To the Graduate Council:

I am submitting herewith a dissertation written by Hageun Suh entitled "Biodegradability and Process Characterization of Nonwovens Formed from Cotton and Cellulose Acetate Fibers." I have examined the final copy of this dissertation for form and content and recommended that it be accepted in partial fulfillment of the requirements for the degree of Doctor of Philosophy, with a major in Human Ecology.

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Kermit Duckett, Major Professor

We have read this dissertation
and recommend its acceptance:

Accepted for the Council:

Associate Vice Chancellor and
Dean of The Graduate School

BIODEGRADABILITY AND PROCESS CHARACTERIZATION OF NONWOVENS FORMED FROM COTTON AND CELLULOSE ACETATE FIBERS

A Dissertation
Presented for the
Doctor of Philosophy
Degree
The University of Tennessee, Knoxville

Hageun Suh
May 1997

DEDICATION

This dissertation is dedicated to my parents,

Mr. Sooman Suh

and

Mrs. Sunyang Chung

who have always given me their support and encouragement.

ACKNOWLEDGMENTS

Throughout my graduate school life at the University of Tennessee, there are many people to whom I should be thankful. I am particularly grateful to my advisor, Dr. Kermit Duckett, for his support, guidance and encouragement during my entire doctoral program. Also, this work could not be possible without the valuable support and knowledge from my Dissertation Committee, Dr. Gajanan Bhat, Dr. Larry Wadsworth, and Dr. Spiro Alexandratos.

There are a number of people whose assistance and advice have been valuable to this work. I would like to thank Mr. Richard Meade for his help on nonwoven processing, and Dr. Arnold Saxton for his help on statistical analysis. The support from the Department of Textiles, Retailing and Consumer Science, the Graduate School and the University of Tennessee Agricultural Experiment Station should be also appreciated.

Lastly, I would like to thank my husband, Dr. Myungsoo Joo, for his encouragement, support and inspiration, and for his tremendous help on biodegradation.

ABSTRACT

A possible candidate as an environmentally friendly nonwoven fabric is one which can be formed from the thermal calendering of a cotton/cellulose acetate blend. The results presented have focused on biodegradable properties of the fibers, physical properties of the fabric, and process optimization of the thermal calendering. Cotton, which is a comfortable, absorbent and biodegradable fiber was the base fiber in the nonwovens. Cellulose acetate (CA), which is a thermoplastic, hydrophilic, modified cellulosic fiber was used for the binder fiber.

Biodegradation of cellulose has been intensively studied, and cellulose is believed to be readily biodegraded by many microorganisms due to the activity of cellulase enzymes catalyzing the hydrolysis and/or oxidation of cellulose. However, the biodegradability of CA is less than certain. To determine a semi-quantitative measure of the biodegradation of CA fibers, the standard test method of AATCC 30-1988 was selected. After a 12-week soil burial test, evidence of microbial attack on CA fabric was obtained on the basis of 27% strength loss. As a more reliable method for monitoring the activity of aerobic microorganisms, the ASTM test method D5209-91 was adopted. From the aerobic sludge test, the amount of CO_2 evolved from the decomposition of CA, cotton and the fibers in the blend was measured. The biodegradability of CA fibers, with a degree of substitution of 2.5, was confirmed by showing carbon dioxide evolution. In addition, the synergistic effects of multi-enzyme systems between cellulase and esterase were suggested based on the increased biodegradation rates in fiber blends.

Opening, blending, carding, and thermal calendering processes were used in the fabrication of the nonwovens. Pretreatment with solvent vapors was introduced for modifying the softening temperatures of CA and for lowering the calendering temperatures required otherwise. The success of the solvent-assisted thermal calendering is demonstrated in enhanced tensile strengths of the nonwoven. In addition, modification of tensile properties by solvent vapor pretreatment shows how enhanced strength can be obtained with lower calendering temperatures.

Process optimization in thermal calendering is a necessary procedure to understand the bonding behavior of raw materials and to obtain the adequate properties of the resulting nonwoven fabrics. The experiment was designed for a 3-way factorial design (3x3x8) with the following factors: bonding temperature, blend ratio and solvent treatment time. The effects of the factors on a total number of 18 physical properties were determined by analysis of variance, least-square-means comparison, regression and correlation techniques. The results indicated that all the physical properties were significantly influenced by the bonding temperatures and the solvent treatment times. In addition, for optimal conditions, four responses were selected for stronger, softer, more extensible and more breathable nonwoven fabrics. The optimal processing conditions in a given blend ratio were suggested by a response surface technique based on multivariate-multiresponse analysis, and distance and desirability functions.

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CHAPTER 1

INTRODUCTION

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 is increasingly demanding biodegradable or environmentally friendly textiles, especially disposable nonwoven products. The possibility of compostable/biodegradable disposable nonwoven products such as diapers, incontinence products and surgical gowns in heavily used landfills has attracted special attention in an effort to solve a solid waste crises. Unfortunately, there are only a few biodegradable fibers available which can serve as raw materials in nonwoven production, and in most cases newly developed biodegradable fibers are expensive.

Examples of important disposable nonwoven products are baby diapers, sanitary napkins, medical disposable, adult pads, wipes, fabric softeners, etc. [6], which generally end up in solid-waste and sewage systems. According to the US Environmental Protection Agency (EPA), approximately 150 million tons of solid wastes are generated annually in the US alone. Approximately 2% of total garbage (3.6 million tons) consists of disposable baby diapers [4]. Most solid wastes are disposed of in landfills, while others are recycled, composed and incinerated [4]. In addition, many disposable nonwovens such as flushable wipes end up in a sewage system [13]. Recycling alone is not yet well developed and there exit difficulties such as collection, energy consumption and application problems. The incineration system, mainly for medical waste, also has energy consumption and toxicity problems. From their complexity has arisen interest in the biodegradability of disposable goods in landfill, composting and sewage disposal systems. These systems then can be considered as natural recycling processes, in which final products such as CO_2, CH_4 and biomass are returned to the earth [13]. Hence, biodegradable nonwoven products, especially for short term usage, are advantages for environmental concerns through natural decomposition processes.

Cotton fiber has attracted significant interest in nonwoven markets, especially along with thermal bonding processes, because of its natural comfort properties and biodegradability, and the commercial development of bleached cotton processability. Cotton, a cellulosic fiber, is hydrophilic and has good absorbency and comfort properties. Cotton fibers are known to be easily biodegradable and have been utilized in disposable nonwoven products produced by chemical or thermal bonding processes using chemical binders or synthetic binder fibers. However, the synthetic binder fibers such as polyester and polypropylene are normally not biodegradable and the chemical bonding processes involve environmental pollution. Several different heat-fusible, thermoplastic synthetic fibers have been used as binder fibers in nonwoven products containing cotton fibers as the base fiber. Low melting polyester copolymer, polypropylene and polyethylene have been used and studied as binder fibers [33, 34, 35]. However, the studies indicated that a higher cotton blend content resulted in the decrease in strength and that higher bonding temperatures were required to raise the fabric strength. In addition, the synthetic binder fibers are limited in disposability.

