low strength if the web is not bonded with a screen of synthetic fibers. Recent research tried to combine the biodegradation requirement and the structural strength of nonwoven webs by using biodegradable synthetic polymers, such as biodegradable polyesters. In line with the idea of adding value to bagasse and the use of natural fibers (cotton) and biodegradable synthetic polymers, the present work describes the preparation and certain properties of biodegradable composite nonwovens made of bagasse, cotton and melt-blown mats of poly(tetramethylene adipate-co-terephthalate).

4.2 Materials and Methods

4.2.1 Cellulosic Fibers

Cotton fibers used were greige Maxxa type (provided by USDA ARS Southern Regional Research Center, New Orleans, LA). Bagasse fibers were obtained as described in the following; selected crushed and squeezed cane stalks from a Louisiana sugar mill were boiled for one hour in a 2.0N NaOH solution (the ratio used was 1:20 weight/volume). After washing thoroughly with water the delignified bagasse fibers were dried in an air-circulating Blue M Electric Company Oven at 135 °C for 45 minutes. The cleaning process was carried out at SRRC using a Cleaning McPhearson Machine.

4.2.2 Bonding Synthetic Polymer

The polymer used as an adhesive for binding the cellulosic webs was Eastman poly(tetramethylene adipate-co-terephthalate), referred also as Eastar biocopolymer (EBC). It is biodegradable random copolyester obtained from adipic acid, terephthalic acid and butanediol:

$$x\text{HOOC-}(CH_2)_4\text{-COOH} + y\text{HOOC-}C_6H_4\text{-COOH} + z\text{HO-}(CH_2)_4\text{-OH} \Rightarrow\Rightarrow \text{EBC}$$

Polyester pellets received from manufacturer (Eastman Chemical Co.) were transformed in a melt blown nonwoven fabric (57 g/m^2) at the Textile and Nonwovens Development Center (TANDEC) unit from the University of Tennessee, Knoxville, TN.

4.2.3 Carding and Needle-Punching

Cotton fibers were blended manually with bagasse and fed twice into a F015D Universal Laboratory Carding Machine. For the web bonding a needle punching process was employed using a Morisson Berkshire needle-punching machine with a speed of 5.4

feet/min and a corresponding 228 strokes/min. Each sample was needle-punched two times faced up and down.

4.2.4 Thermal-Bonding

To obtain the nonwoven composites shown in Figure 4-1, the cellulosic webs and the melt blown polyester nonwoven sheets (in a 70:30 weight ratio) were sandwiched and pressed into a flexible bonded sheet for one minute at 150°C and 8.62 Pa (12,500 psi) using a Carver Laboratory Press with heated plates. Three samples were considered for the present investigation (Table 4-1). The designed composition was 70(70/30 Bagasse/Cotton)/30 EBC. The actual bagasse content was about 54%.

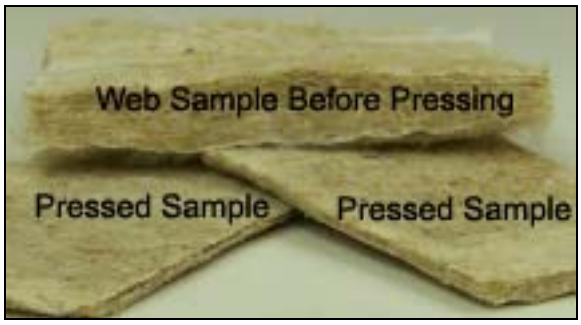


Figure 4-1. Layered composite nonwovens made of bagasse/cotton webs and EBC melt blown nonwovens before and after hot-pressing.

4.2.5 Thermal Analysis

Thermo gravimetric analysis (TGA) of the composite nonwoven and its components was carried out both in air and inert atmosphere (nitrogen) with a rate of 5°C/min using a TA thermobalance. Glass transition (T_g), softening (T_{soft}) and melting (T_{melt}) of EBC polyester were determined by differential scanning calorimetry (DSC) with a rate of 5°C/min using a TA DSC instrument.

Table 4-1. Construction characteristics of nonwoven bagasse/cotton/EBC samples

Sample	Thickness (in)	Size (in)	Number of Layers	Weight (grams)	Surface density (g/m^2)	Volume density (g/cm^3)
Sample#1	0.5	10x10	12	390	6045	0.762
Sample#2	0.25	9.8x10	6	213	3430	0.792
Sample#3	0.25	10x10	6	201	3115	0.803

4.2.6 Dynamic Mechanical Analysis.

Dynamic mechanical analysis (DMA) of nonwoven materials was performed in bending mode for rigid samples (Seiko dynamo-mechanical spectrometer DMS 110) and in stretching mode for soft EBC samples (Seiko DMS 200) in a large temperature range (-100°C to 200°C) using different frequencies (0.1Hz to 100 Hz).

4.2.7 Measurement of Thermal Conductivity and Thermal Transmission.

Thermal conductivity and thermal transmittance tests were performed using a thermal conductivity meter (FOX 200, LaserComp Corporation) according to ASTM D1518- 85 standard procedure. Measurement of thermal conductivity and transmittance of poorly conductive materials is based on the simultaneous measurement of ΔT (variation of temperature) across the sample and of the heat flux through the sample. The thickness of the sample was determined according to the standard procedure ASTM D1777-85. Each sample (8 in x 8 in) was conditioned at 20°C and 65 % RH for at least 24 hours, and placed between two plates, referred as "cold" [T_{cp} = 21.0°C (69.9°F)] and "hot" [T_{hp} =36.6°C (97.9°F)], respectively. The average of three samples with triple

measurement for each sample was used to calculate the mean values of the thermal conductivity and thermal transmittance for each nonwoven specimen.