Cellulose acetate (CA) fiber is a feasible choice to solve the above limitations. In fact, cellulose acetate fibers have several advantages. Cellulose acetate, a modified cellulose, is a thermoplastic fiber and can be used as a thermal binder fiber for cotton [1]. Its relatively low softening temperatures in the range of 180 to 205°C and easy wettability are advantageous in its choice as a binder fiber, eliminating the use of any truly non-biodegradable synthetic fiber or chemical binder. In addition, CA contains both acetyl and hydroxyl groups and is soluble in many common solvents such as acetone, low boiling ketones and methylene chloride. In reality, CA can be modified with many solvents acting as plasticizing agents. The chemical modification of CA by using plasticizing agents provides additional flexibility in thermal bonding by possibly enhancing bonding adhesion and lowering effective operating bonding temperatures.

In terms of the biodegradability of acetate fibers, the results from a literature review are somewhat contradictory. Cellulose acetate fibers have been promoted as biodegradable, or to be easily incinerated or recycled [1, 65, 95]. On the other hand, some studies have indicated that CA is not biodegradable. From the above literature review, several questions were raised that might require additional study. First, is or is not CA biodegradable? Can one distinguish the biodegradability results of Gu [42, 43] from the contradictory results of Siu [93]? The samples of Gu and of Siu were not identical and, hence, may not be comparable because of differences in crystallinity and orientation, the degree of polymerization or chemical additives, etc. Furthermore, the biodegradation processes were not equivalent. Few standard methods are available for reproducible testing and assignment of biodegradability of textile fibers. The biodegradability of CA fibers should be evaluated by a valid test procedure.

Over the past several years, nonwoven products containing cotton and synthetic binder fibers which could be used in medical and consumer applications have been studied [32, 33, 34, 35]. However, there have been the continuing problems of low strength at high cotton content and the concern over limited natural disposability. Cellulose acetate fiber is one of the commercially available fibers which have thermoplastic, hydrophilic and potentially biodegradable properties, as well as which can be used for binder fiber. One disadvantage in using cellulose acetate fiber over other binder fibers is its relatively high melting temperatures. With an innovation toward optimizing the nonwoven fabric properties, pretreatment with acetone vapor, a common solvent for cellulose acetate, is one of the choices to lower the effective bonding temperatures, as well as to improve the physical properties of the nonwovens from cotton and cellulose acetate blends.

Process optimization in thermal calendering is a complicated and tedious, but necessary, procedure to understand the bonding behavior of raw materials and to obtain the adequate properties of the resulting nonwoven fabrics [25]. The effect of varying the processing variables, bonding temperature, feed-roll speed and nip-roll pressures, and the effect of blend ratios on bonding have all been recognized as important. In most studies, optimal processing conditions for selected properties of nonwovens produced by thermal calendering have been examined through empirical data. However, in many cases, the effects of processing conditions are interactive, and an optimal processing condition for one property of the nonwovens is not necessarily applied to other properties. Using statistical analysis is the one way to examine significance of the effects of processing conditions on the physical properties of the nonwovens from the solvent-assisted thermal calendering. Statistical methods including analysis of variance, least-square-means comparison, regression and correlation techniques provide simple tools to understand relationships between and/or among processing conditions and properties of nonwoven fabrics. In addition, optimization by response surface techniques is, possibly, the simplest way to optimize several processing conditions and/or physical properties, simultaneously.

This study is based on the possibility of producing biodegradable nonwoven fabrics for disposable applications by using renewable natural resources in an environmentally safe way. The objectives included: (1) the examination of the biodegradability of cellulose acetate and cotton/CA blended fibers by standard test procedures, (2) the utilization of a solvent-assisted thermal calendering technique to enhance the physical properties of cotton-based cellulosic nonwovens and to lower the bonding temperatures, and (3) the optimization of the thermal bonding process by statistical methods.

CHAPTER 2

LITERATURE REVIEW

BIODEGRADABILITY

BIODEGRADATION MECHANISM

Enzyme-Catalyzed Process

The term, "biodegradability", is difficult to define. According to the ASTM definition of a biodegradable plastic, it is a "plastic in a loss of some properties, in which the degradation results from the action of naturally-occurring micro-organisms such as bacteria, fungi and algae" [12]. From the above definition, the polymers and fibers, which are vulnerable by microbial attack under certain conditions are classified as biodegradable materials. Because of the increased need of disposal textile products, the biodegradability of fibers should be reexamined and reemphasized in terms of the evaluation methods and designing biodegradable textiles. However, there are no strict definitions or standards for the term, "biodegradable." Also, there are no specified conditions of microbial activities such as temperature and pH, and specified time frame for the total mineralization process.

The biodegradation process is generally explained by the enzymatic catalyzed process, described frequently as analogous to a key fitting into a lock [20]. Enzymes produced by microorganisms catalyze the degradation of polymers and fibers. The production of enzymes by microorganisms depends on several factors, such as temperature, pH and oxygen availability. Moisture and nutrients are needed also for the growth of microorganisms. In addition, it is generally believed that enzymes must be physically adjacent to the polymer molecule and should fit into the stereochemical conformation of the molecules [20]. The biodegradation process involves a hydrolysis and/or oxidation mechanisms, resulting in polymer chain scission. The continuation of the scission procedure finally results in a water-soluble material and a total mineralization to products such as CO_2, H_2O, CH_4 or biomass [100].

Effects of Polymer Structure

Although different types of microorganisms and different conditions of environments are involved in biodegradation, there are basic guidelines for understanding how polymer structures respond to the enzymatic breakdown. Important variables in polymer structures are the chemical linkages in the polymer backbone, and the pendant groups, their position and chemical activity [20].

The guidelines for the biodegradability of polymers are based on several characteristics of polymer structures: hydrophilicity, polymer backbone, morphology, molecular weight and branching [20, 50, 99]. Wettable and hydrophilic polymers tend to be biodegradable, since most of the enzyme-catalyzed reactions occur in aqueous media. Flexible polymers with heteroatom environments (e.g., ester, ether and amide linkages) and with long repeating units tend to be biodegradable, since the flexible chains can fit into the active site of the enzymes. Therefore, polymers with aliphatic rather than aromatic chains have a tendency to be biodegradable, and carbon-carbon chain polymers are not usually biodegradable with the exception of poly(vinyl alcohol). Less crystalline polymers tend to be biodegradable, since the degradation of amorphous regions occurs prior to the degradation of crystalline regions. Lower molecular weight polymers tend to be biodegradable. Polymers without branching and pendant groups tend to be biodegradable, since those units may prevent the enzyme from attaching it self to the polymer backbone.