4.2.8 Biodegradation of Composite Nonwovens

Duplicate samples were soil-buried for 1, 2, 3, 4, 5 and 6 weeks and analyzed thereafter for loss of mechanical strength.

4.3 Results and Discussion

Thermal stability (in nitrogen atmosphere) of the composite nonwoven is dictated by the content of the cellulosic fibers, i.e., it starts to loose weight at the same temperature as the bagasse/cotton web (220°C, Figure 4-2). Up to this temperature, the TG curves reflect the loss of water, viz., 0.2% for polyester, 4.3% for bagasse/cotton web and 2.2% for the composite nonwoven. However, a comparison of E' (storage modulus) curves plotted in Figures 4-3 and 4-4 shows that the viscoelastic character of the nonwoven composite is dictated by the bonding polymer up to the temperature at which the polymer starts to soften and eventually melts. Softening and melting temperatures of EBC polyester were determined from DSC data (Figure 4-5). The dependence of the storage modulus upon the temperature is similar for the polyester and for the nonwoven composite both at low and high temperatures. The elasticity as reflected by E' drops dramatically after T_{soft} (70°-100°C, Figures 4-3 and 4-4), but the nonwoven composite keeps its integrity with a high elastic contribution (E' > 10E+08 Pa) even after the polymer softens and melts to a very viscous material (T > 100°C). The values of the storage modulus do not change significantly with frequency for the three orders of magnitude used (0.1Hz to 100 Hz).

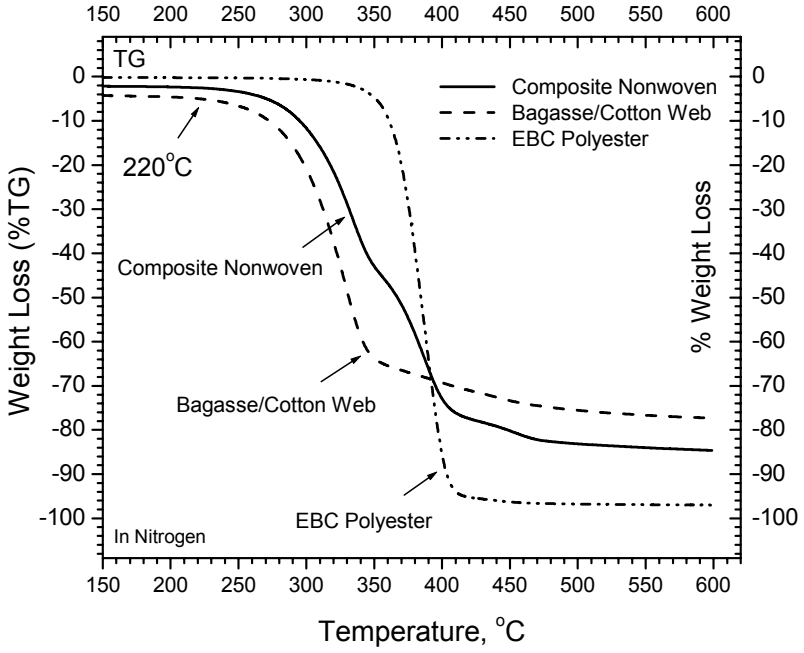


Figure 4-2. Thermal degradation of nonwoven materials in inert atmosphere (nitrogen)

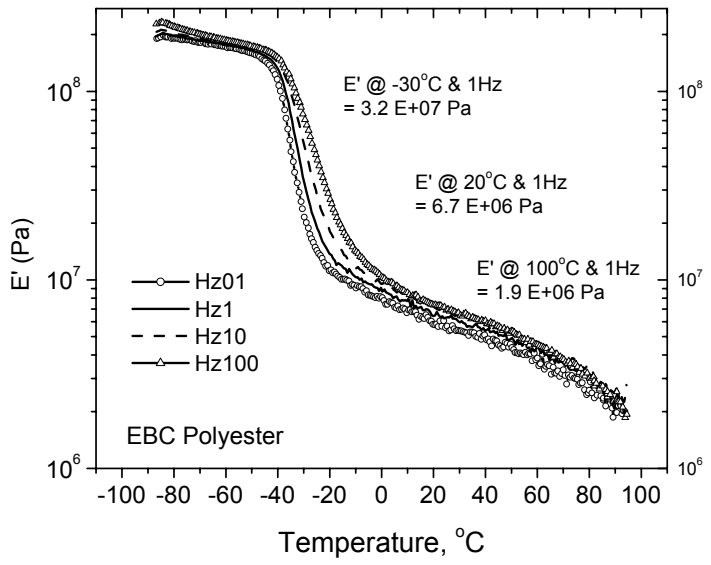


Figure 4-3. Dependence of E' of the EBC upon temperature and frequency.

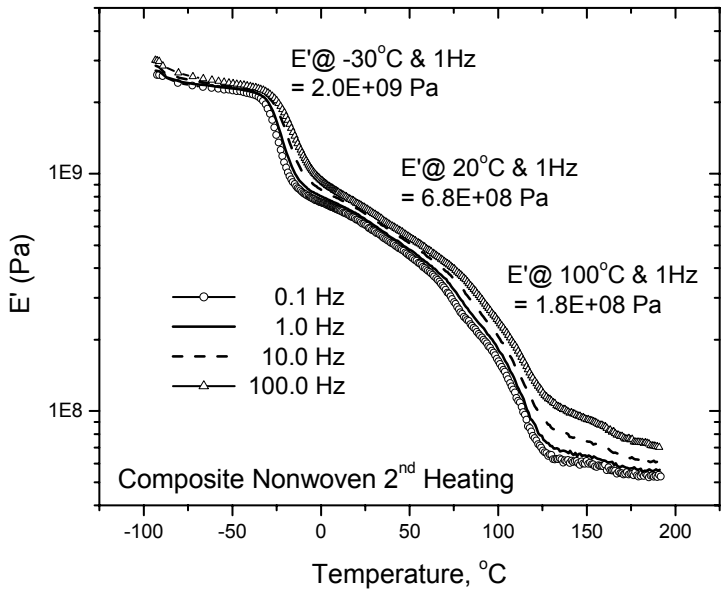


Figure 4-4. Dependence of E' of the nonwoven composite upon temperature and frequency

Both the loss modulus, E", describing the viscous character of the material, and the tangent of the loss angle (tanδ = E"/E') exhibited a significant shift with the frequency used, viz., the higher the frequency, the higher the temperature of the transition, such as the glass transition, T_g. The EBC polyester is a random copolymer of low crystallinity and therefore it has a rather large mobility of chain segments, exhibiting an evident T_g. The glass transition of polymers can be conveniently determined by DSC or by DMA measurements. If determined by DMA, T_g can be expressed as the maximum of the E" or of the tanδ peak, respectively. It can be also read at the middle of the inflexion of the E' curve, similarly to the determination of T_g by DSC analysis (Mathot, 1994). The T_g value expressed as the E" peak temperature is usually with few degrees higher than that expressed by tanδ peak. As a rather standard expression, the glass

transition of materials is given by the temperature of the E" peak determined at 1Hz. Figures 4-5, 4-6, and 4-7 present the glass transition temperature of the EBC polyester from a series of DSC and DMA measurements. It occurs, depending on frequency, in a rather large temperature range (Figure 4-6). At the same time, the E' curves exhibit a sudden drop right after the setting of the glass transition temperature (Figures 4-3 and 4-4). If determined by DSC – a static method - T_g it is close to that given by the E" peak at low frequencies, viz., <1Hz (Figure 4-5). However, from the same figure it may be seen that the glass transition of the pure EBC polyester given by E" at 1 Hz (-32°C) is much lower than that determined in the same way for the nonwoven composite (-12°C). This difference points to some kind of interaction between the polyester and the cellulosic components of the nonwoven, set perhaps during the hot pressing, that hinders the movement of polyester chain segments at low temperatures. An interaction between the EBC polymer and cellulosic components has been pointed out previously from differences seen in the thermal degradation of these materials. Practically the DMA measurements show only the transitions due to the EBC polymer (Figure 4-7).

The glass transition of cellulosic components might be barely seen in the dependence of E' upon the temperature at high frequencies (Figure 4-8) above the softening and melting region of the synthetic polyester (> 120°C). Even if the bagasse/cotton/EBC nonwovens do not start to decompose until the degradation temperature of the cellulosic components is reached (220°C), the exploitation temperature is limited to 70-100°C by softening and melting of the synthetic polymer.

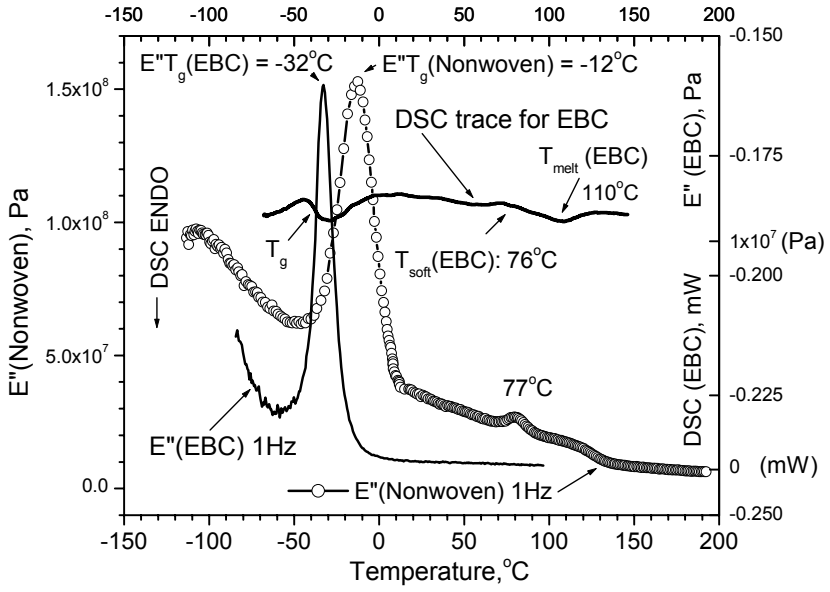


Figure 4-5. Glass transition, melting and softening of the EBC polyester as reflected by DSC and DMA (E") data for the bonding polymer (EBC) and for the EBC bonded nonwoven.