BIODEGRADABLE POLYMERS

Natural Polymers

Cellulose is a linear condensation polymer of β-1,4-linked-D-glucose, which is biodegradable by many organisms that produce enzymes, called cellulases. The cellulase enzymes catalyze the hydrolysis and/or oxidation of cellulose chains into cellobiose, glucose and finally mineralization [20, 66]. The regenerated celluloses such as cellophane and viscose rayon are biodegradable, since their microstructures are also celluloses with only different crystalline structures [66]. Cotton and cellulose acetate, a cellulose ester, will be discussed later.

Starch is a carbohydrate obtained from plants and consists of two types of glucose polymers: a linear polymer of α-1,4-linked D-glucose (amylose) and a branched polymer of α-1,4- linked-D-glucose with α-1,6-linked-D-glucose branches. Starch is biodegradable through an enzyme-catalyzed hydrolysis process [66]. Starch is often used in the manufacture of biodegradable polymers by plasticizing or blending with other non-biodegradable polymers. Various types of natural polymers are utilized also to modify non-biodegradable polymers, such as Konjac, Chitin, gelatin, keratin and low-molecular-weight natural rubber [20, 66].

Biosynthesized Polymers

Poly(β-hydroxybutyrate-co-β-hydroxyvalerate) (PHBV) is a biodegradable polymers developed by ICI, called Biopol. Biopol is a semicrystalline aliphatic polyester produced by the fermentation of sugar by bacteria [4, 66, 99]. The properties of the copolymer are based on the content of poly(hydroxy valerate) (PHV). High valerate content in copolymers increase melt processability but lower tensile strength [117].

Poly(lactic acid) is a thermoplastic aliphatic polyester polymerized from biologically produced lactic acid, which is expensive and mainly used for medical purpose. Recently inexpensive poly(L-lactic acid) has been introduced [117]. Pulluman is a water-soluble polysaccharide produced by fungi, and Chitosan is deacetylated chitin which is a polysaccharide derived from shellfish [117].

Synthetic Polymers (Petroleum Derivatives)

There are two biodegradable polymers derived from petroleum: polycaprolactone and poly(vinyl alcohol). Poly(ε-carpolantone) (PCL) is an aliphatic polyester synthesized by ring opening polymerization. It has been developed by Union Carbide and Phone-Poulenc [50, 66].

Poly(vinyl alcohol) (PVA) is synthesized from the alcoholysis of poly(vinyl acetate). The properties depend on molecular weight and degree of hydrolysis. Water-soluble PVA, with a degree of hydrolysis, is low in crystallinity, while water-insoluble PVA, with a degree of hydrolysis above 98%, shows a greater crystallinity due to packing of the hydroxyl groups [66]. PVA is readily degraded in waste-water-activated sludge. The initial mechanism is based on the enzymatic oxidation of the hydroxy groups to ketone groups, which progresses to chain scission due to hydrolysis [20, 50, 117].

Other commercially available synthetic polymers have been investigated and modified to increase their biodegradability. Polyolefins are generally not biodegradable, but some low-molecular-weight linear polyolefins can be made biodegradable. Aliphatic polyesters are biodegradable due to their flexible chains. Polyurethane, based on aliphatic polyester, shows increased biodegradability when compared the those based on aromatic polyester [20, 50].

TEST METHODS FOR BIODEGRADABILITY

As mentioned before, there are several ways to dispose of nonwoven fabrics: landfill, composting, sewage, recycling and incineration. Several methods have been developed to examine the biodegradability of polymeric materials that simulated landfill, composting and sewage systems. Differences in these methods are in the environmental sources for microorganism growth and oxygen availability. Response to biodegradation process is based on changes in mechanical properties such as tensile strength, weight loss, molecular weight change, the amount of gas evolved and total mineralization time.

Simulated landfill systems

A simulated or synthetic municipal solid waste (SMSW) mixture is the main environment for microorganisms in this procedure. SMSW can be prepared on an assumed averaging of constituents of actual municipal landfill, which usually contains paper, food and plastic waste [26, 42, 67]. Weight loss and total gas production curves can be used to assign the biodegradability of materials.

Composting Systems

In this procedure [42, 43, 100], externally activated microorganisms are added. For aerobic composting conditions, an inoculum is derived from mature compost, and the amount of CO_2 evolved is measured as an indicator of biodegradability. For anaerobic conditions, an activated inoculum is prepared from a stabilized mixture of various bacteria derived from the anaerobic treatment of a organic fraction in the municipal landfill, and the carbon conversion to CH_4 gas is monitored [100].

Sewage Systems

The test procedures are mainly for flushable products such as napkins and wipes [13, 60]. One way to conduct biodegradability in sewage systems is to do a field study at municipal sewage plants, where pieces of the polymeric materials such as films, fibers and webs can be added to the aerobic or anaerobic digester tanks. As an example, rayon fibers showed the microbial attachment and decomposition of the surface of rayon fibers after the direct sewage treatment [13].

In addition, preparation of an inoculum from sludge samples allows one to accelerate biodegradation testing of polymeric materials due to the severe conditions in the test media. Two ASTM standard procedures are available for this, based on the sludge inoculum from the aerobic or anaerobic digesters in the municipal sewage plants [10, 11].

Also, other testing environments can be used to assign biodegradability ratings to polymeric materials. Pure cultures of specific fungi, bacteria or purified enzymes can be used to evaluate the colony growth of microorganisms in agar plates [20, 50]. When microorganisms are unknown, mixed cultures of several fungi and/or bacteria can be used [20]. In some applicable cases, C isotropic labeling systems can be used, in which the extent of carbon conversions to CO_2 or CH_4 can be accurately detected [50].

BIODEGRADABILITY OF CELLULOSIC MATERIALS

The biodegradation of cellulose has been intensively studied, and cellulose is believed to be readily biodegradable and mineralized by many microorganisms due to the activity of cellulase enzymes catalyzing the hydrolysis and/or oxidation of the cellulose [14, 20, 24, 36, 66, 93, 102]. The mechanism of microbial degradation of cellulose is based on the action of specific enzymes secreted by microorganisms only in the presence of cellulose.

Cellulose is a linear polymer consisting of the cellobiose basic unit, 2-anhydroglucose units linked together, (1→4)-β-D-glucan. Due to the intra- and inter-molecular hydrogen bonds, cellulose contains rigid and insoluble microfibrils [14, 24]. Thus, native cellulose contains a large amount of crystalline microfibrils, which are less vulnerable to enzymatic attack.

The main microorganisms responsible for the degradation of cellulose are fungi, bacteria and acetinomycetes [21, 24]. They often interact synergistically, which results in the complete degradation of cellulose into carbon dioxide and water under aerobic conditions, and into carbon dioxide, methane and water under anaerobic conditions [24].