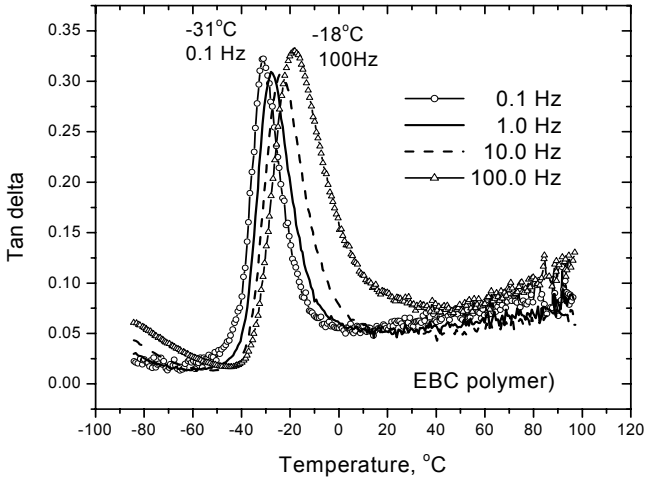


Figure 4-6. Dependence of the EBC glass transition upon frequency.

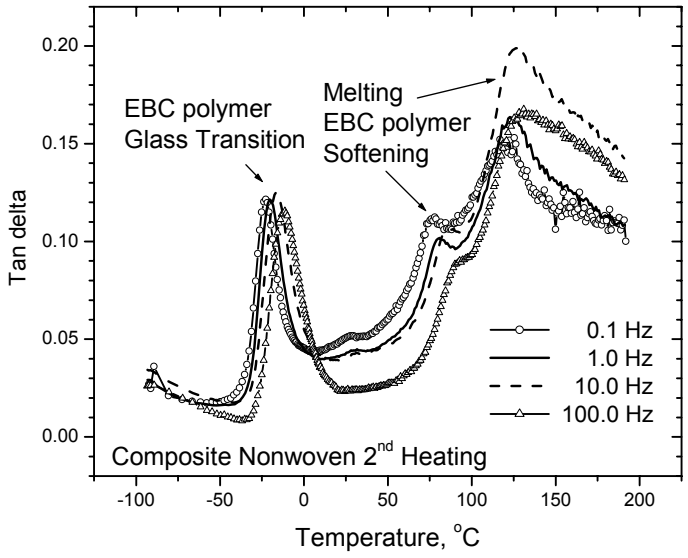


Figure 4-7. Thermal transitions of EBC polyester in composite nonwovens

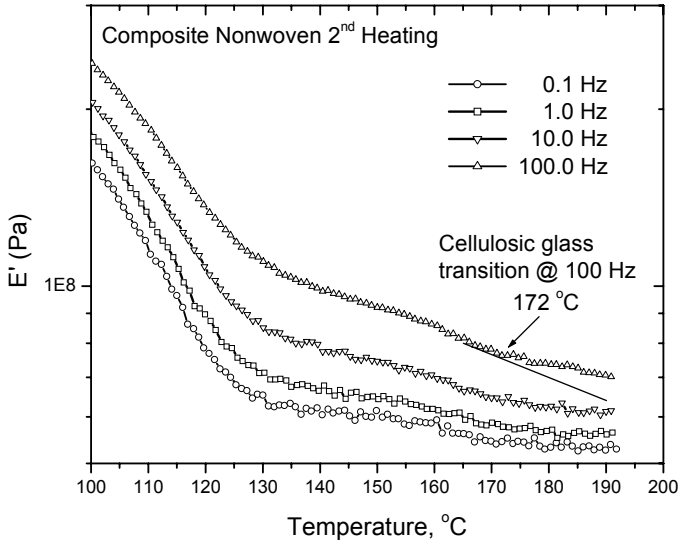


Figure 4-8. Blow-up of Figure 4 at higher temperatures: glass transition of cellulosic chains.

The thermal conductivity, λ (viz., the time rate of unidirectional heat transfer per unit area, in the steady-state, between parallel planes separated by unit distance, per unit difference of temperature of the planes), of bagasse/cotton/EBC composite nonwovens and its value normalized by density, λ/d, are very close, regardless of the construction of the nonwoven, i.e., the number of layers (Table 4-2). Slight differences seen after normalization versus the density are due to the heterogeneous volumetric distribution of components and air spacing. Keeping the same composition, thermal conductivity can be controlled by nonwoven density that can be adjusted by the distance between the two plates during the hot pressing, while the heat transmittance can be controlled by density and construction.

Table 4-2. Thermal conductivity data for bagasse/cotton/EBC composite nonwovens

Sample	**d density g/cm^3**	**Temp. ^{o}C**	**λ* W/ m ·K**	**λ/d****	**Heat Transmittance W/m^2 K**
Sample # 1 (12 layers)	0.762	28.8	0.0864	0.113	161
Sample # 2 (6 layers)	0.792	28.8	0.0762	0.096	258
Sample # 3 (6 layers)	0.803	28.8	0.0784	0.098	293

*coefficient of thermal conductivity; **normalized thermal conductivity

The main reason to use EBC polyester as a bonding agent for nonwoven fabrication was its biodegradable character. The EBC polyester is designed to perform required life time reliability for an industrial product and then fully degrade within a composting environment. In a time frame comparable to cellulosics (paper), this aliphatic-aromatic polyester fully degrades to carbon dioxide (CO_2), water and biomass. Within 12 weeks in an active composting site, an article made from this copolymer typically becomes invisible to the naked eye and completely biodegrades within six month (Haile, 2001). It is assumed therefore that biodegradation of bagasse/cotton/EBC nonwoven composite will imply a degradation of components in the same time frame. Biodegradation of nonwovens was monitored as the loss of mechanical properties after 6 weeks in soil.

The initial modulus of the composite nonwoven is significantly higher than that of the melt blown biodegradable polyester nonwoven fabric (Table 9). Therefore a loss of mechanical properties after soil burial is indicative of the degradation of cellulosic components and/or of the bonding polymer. Indeed, except for the sample soil buried for three weeks, all the others show a descending trend of the strength (i.e., stress at maximum load), strain and modulus, lower values being recorded after 6 weeks (Table 4-3). This can be explained by the fact that the cellulosic components, cotton and bagasse, and the biodegradable polyester started to degrade. The rate of degradation was almost constant in time. The same trend was shown by the variation of the complex modulus of elasticity (E*) with the time in soil (Figure 4-9). After 5 weeks E' was almost 2.5 less than the initial values. However, a visual examination showed that the rate of degradation for cellulosic components was higher that that of the EBC polyester.

Table 4-3. Mechanical properties of nonwoven materials before and after soil degradation

Material	Stress at Max Load (kPa)	Strain at Max Load (cm/cm)	Maximum Percent Strain (%)	Modulus (kPa)
EBC Melt Blown Nonwoven Fabric	1004	0.79	133.5	35
Composite Nonwoven (CN)	10209	0.11	52.9	1289
CN Week 1	11473	0.21	47.5	1280
CN Week 2	10688	0.23	62.6	1257
CN Week 3	7039	0.16	35.4	878
CN Week 4	8720	0.20	59.8	1020
CN Week 5	9072	0.20	35.8	972
CN Week 6	7756	0.23	30.4	825

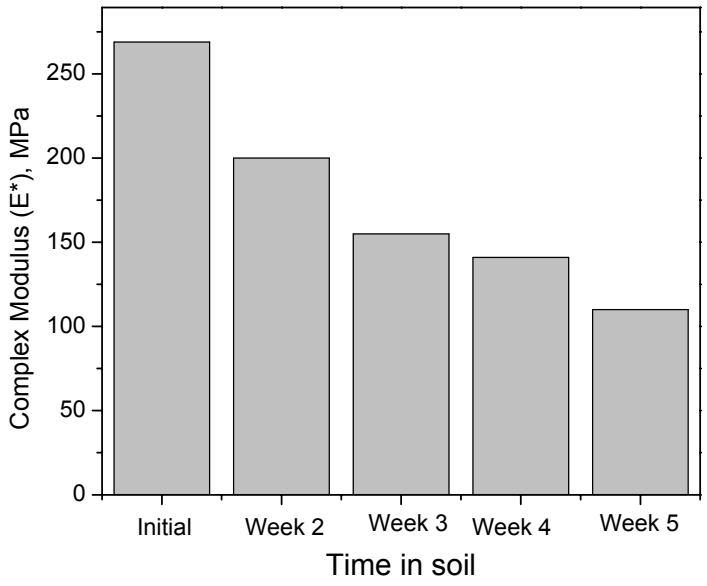


Figure 4-9. Decaying in time of the complex elastic modulus of bagasse/cotton/EBC composite nonwovens after soil burial.

4.4 Conclusions

Visco elastic nonwoven composites exhibiting good mechanical characteristics were successfully prepared by hot pressing bagasse/cotton webs sandwiched between EBC melt blown nonwoven fabrics. The nonwoven composite degraded at a rather high temperature (220°C), but the exploitation temperature is limited to 70-100°C by softening and melting of the synthetic polymer. Nonwoven composite samples exhibited low thermal and heat transmittance coefficients. For the same composition these coefficients can be tuned by changing the construction parameters (density and number of layers). The soil burial method clearly evidenced the starting of the biodegradation process by the diminution of the mechanical characteristics (strength, modulus) of the test samples after weeks in soil.

Chapter 5 Conclusions and Recommendations

In response to the Louisiana's agriculture sector for finding new options for bagasse use, this research work focused on a study of characterization of bagasse fibers and of bagasse-based nonwovens. The extraction of bagasse fibers from sugar cane does not require a complicated technological process. One of the goals of this study was extracting bagasse fibers for nonwoven use employing an atmospheric extraction process. From previous studies, performed using a steam explosion process, it was determined that the influential parameters for bagasse extraction were: alkali concentration of the solution, time of the reaction, and mechanical agitation. The same extraction variables were used in characterization for atmospheric extraction performance. From all the parameters, the alkali concentration of the solution was found to be the most influential when varied more than 0.5 N. The study demonstrated the possibility to apply the atmospheric extraction process on a large scale for industrial operation. The extraction time in atmospheric process can be improved by using a continuous method.

Depending on the final use for the bagasse the extraction conditions can be adapted accordingly. Fibers extracted at 1.0 N alkali solution concentration showed good properties, which will make them usable in a large range of nonwoven materials.