Cellulases

The cellulase enzymes are classified into three groups according to their catalyzed reactions; hydrolases, oxidases and phosphorylases [36]. The hydrolase enzymes which catalyze the hydrolysis of cellulose, are divided into 2 main groups based on the sites of cleavage in the polysaccharide chains. Endo-glucanases are responsible for random scissions and attack the cellulose chains in amorphous regions or at the surface of microfibrils. Exo-glucanases attack the non-reducing end of the cellulose chains by releasing cellobiose and in some cases, glucose [92]. The other hydrolytic enzyme is β-glucosidase, which is responsible for eliminating oligosaccharides, especially cellobiose [36, 92]. Presently the involvement of oxidative enzymes and phosphorylases in cellulose degradation is not well understood.

Although the exact mechanisms of the complicated cellulase systems are not clear yet, many studies have shown that enzymatic degradation is the result of their synergistic actions, which is susceptible to inhibition and induction processes [21, 36, 93]. The evidence of enzymatic action in a synergistic manner is based on the higher activity of the recombined enzymes than the sum of the individual activities [36]. There are possible two pathways of synergism among cellulase enzymes. The first synergism occurs between exo- and endo-glucanases. Newly formed non-reducing ends of the glucan chains from random cleavages of glycosidic linkages result from the action of endo-glucanases, which are later attracted by the exo-glucanases. The second pathway is the further degradation of oligosaccharides by the glucosidases. However, the activities of the endo- and exo-enzymes are inhibited by cellobiose. Wood and McCrae [126] showed that the enzymatic attacks on cotton cellulose was inhibited by the addition of 0.01% concentration of cellobiose. Since the β-glucosidase would prevent the accumulation of cellobiose, the glucosidase action could be a rate-determining step in cellulose degradation [92]. Sui et al. [94] found that the presence of sugar such as glucose, sucrose and lactose with cellulose could inhibit the microbial attracts on cellulose due to the preferential sugar substrates for the microorganisms. On the other hand, the cellobiose and the sugar could act sometimes as inducers, depending on their concentrations since cellulases are induced in the presence of cellulose.

Factors in Cellulolytic Degradation

The enzymatic activities on cellulosics are influenced by many factors like their morphological, chemical and physical structures [24, 93]. The length of cellulose molecules, i.e., the degree of polymerization(DP), in a fiber exists across a broad range. The higher the DP, the less susceptible the microbial attack expected. A cellulose fiber is a polycrystalline substance with both crystalline and amorphous regions, and a mesomorphous portion acting as a transition region. Due to its high density, the crystalline cellulose is highly resistant to enzymatic degradation. In that case, only surface and chain-end attacks are possible. Also, the degree of orientation influences the rate of microbial attack in an inverse manner.

The effects of the degree of substitution in each anhydroglucose unit on the level of microbial attack have been intensively studied [59, 84 93]. Siu, Reese and Levinson have showed that at least one substituent on every anhydroglucose unit resulted in complete resistance to microbial attack on cellulose due to the chemical blocking of one or more of the hydroxyl groups. Also, the increase in less biodegradable, non-cellulosic components such as lignin, waxes and antibiotics results in a increased resistance to microbial attack. The lignin-hemicellulose-cellulose complex is especially very stable and resistant to microbial attack [92].

Various mechanical disturbances and chemical modifications in a fiber affect enzymatic degradation [21, 24, 92]. Mechanical or chemical processes - like UV exposure, weathering, photochemical modification, grinding, and steam, mineral acids or alkali treatment - render the processed cellulosics more susceptible to microbial attack. However, Cooke [21] has showed that chemically modified cellulosic fabrics by resin treatments such as acetylation, cyanoethylation, phosphorylation and reaction with formaldehyde were resistant to degradation by cellulolytic microorganisms.

BIODEGRADABILITY OF COTTON AND CELLULOSE ACETATE FIBERS

Cotton

Cotton fibers are comprise of approximately 90% cellulose, and the cellulose molecules consist of a linear condensation polymer of β-1,4-linked-D-glucose. The degree of polymerization can be as high as 14,000 and the degree of crystallinity is usually formed to be in a range of 50 to 94% [21, 36, 66]. Even though cotton fibers have a high degree of crystallinity, due to the closely packed cellulose molecules, the large number of hydroxyl groups in cellulose make cotton fibers hydrophilic and therefore, susceptible to the growth of microorganisms [20]. Thus, it is generally accepted that unfinished cotton fibers are readily biodegradable by microbial attack.

Cellulose Acetate

Cellulose acetate (CA), a kind of cellulose ester, is produced by the hydrolysis of cellulose triacetate. Since the hydroxyl groups in CA are blocked and substituted by the acetyl groups in various degrees, the biodegradability of CA is less well established. Cellulose acetate is less hydrophilic than cotton, and enzymatic accessibility of CA is hindered. The greater the degree of substitution (acetylation) (DS) the farther away will the enzyme be positioned and therefore, the less effect there will be on biodegradation. According to Siu and Reese [84, 93], cellulose derivatives with a DS above 1.0 are not biodegradable at all. Siu [93, 94] concluded that all cellulose triacetate with different degrees of polymerization were completely resistant to microbial attack and that cellulose acetate with a DS of 1.0, 22.3% acetyl content, were not susceptible to enzymatic degradation. Since CA fibers usually have a degree of acetylation above 1.0, it is generally believed that cellulose acetate fibers are not biodegradable.

However, Abrams [8] found an enzyme in cellulolytic fungi which was capable of deacetylating insoluble cellobiose octaacetate, esterase. Reese [84] also showed that the enzyme was produced on soluble cellulose acetate with DS 0.76. The isolated esterase could hydrolyze cellulose to cellobiose and also could deacetylate short chains. With the addition of β-glucosidase into esterase filtrates, the soluble cellulose acetate was deacetylated and converted into a large amount of unsubstituted glucose. Another conflicting study, relating to the potential biodegradability of cellulose acetate, has been reported by Cantor and Mechalas [18] whereby they observed microbial attack on CA membranes with a DS of 2.5 with losses, in semipermeability.

Recently, CA films have been shown biodegradable in various environments [42, 43, 78]. In both aerobic compost and anaerobic bioreactors, CA films with the degree of substitution values of 1.7 and 2.5 were biodegradable. The bacterium Pseudomonas paucimobilis was isolated as the responsible agent for the microbial growth on the CA films.