New methods were used to characterize some of the physical properties of bagasse fibers. The length of the fibers is determined by the crushing process of the sugar cane stalk. Using computer implemented technology, accurate measurements for the length of the fibers were taken. This involved a minimum and affordable level of technology. To determine the fineness a regression model was employed. This model gave the relation between fineness and cross-sectional area of the fibers. Confidence and

prediction intervals were determined. Depending on the required accuracy of results, the model can be used for other unconventional fibers. Also different models can be derived for each individual type of natural unconventional fiber. Given the fact that only a magnification level of 50x is necessary in order to have an accurate reading for the cross-sectional area, a simple optical microscope can be used. This method can be used conversely to determine the cross-sectional area of a fiber, if the fineness is known.

Different nonwoven structures were created using a minimum level of technology. The technological process used involved extraction, cleaning and carding as the preparation of the fiber web step, and needle-punching and thermal-bonding as the bonding of the fibers in the web step. It was determined by laboratory trials that, due to the short length and stiffness of the bagasse fibers, nonwovens materials with a composition of more than 70% bagasse are not easy to manufacture. In the web formation step the layer coming out of the carding machine was not uniform and continuous. To improve the web formability bagasse needed to be mixed with some other fibers with better carding properties. These fibers, depending on the final product use, were cotton, kenaf, flax, ramie, jute, polyester and polypropylene. The presence of synthetic fibers also made possible thermal bonding of the web. Thickness was controlled by the number of layers. Once the web has been formed a mechanical or/and thermal treatment was given to bind the fibers together. The mechanical method used to entangle the fibers was needle-punching. For the theremal bonding a press with heated plates was used.

This study focused on making two types of nonwoven materials: one based on bagasse and/or other bast fibers blended with synthetic polymers, and the second one based on bagasse, kenaf, and biodegradable co-polyester.

A battery of tests was performed on all structure types in order to assess the mechanical and thermal properties. It was determined that even though the degradation temperature for the nonwoven materials is rather high, the temperature at which the webs can be reasonably manufactured and used is limited by the presence of the synthetic polymers in the composites. Also mechanical properties were analyzed as a function of frequency. At higher frequencies, the nonwoven materials displayed lower mechanical characteristics.

As expected, nonwovens showed good insulation properties having low thermal and transmittance coefficients. The structure and construction parameters of nonwovens can be adjusted according to the end use requirements.

Soil burial proved that over time a biodegradation process took place, influencing the mechanical and physical characteristics of nonwoven materials. These characteristics were assessed in both static and dynamic domains. After just a relative short period of time in which the nonwovens samples were buried the static modulus diminished with about 30%, and the complex modulus with about 50%. The rate of degradation was almost constant in time.

The present work represents initial studies of bagasse fiber characterization, from the perspective of using image analysis methods. The new approach of determining fiber fineness and some other physical characteristics by this method can be extended to other unconventional coarse fibers, for which there is a lack of testing instrumentation. Nonwovens appear to dominate the market for composite materials, finding applications in all sectors. Finding new methods for making and characterizing these new materials will impact their applicability.

Several areas for future work may be:

1) Optimize the extraction process for different lignocellulosic materials by using safe and environmentally friendly processes. The lignin presence is a direct result of different extraction conditions (alkali concentration, reaction time, mechanical agitation, and presence or absence of steam explosion).
2) Investigate new methods of extraction and subsequently softening bagasse fibers in order to increase their use potential in wovens. That will require certain properties to be met, so that bagasse fibers could be spun.
3) Develop new analytical methods for testing lignin concentration after the extraction process. Thermal analysis methods are begginig to be used to determine the variation of lignin concentration with several process parameters.
4) Explore the possibility of creating a comprehensive data base with regression models for each type of unconventional fiber, which will allow determining the fineness in real time and without elaborate and expensive testing machines.

Elements of novelty

This work provides a new approach for determining the physical characteristics for unconventional fibers. The machinery and instrumentation presently used in determination of physical charcteristics for fibers are designed to be in accordance with cotton's characteristics. The fibers used in this work have physical properties considerably diferent from cotton. This conveyed the idea of finding alternative methods appropriate for this type of fibers. The method used in this study, based on image analysis, provides a simple and an unexpensive way of overcoming this problem. This

new approach opens the path for further use of computer based technology in different aspects of Textile Science.

For the first time the extraction of bagasse fibers was acomplished using an atmospheric process. Both methods used, continous and batches, gave similar results from physical and mechanical characteristics poin of view. The same processed was used for extracting kenaf fibers.

The present work brings for the first time the isssue of products based mainly on bagasse, other then geotextiles. In several different structures, in this study, bagasse has been used as the principal component. The composite materials resulted proved that bagasse can be a succesful substitute for other fibers with similar or better properties. Finding alternative uses for bagasse can alievate the agricol sector of Louisiana and other states where sugar cane is grown.

A new level of approching the manufacture of nonwoven composites based on unconventional fibers was achived by designing and making thermal-bonded entirely biodegradable nonwovens. This have been accomplished using a new biodegradable synthetic copolymer. Thermal-bonding gives the necessary dimensional stability and resistance while the biodegradability assures the environmentally friendly degradation of the product after the intended use.