Another promising results related to the potential biodegradability of CA films and fibers have come from studies by Buchanan, et al [17]. They showed that CA films with DS's of 1.7 and 2.5 were degraded by microorganisms, when interpreted through weight loss, decrease in thickness, and reduction of tensile strength. In the same study, cryogenically ground CA fibers, having a DS of 2.5, were deacetylated and degraded by inoculation from a mixed culture isolated from activated sludge. Degradation by microbial activity resulted in reduced DS and molecular weight. Also, in this study, carbon 14-labeled CA fibers with a DS of 1.6 and films having a DS of 1.85 were examined by monitoring radioactivity of ^{14}C carbon dioxide. The 45% of the label from the fibers was collected between 20 and 25 hours, while the 68% of the ^{14}C label was collected between 200 and 400 hours. The results provided evidence of the potential biodegradability of cellulose acetate, even though the rate of biodegradation is strongly influenced by the degree of substitution. In addition, it was suggested that the synergistic action of esterase and cellulase produced by microorganisms is responsible for the microbial attack on cellulose acetate. The suggested biodegradation mechanisms are based on the enzymatic attacks on the unsubstituted residues by cellulases and on the acetyl groups by esterases, which eventually leads to complete degradation CA in environment.

In summary, cotton fibers consisting of cellulose molecules are biodegradable due to their hydrophilic nature. In terms of the biodegradability of cellulose acetate, a modified cellulose, it is broadly believed that CA is not biodegradable, due to blocking the hydroxyl groups. Contradictory results have shown that microbial growth of CA films is not totally prevented. Hence, there may be opportunity for finding microbial activity for CA fiber use as a component of cellulosic nonwovens.

NONWOVENS IN GENERAL

Nonwoven fabrics are broadly defined as sheet or web structures made by mechanically, thermally or chemically bonding or entangling fibers or filaments, without the yarn preparation needed for weaving or knitting [6]. According to the ASTM definition [9], nonwoven fabric is a structure produced by mechanical, chemical, thermal or solvent means, and the combination thereof. Paper or fabrics which are woven, knitted, tufted, or those made by wool or other felting processes are not included. Therefore, a wide range of products exist from paper-like materials where fibers are held together by hydrogen bonding to woven-like structures [27]. The growth of the nonwoven industry observed in recent years is partially because of the variety of ways to produce and to engineer nonwoven fabrics.

The first patent for nonwoven technology was presented in 1936 [104]. A moderately bonded nonwoven fabric from carded web was made by printing a latex adhesive in a repetitive pattern. Another method of making nonwoven fabrics was introduced in 1942 [105]. A flexible bonded sheet from a random mixture of fusible and nonfusible fibers was produced when the web was subjected to chemical and physical conditions that softened the fusible fibers. In the US, these commercially produced fabrics were called nonwovens for the first time [6]. Early nonwovens were also made by bonding dry-laid webs with latex binders [28]. Since then, newly designed fibers for nonwovens and new web forming and bonding techniques have been developed. These have led to the production of a variety of modern nonwoven products, from disposables (diapers, surgical/medical supplies, wipers, filters) to durables (interlinings, automotive fabrics, home furnishing fabrics, geotextiles) [6, 27, 98].

The major components of nonwoven technology are generally divided into four categories: fiber (raw) materials, web forming methods, bonding methods and finishing (post-treatments). Various types of fibers or polymers, conventional or newly developed, can be used to manufacture nonwovens. A web is formed by arranging those materials into a layered collection of overlapping structure. Fibers are held together by the adhesive or frictional forces of chemical, thermal or mechanical bonds. Mechanical means and chemical additives are used in post-treatments to modify and impart strength, color, flame retardency, wettability, repellency and softness [6, 63].

WEB FORMING METHODS

One of the most important considerations in nonwoven technology is determining how to lay or arrange the fibers. The orientation or geometry of fiber webs is an important parameter of web characteristics, along with inherent properties of fibers or polymers, and web weight [27]. There are three ways to produce webs: dry-laid, wet-laid and polymer-laid.

Dry-Laid

In dry-laid processes, fibrous webs are formed either by carding or air-laying methods. In the carding method, staple fibers are separated mechanically into individual fibers by a carding machine consisting of opposing, moving beds of closely spaced needles, wires or metallic teeth that pull the clumps of fibers apart [27]. The webs from the carding process are anisotropic with a three-to-one orientation ratio in machine direction [6, 27]. To increase strength in the cross direction, air-doffing or cross-laying systems are used [6, 63]. In air-laying methods, the fibers are captured on a screen from an air stream transporting the fiber. With this method the orientation problem from the carding method is overcome [63, 112]. Shorter fibers are suitable for this method to produce uniform webs at high speed [27, 28].

Wet-Laid

In wet-laid processes, very short fibers, such as wood pulp, produce random or isotropic webs. The fibers are first dispersed in a large volume of water and then caught on a moving screen. The webs are formed, after the water is removed by rolls and heated drums [28]. This process is like a modified paper-making technique [6, 27, 28, 63].

Polymer-Laid

In polymer-laid processes, webs are formed directly from bulk polymer, in which the polymer is melted, extruded and drawn into filaments which are deposited in a random fashion onto belts as a continuous web [27]. Two useful and relatively new techniques utilizing direct laid processes are spunbond and meltblown processes. In spunbond processes, the extruded filaments from a shower nozzle (spinneret) are blown and spread on a moving belt to form a web [6]. The webs produced consist of filaments similar to standard textile fibers in terms of birefringency and diameter. In meltblown processes, the molten polymer is forced through very fine holes in a special die and blown with hot and high-velocity air [6, 27]. Very fine and irregular filaments of indeterminate lengths are formed into a web. Both spunbond and meltblown webs may be further bonded with rolls by heat and pressure.

BONDING METHODS

To consolidate webs, there are four main bonding categories: chemical (adhesive), mechanical, thermal and solvent bonding processes. Under these categories, there are many different web bonding techniques. Sometimes the combination of two or more techniques are employed. Therefore, the possibilities for new bonding methods are virtually limitless. The choice of the bonding process is based on the end-use applications, which determines the properties of the final nonwoven product. Economics and general application feasibility are important considerations [48].

Chemical (Adhesive) Bonding

In chemical bonding, the webs are consolidated by the use of binders as bonding agents. Typical binders are aqueous dispersions, solutions or solids in thermoplastic form [3]. The mechanism of adhesive bonding is not clearly understood but assumed to be directly related to shear or peel adhesion, or a combination of the two. Adhesion also can be obtained by the mechanical entrapment of fibers within encompassing bonds [27]. The oldest and the most common binders are latexes, fine dispersions of polymers in water. The latex bonding technology is old but still widely used because of its easy application, versatility and low cost. However, generally the high strength of final nonwoven fabrics can be obtained only by the sacrifice of softness.

The most common latexes made by emulsion polymerization are vinyl polymers and acrylic ester polymers. After the wet binders are applied to the webs, a capillary action forces the binders to migrate to the crossover points. Upon removal of water, a film or a spot weld is formed [3]. To improve the adhesion, strength and durability, a small amount of a cross-linking agent, such as *N*-methylolacrylamide, is added to the binder formulation. Latex binders can be applied to webs in several ways: by saturation, foam, spray or print bonding techniques [6, 27, 48, 63].

Mechanical Bonding

In mechanical bonding processes, bonding is achieved through frictional forces between fibers, which is proportional to the strength, but not to the softness, of the final nonwoven products [28]. There are three main ways to bond the webs through the mechanical means: needle punching, spunlacing and stitch bonding processes. In needle punching, the oldest mechanical process, fibers in a web are interlocked by rapidly reciprocating barbed needles which repeatedly pass in and out of the web [3, 27, 48, 61]. This process is relatively slow but very useful in producing strong, extensible and bulky heavy-weight nonwovens [3, 27].

In spunlacing (hydraulic entangling) processes, fibers are entangled by the use of water jets. This technique was developed by Du Pont in 1969 [107]. High pressure water jets penetrate fiber webs placed on porous backing belts, resulting in bonding by frictional force. The pattern of the final products depends on the shape of the backing materials. Strong, soft and planar nonwovens with low elastic recovery are produced [27, 48]. In stitch bonding, nonwoven fabrics are made by stitching yarns or filaments across fiber webs [6, 28, 48, 63]. The final products resemble woven or knitted textile fabrics.

The other two main bonding categories, thermal and solvent boding processes, which utilize heat, pressure or solvents, are examined in detail in following chapters.

THERMAL BONDING

Thermal bonding techniques are based on the use of heat in various ways. The bonding is achieved by the softening, or the fusion of heat-activated binders or thermoplastics in the webs (dry-laid, wet-laid or polymer-laid) at cross-over points. Thermal bonding can be accomplished by various methods, but two things are always required: thermoplastic materials and heat sources [27, 48]. Thermoplastic materials can be fibers, sheath polymers on bicomponent fibers, powder, granules, film or netting. Binders are softened or fused by heat, or heat combined with pressure. Four such bonding methods are: calendering, through-air oven transport, ultrasonic and radiant heat.

Along with the growing markets of disposable and durable nonwovens, thermal bonding processes play an important role in manufacturing. Today, the thermal bonding method is the leading process in nonwoven technology [6, 27] and offers many advantages over other bonding methods. High production speeds, availability of diverse raw materials, energy conservation (compared with chemical bonding which requires high energy consumption to dry wet-bonded products), the absence of chemical residuals, and success with a variety of conventional textile fibers (such as cotton), are reasons that make thermal bonding an attractive process in the manufacture of both durable and disposable nonwovens [25, 49, 76, 102, 118, 120].

FIBERS USED

In thermal bonding, thermoplastic meltable fibers are used in 10 to 100% of all final products [77]. In nonwovens of fiber blends, bonding fibers are called binder fibers, and structural fibers are called carrier or base fibers. Binder fibers can make up to 50% of the products. Those containing 10% of binder fibers are open, bulky and porous, but low in strength [49, 120].

Many different types of heat-fusible fibers are used as binders in nonwovens [49, 29]. First, any inherent thermoplastic fibers can be utilized for thermal bonding. Among them, polypropylene, polyester and polyamide fibers are commonly used. Second, modified fibers have been developed to improve processability by widening or lowering melting temperature range. For example, polypropylene polymer can be physically modified by orientation and obtained by slightly chemical polymer variation. Polyester and copolymer polyesters are available with different ranges of melting temperatures. Third, bicomponent fibers also have been developed for lowering processing temperatures and preserving fiber integrity. High melting or non-thermoplastic polymers are used for the core of the fiber, and lower melting polymers cover the core either as a skin or in a side by side configuration. The main advantage of bicomponent fibers as a binder is the preservation of strength by the core component [29]. Examples are polypropylene-core and polyethylene-skin fibers, high- and low-melting nylons, and high- and low-melting polyesters. The overall effect is that the selection of a binder influences the properties of the final product as well as the choice of bonding process [49].

BONDING OPTIONS

Thermal bonding methods are generally divided into four categories depending on the type of heat sources [25, 27, 49]. Among them, hot-roll calendering is the leading process, determining more than 60% of thermally bonded products [49]. The detailed review of thermal calendering, the most widely practiced method [25], will be covered in the next chapter.

Thermal Calendering

Thermal calendering methods are divided into two subcategories depending on the bonding area or pattern; area-bond hot calendering and point-bond hot calendering. The area-bond hot calendering process involves a calender consisting of smooth surfaced rolls. Thin, inextensible, stiff and strong nonwovens are produced by this method. On the other hand, the point-bond hot calendering is the main thermal bonding method able to produce a wide range of nonwoven fabrics in terms of properties. These properties are sensitive to the size and the density of the bond points or patterns, as well as processing conditions. This process involves a calender with two rolls, one of which is a smooth roll and the other a patterned or embossed roll.

Through-Air Oven Bonding

This bonding method employs hot air to soften the binder materials, by circulating or drawing the hot air through the webs. Bulky, open, soft, strong, extensible, breathable and absorbent nonwoven fabrics are made by this method. Typical examples include medium or high basis-weight nonwovens such as geotextiles and carpet backing. When followed by cold calendering, a compact, tough fabric is produced. It will be softer than one produced by area-bond or smooth-roll calendering. In this method, the temperature of the webs can be accurately controlled, since heat is transferred in a uniform and rapid manner. Bicomponent fibers bond especially well in this method, since the controlled temperature melts only sheath materials, protecting core polymers from thermal damage.

Ultrasonic Bonding

This process involves the use of ultrasonic energy, or mechanical vibratory energy, at frequencies beyond hearing [3]. The rapidly alternating compressive forces result in intermolecular mechanical stress build-up within the thermoplastics. This softens the binder materials. Strong, soft and breathable nonwovens are produced.

Radiant-Heat Bonding

This process employs infrared radiation as a heat source to soften the binder materials. This economical and versatile technique is essentially useful for powder-bonded or light-basis-weight nonwoven fabrics. Soft, open and absorbable nonwovens are produced.

THERMAL CALENDERING

Among all thermal bonding processes, thermal calendering, especially point bonding, is the predominantly used method. This process allows the production of nonwoven fabrics having a wide range of basis weights, a variety of patterns and bond dimensions [25, 58]. In the calendering processes, heat is applied to a web as it passes between the nip of two heated rolls or cylinders. Point-bond, or embossing, calendering processes are widely used to produce disposable nonwovens such as diapers, sanitary and medical products. In this method, two calendering rolls, consisting of a dot- or diamond-patterned roll and a smooth roll, are used [29, 49]. Bonded areas which range between 10 to 40 %, typically in a range of 15 to 25 % [58], are compressed, whereas unbonded areas provide openness and bulkiness to the final products.

Factors That Effects Thermally Processed Webs

Many factors must be considered when manufacturing nonwoven fabrics by thermal calendering. Binder materials, basis weight and processing parameters influence the properties of nonwoven fabrics and determine their end-use applications.