References

1. Akin, D. E., Morrison, W. H., Rigsby, L. L., Dodd, R. B., and Raymer, P. L. Chemical and Structural Properties of Kenaf Cell Types. Kenaf Properties, Processing and Products. Edited by Terry Sellers and Nancy Reichert, 1999.

2. ASTM D1448-97. Standard Test Method for Micronaire Reading of Cotton Fibers.

3. Barnes, A. C. (1964). The Sugar Cane, Leonard Hill, London, 1964.

4. Batra, S. K., Other Long Vegetable Fibers: Abaca, Banana, Sisal, Henequen, Flax, Hemp, Sunn, and Coir, in Handbook of Fiber Science and Technology, vol. IV, M. Lewin and E. M. Pearce, Eds., Marcel Dekker, New York, 727-807, 1983.

5. Bhat, G. S. and R. Haoming, "Effect of Binder Fibers on the Processing and Properties of Thermal Bonded Cotton-Based Nonwovens, Beltwide Cotton Conferences, Atlanta, GA, January 8-12, 2002

6. Buco, S. M., and J. G. Montalvo, Jr. 2001. Analysis of white specs by NIR HVI: A fesability study, in Proceedings of the Betwide Cotton Conference, ed. P. Dugger

7. Calamari, T. A., 2002. Advantages of Using Natural Fibers in Nonwoven Composites, CD-Rom Book of Papers, Beltwide Cotton Conferences, Atlanta, GA, January 8-12.

8. Chiparus, O., I. Negulescu, Y. Chen, M. Warnock and L. Wadsworth, "Biodegradable Nonwovens Based on Cotton and Polyesters", Proceedings, Beltwide Cotton Conferences, Atlanta, GA, January 8-12, 2002.

9. Clements, H. F. (1980). Sugarcane Crop Logging and Crop Control. The University Press of Hawaii, Honolulu.

10. Collier, B. J. and Arora, M. S., 1994, "Water Pretreatment and Alkaline Treatment for Extraction of Fibers from Sugar Cane Rind," Clothing and Textiles Research Journal, 14(1): 1-6.

11. Collier, B.J., Tortora, P. G. (2001). Understanding Textiles 6th edition. Prentince Hall, 2001.

12. Collier, B. J., Collier, J. R., Agarwal, P., and Lu, Y-W. (1992). Extraction and Evaluation of Fibers from Sugar Cane, Textile Research Journal, 62(12), 741-748, 1992.

13. Collier, J. R., Tao, W. Y., and Collier, B. J. (1991). Bending of Internally Reinforced Fibers, Journal of Textile Institute, 82, 42-51, 1991.

14. Collier, J. R., Collier, B. J., Thames, J. L., and Elsunni, M. M. (1995). Production and Evaluation of Sugar Cane Geotextiles. Report submitted to Louisiana Transportation Research Center, Baton Rouge, Louisiana.

15. Cook, J. G. (1984) Handbook of Textile Fibers. Natural Fibers, Merrow Publishing Co. Ltd, Durnham, England

16. Cooke, T. F. (1990). Biodegradability of Polymers and Fibers – A Review of the Literature. Journal of Polymers, 9, 171-211

17. D.A. Richter, Vol 2, 1267-1269. Memphis, Tennessee: National Cotton Council of America, 2001

18. De Jong, E., Huigen, M., van Roekel, G. (1999) Considerations About the Commercial Production of Elementary Flax Fibers for Nonwoven Applications. Index '99 Nonwovens Congress, Fibers Session. EDANA, Brussels, Belgium.

19. DeLong, E. A., Process to Dissociate and Extract the Lignin and the Xylan from the Primary Wall and Middle Lamella of Lignocellulosic Material which Retains the Structural Integrity of the Fiber Core, U.S. Patent 4,908,099; March 13, 1990.

20. Elsunni, M. M., and Collier, J. R. (1996). Processing of Sugar Cane Rind into Nonwoven Fibers. Journal of American Society of Sugar Cane Technologists, 16, 94 – 110.

21. Finch, P., and Roberts, J. C. (1985). Enzymatic Degradation of Cellulose, in "Cellulose Chemistry and Its Application," T. P. Nevell and S. H. Zeronian, Eds., Ellis Horwood, West Sussex, England, pp. 312-343

22. Foreman, J. (1997) Dynamic Mechanical Analysis of Polymers. American Laboratory, January 1997.

23. Haille, W. A., Tincher, M. E., Tanner, C. M. (2001). New Biodegradable Copolyester for Fiber and Nonwoven Applications. Nonwoven Symposium, EDANA'S 2001.

24. Hassel, R. L. (1991) Using Temperature to Control Quality. PI Quality , 1991.

25. Hatakeyama, T., Quinn, F. X. (2000). Thermal Analysis: fundamental and application to polymer science. 2nd ed. Chichester [England] : Wiley, 1999.

26. ISO 2061-95. Determination of Twist in Yarns. Direct Counting Method.

27. Mansfield, R. (2001). 30 Years of Big Ideas. Textile Industries, 50 June, 2001.

28. Marienfeld, M. (1995). Geotextile trends and developments. TAPPI Journal, Vol.78, No. 9, September, 1995.

29. Marthur, R. B. L. (1975). Handbook of Cane Sugar Technology, Oxford anfd IBH, New Delhi, 1975.

30. Mathot, V. B. C., "Calorimetry and Thermal Analysis of Polymers", Hunser Publishers, Munich, 1994

31. Meade, G. P., Chen, J. C. B. (1997). Sugar Cane Handbook, Wiley, New York, 1997.

32. Montalvo, J. G., Jr. and T. M. Von Hoven. SDL Calibration Cottons Packed in Tubes. 2000. Tektran. Agriculture Research Service.