Fiber Properties

Fiber properties largely determine the ultimate properties of nonwoven fabrics. Important fiber factors include length, diameter, density, crimp, cross section, spin finish, delustering and draw ratio [27, 120]. Inherent polymer properties of fibers, such as chemical composition, molecular weight, melting point, glass transition temperature, viscosity and surface tension, are also significant contribution. These inherent properties govern the interfacial bonding between the binder and the base fiber [25]. For example, compatibility between the binder and base fiber can be expressed by the solubility parameter, wherein small differences in the solubility parameters are expected to result in stronger interfacial bonding and, hence, higher mechanical strength [91]. Some studies have been carried out to examine the relationship between fiber morphology and the properties of nonwoven fabrics made by area-bond hot calendering. One study showed that less oriented fibers with low birefringence resulted in nonwoven fabrics with high tensile strength and flexural rigidity because of the promoted fusion between fibers [121].

Basis Weight

Web weight limits the effectiveness of processing parameters, product properties and end-uses [31]. Webs are divided into light, medium and heavy weight. Webs. Light weight webs in the range of 15 to 80 g/m^2 are successfully bonded with thermal calendering methods [120]. Examples of use of such light-weight webs are coverstocks and interlinings. Medium and heavy weight webs in the range of 80 to

350 g/m^2 are used for filtration webs, geotextiles, carpet backing, and coating substrates for plastics or bitume. In most cases, the upper limit of basis weight for contact thermal bonding is approximately 150 g/m^2. To obtain uniform bonding throughout the web thickness, heavier webs are usually thermally bonded by a hot-air flow, and even calendering is sometimes followed by air-flow heating [77].

Processing Parameters

Several factors control the thermal calendering process. Primary parameters are roll temperature, nip roll pressure, and speed. These influence the heat transfer mechanism (mainly conduction) as well as the properties of the bonded products [25, 31, 44, 49, 77].

The temperature of either a patterned or smooth roll is the most important factor to be selected and controlled. The relationship between the bonding temperature and the tensile strength of thermally nonwoven products has been studied intensively. Generally, the strengths increase with bonding temperatures up to certain point under, otherwise, fixed processing conditions. Above some critical bonding temperature, the strength decreases due to the loss of fiber integrity and film formation at high temperatures [23, 58], as well as the reduction in load transfer from fibers to film [77]. One study has suggested that the roll temperatures should be, at least, near the softening temperatures for adequate strength of fabric to occur [77]. In most operation, the temperature of the patterned roll is around the melting temperature of binder fibers, and the temperature of the smooth roll is slightly lower than that of the engraved roll to prevent stiff surface [49, 58].

Nip pressure is another factor that must be taken into account for thermal bonding of light- and heavy-weight webs, at least, in terms of adjustments in tensile strength, elasticity and handle [49, 77]. Higher pressure normally results in higher strength by compression, which forces the binder to flow in and around the base fibers. However, there is a level at which the increase of nip pressure is detrimental. Nip pressure that is too high leads to lower tensile strength due to flattened fibers and disturbance of the melting point of binder fibers. Some studies [23, 33], however, have provided somewhat different conclusions about the effects of nip pressure on strength. It was suggested that although strengths increased with nip pressure, the effect of the pressure is almost always less significant than that of other parameters such as temperature and speed. Therefore, the effect of pressure on the bonding properties of nonwovens is related to bonding temperatures, as well as other processing parameters. These effects are not yet clearly understood. However, heat generated from roll temperatures and pressure is sufficient enough to cause the flow of binder materials, which contributes to the formation of a good bonding [119].

Production speed or surface speed of rolls determines not only productivity but also the properties of the final products. A sufficient contact time, influenced by the production speed and roll diameter, is required to transfer the heat through the webs [25, 77]. In typical thermal calendering lines, the contact time is in the order of milliseconds. Along with roll temperature and nip pressure, the feed speed determines the degree of bonding, and generally, the higher the speed the lower the strength of the fabric.

Secondary parameters in thermal calendering processes that influence the properties of the nonwoven fabrics are calender pattern, quench rate after calendering and roll diameter [25, 44, 49, 77]. The pattern area and the height of lands on the roll affect the thickness, softness and strength of the bonded nonwoven fabrics. The lower the quench rate and the slower the cooling and recrystallization, the higher the strength [91].

Optimization

Process optimization in thermal calendering is complicated and tedious, but necessary to understanding the bonding behavior of raw materials, as well as to obtaining desired properties of the resulted nonwoven fabrics. Difficulties in optimization arise from the many factors and interactions involved in the bonding process. Normally in production lines, fiber selection, speed of operation are fairly well fixed. Then, the temperature and nip pressure are adjusted for selected properties desired of the nonwoven fabrics [25, 58, 76]. In modeling procedures, there are three approaches to process optimization.

One approach is to generate thermal profiles of bonded nonwoven fabrics by solving approximate heat transfer equations. From the thermal profiles, the effect of processing parameters on the degrees of heat transfer through a web can be evaluated.

However, in this approach, complicated interactions of the many factors in calendering are simplified, which is often treated as negligible in the list of assumptions. One study [31] solved the conduction equation by using the finite-element technique. The effects of different calendering speeds, blend levels and basis weights on bonding properties were predicted, and the suggestion on preheating to improve bonding was made.

The second approach is to interpret raw and empirical data by examining trend lines in graphs. This approach should be most accurate, if the full range of processing parameters are covered. This is virtually impossible as well as time-consuming, even though it is used in the majority of studies.

The third approach is the use of multivariate or multiresponse statistical analysis methods. Here, the process parameters or the several selected properties can be optimized simultaneously in a given fiber or production system. Some studies [52, 125] have been done to investigate the relationship between fiber and processing factors and various properties of thermally bonded nonwoven fabrics using statistical methods. Through a multivariate approach, one of each properties (responses) of nonwoven fabrics was studied as functions (multivariates), i.e., fiber properties, web formation and processing variables [125]. The results indicated that fiber properties such as denier and certain type of binder fibers, bonding temperature and pressure significantly influenced the mechanical properties of the nonwoven fabrics studied.

The higher the bonding temperature and pressure, the stronger, stiffer and denser were the nonwoven fabrics.

A more thorough study including a multiresponse optimization procedure of thermally bonded nonwoven fabrics of cotton/synthetic fiber blends has been conducted [52]. Physical properties were studied by a nested analyses of variance method and the statistics showed significant differences among fiber types and among bonding temperatures. Through a univariate-mean-comparison test, the best fabric type for each physical property was determined. Regression analysis and least-squares-mean tests were performed to determine significant correlations between fabric thickness and bonding temperature. By a multiresponse optimization procedure, the optimal bonding temperature for each fiber type was identified in the content of stronger, softer, permeable and extensible nonwoven fabrics.