33. Mueller, D. H., Krobjilowski, A., Schachtschneider, H. (2002). Acoustically Optimized Structural Parts Basing on Hybrid Fleeces. 2002 Beltwide Cotton Conferences, Atlant, GA, January 8-12

34. Moreau, J. P., Bel-Berger, P., Tao, W. (1995). Mechanical Processing of Kenaf for Nonwovens. TAPPI Journal, February 1995

35.Paturau, J.M., 1989. By-Products of Sugar Cane Industry, 3rd Edition, Elsevier, Amsterdam.

36. Parikh, V, Calamari, T. A., Sawhney, A. P. S., Warnock, M. (2002). Refining of Kenaf Fibers for Processing into Automotive Nonwovens. Fifth Nonwoven Conference 2002

37. Pierce, D. M. (1996). A campaign for real thermal analysis. TRIP Vol. 4, No. 12, December 1996.

38. Romanoschi, O., Romanoschi, S., Collier, J. R. and Collier, B. J., 1997b, "Kenaf Alkali Processing," Cellulose Chemistry and Technology, 31(5-6): 347-359.

39. Thames, J. L., Collier, B. J., and Collier, J. R., 1994, "Production and Evaluation of Sugar Cane Fiber Geotextiles," Book of Papers, AATCC International Technical Conference and Exhibition, Research Triangle Park, NC, 1994, pp 42-47.

40. Webber, C. L. Industrial Crops and Products 2: 27(1993)

41. Unknown, U.S. Dept. of Agriculture, Agricultural Research Service, Agricultural Research 41(6): 19(1993).

42. Scion Corporation Page. http://scioncorp.com/index.html. Date Referenced November 25, 2002.

43. Sellers, T., Miller, G. D., Fuller, M. J. (1993). Kenaf core as a broad raw material. Forest Products Journal, 43, 69-71.

44. Smith, D. K., Natural Fibers in Nonwovens Processes. Proceedings Fourth International Nonwoven Symposium. 2001 Beltwide Cotton Conference, Anaheim, California.

45. Suh, H., Duckett, K., and Bhat, G. (1996). Biodegradable and Tensile Properties of Cotton/Cellulose Acetate Nonwovens. Textile Research Journal. 66(4), 230-237

46. Tilby, S. E., Mason, W. C. W., and Hodge, D. W. Sugarcane Separation, U. S. Patent 3,976,498, August 24, 1976.

47. Valiente, J. M., Carreterro, C. M., Gomis, J. M., and Albert, F. (2001). Image Processing Tool for the Purpose of Textile Fabric Modeling. Procedeeings XII ADM international Conference, Rimny, Italy

48. Van Dillewijn, C. (1952). Botany of Sugar Cane, Chronica Botanica, Waltham, MA

49. Xu, B. 1995. Measurement of Structural Characteristics of Fiber Segments in Nonwoven Fabrics. International Nonwoven Conference, p. 145-153.

50. Woodings, C. (2001). New Developments in Biodegradable Nonwovens. www.technica.net

51. Zhang, W., Matic-Leigh, S.R., and Rippy, D. V. 1996. Image Analysis Used for Quality Control. America's Textiles International 25, no. 3, 6-8.

Appendix: Letter of Permission

6 February 2004 Our ref: HW/smc/Feb.2004.jl050

Dear Ovidiu Chiparus

BIORESOURCE TECHNOLOGY, Vol 90, 2003, Pages 305-309, "An Image Method to Evaluate Bagasse Fiber Dimensions"

As per your letter dated 22 January 2004, we hereby grant you permission to reprint the aforementioned material at no charge **in your dissertation** subject to the following conditions:

1. If any part of the material to be used (for example, figures) has appeared in our publication with credit or acknowledgement to another source, permission must also be sought from that source. If such permission is not obtained then that material may not be included in your publication/copies.

2. Suitable acknowledgment to the source must be made, either as a footnote or in a reference list at the end of your publication, as follows:

   "Reprinted from Publication title, Vol number, Author(s), Title of article, Pages No., Copyright (Year), with permission from Elsevier".

3. Reproduction of this material is confined to the purpose for which permission is hereby given.

4. This permission is granted for non-exclusive world **English** rights only. For other languages please reapply separately for each one required. Permission excludes use in an electronic form. Should you have a specific electronic project in mind please reapply for permission.

5. This includes permission for UMI to supply single copies, on demand, of the complete thesis. Should your thesis be published commercially, please reapply for permission.

Yours sincerely

Helen Wilson
Rights Manager

VITA

Ovidu Iulius Chiparus was born on July 25, 1969, in Iasi, Romania. He is the son of Vasile and Veronica Chiparus. He is married to Olena Nesteruk.

In 1993, Ovidiu received his five year integrated master degree in Mechanical Engineering from Technical University “Gh. Asachi”, Iasi, Romania. In January 1999, he entered the graduate doctoral program in the School of Human Ecology at Louisiana State University. In addition, he enrolled in August, 2002 in a master’s program Experimental Statistics Department at Louisiana State University.