THERMAL BONDING OF COTTON AS A BASE FIBER

Cotton Fiber Properties

Cotton is a natural plant fiber composed of cellulose. The cellulose is almost 100% crystalline, with a fibrillar structure [75]. The constituents of raw cotton are 85.5 % cellulose, 0.5% oil and wax, 5.0% proteins, pectoses and coloring-matter, 1.0% mineral water, and 8.0% moisture [101]. The fine structure of cotton consists of an outer cuticle (skin), a primary wall, a secondary wall, and a central core (lumen). In the longitudinal view, cotton has a ribbonlike shape with twist (convolutions) at irregular intervals [55]. The main characteristics determining the usage quality of cotton fibers are length, fineness, uniformity and color. The length and the width of cotton fibers vary from 12 to 20 microns, and from ½ inch to1¼ inch [55, 124].

Cotton has a tenacity of 3.0 to 5.0 g/denier or 0.19 to 0.45 N/tex. The strength increase when wet. Also, cotton is relatively inelastic and inextensible, with an breaking elongation of 3 to 7 %. The resiliency of cotton is low [22, 55, 75]. It is hydrophilic and absorbent fiber, with a moisture regain of 8.5%. Thermally, cotton is stable up to 150°C (300°F), but long exposure to dry heat will cause the fiber decomposition [55, 101]. The thermal conductivity of cotton, 0.50 W/(m·K), is relatively high, and the fiber, compared with other fibers, is regarded as a cool fiber with a good conductor of heat [22, 32].

Generally, cotton is highly resistant to alkalis and most organic solvents, but strong acids and prolonged exposure to sunlight do degrade cotton. In addition, cotton is highly resistant to insects, but degraded easily by fungi and bacteria [22, 55].

Cotton Fibers in Nonwoven Fabrics

The predominate fibers used in nonwoven fabrics are polyester, polypropylene and rayon, but cotton consumption in nonwoven fabrics such as coverstocks, pre-moistened wipes and surgical gowns continues to grow at an annual growth rate of nearly 5% [68]. Cotton fiber has gained a foot-hold in nonwoven markets because of its natural comfort properties and biodegradability, the development of the processability of bleached cotton, and a consumer preference for cotton [53, 54, 64, 68, 80, 81, 96]. Nonwoven fabrics made from cotton fibers have improved properties over other choices of fiber such as absorbency, hand and biodegradability. According to one survey study [68], consumers recognized the cotton labeling, and they strongly prefer cotton in personal care products such as wipes, diapers and feminine hygiene products. The quality and quantity of bleached and scoured cotton fibers have been improved significantly, along with major studies in the development in processability of cotton fibers for nonwoven processing. Fiber finish have been developed for cotton, like buyoxyethyl stearate. They provide lubricity without interfering with fiber absorbency.

In marketing cotton nonwoven fabrics, biodegradability, comfort and absorbent properties have all attractive appeal to consumers. The one drawback is the high price of cotton fibers compared to synthetic fibers. This applies also to rayon, its biggest competitor. Therefore, the success of cotton nonwoven fabrics depends on high quality products and innovative developments.

Up until now, products made from cotton fibers by needlepunching, spunlacing (hydroentanglement), and thermal bonding are diversified into products like cotton puffs, swabs, medical products, wipes, home furnishing and filtration products [19, 54, 74]. From the hydroentanglement bonding system, strong, durable and absorbent cotton nonwoven fabrics are produced. On the other hand, needlepunch bonding of cotton fiber webs provide highly efficient filter media due to the irregular fiber shape and absorbency with increased strength in the wet state [74]. One study has shown that oil sorption capacities of the needlepunched cotton-containing sorbents were slightly greater than those of 100% polypropylene sorbents [19]. Blending with a small amount of polypropylene fibers increased the mechanical strength, but decreased the oil sorption capacities. The study found that the oil sorption mechanism of cotton fiber was a combination of adsorption on the fiber surface and of capillary action through its lumen. The mechanism of polypropylene fiber was controlled solely by the capillary bridges between fibers.

Several researches were conducted to investigate the thermal bonding of blends of cotton and low melt thermoplastic fibers. Thermal bonding is a favored web consolidation method for cotton fiber blends because it does not interfere greatly with the important capillary and tactile characteristics of cotton fibers required in healthcare products [124].

Cotton Fibers in Thermal Bonding

Thermal bonding techniques provide several advantages over other bonding techniques, such as environmental safety, cleanliness, lower energy cost and the variety of fiber selections [31]. However, cotton fibers are not thermoplastic and can not be melted. Therefore, heat-fusible binders are necessary to produce fabric integrity. Furthermore, there is very little technical information available about the thermal bonding of cotton with thermal binding fiber [73]. Only a few thermoplastic fibers such as polyester, polypropylene and bicomponent fibers have been studied in any detail.

Properties of Cotton Fibers Influence Thermal Bonding

Some research has been carried out to evaluate the properties of cotton as carrier or base fibers in thermal bonding processes [32, 33]. The surface properties of cotton, especially, determine its processability and adhesive behavior.

Web quality and processability for opening, blending and carding cotton fibers with different surface treatments has been examined to draw some understanding of relationship to uniformity, the number of neps per gram of fiber and shear friction [72]. Raw, gray (mechanically cleaned) and bleached cotton fibers without finishes were selected. The bleached cotton fiber were also treated to 1% BES (butoxyethylstearate), 1% Sonostate 668 and 0.5% Dow 111 silicone finishes. The reflectance data for the blended cotton/black-dyed polypropylene indicated that the most uniform web was obtained by opening and blending before carding. The results from the nep counts and the shear friction data showed that bleached cotton fibers without any finish were easily damaged by processing, which resulted in the high number of neps per gram. The natural waxes in raw or gray cotton and the lubricants in the finished bleached cotton improved significantly the processability of the fibers.

The effect of adhesion behavior between cotton fibers and thermoplastic fibers on the tensile strength of cotton and melted polyester was studied with cotton fibers having different surface properties [32]. Raw, gray (mechanically cleaned), and scoured and bleached cotton fibers, with and without finishes (butoxyethylstearate, Sonostate 668 and Dow 111) were selected as comparative samples. They were blended and thermally bonded with low melting polyester. The result indicated that the scoured bleached cotton fibers containing no trash, natural waxes or finishes provided the highest level of tensile properties in the thermally-bonded nonwovens. The raw and gray cotton fibers due to its trash or natural waxes resulted in the poorest tensile behavior of the nonwovens. Among the different finished, scoured and bleached cotton fibers, the butoxyethylstearate-treated cotton provided highest adhesion with the melted polyester, as measured by the high fabric strength. The cotton treated with the Dow 111 finish produced a fabric with low strength. This study showed the importance of surface properties of cotton fibers on the adhesion with the thermoplastic fibers in thermal bonding processes.

Thermal Bonding of Cotton/Synthetic Blends

Studies were conducted to examine cotton and polyester blend nonwovens [32, 33, 34]. Low melting polyester copolymer, Kodel-438, was blended with scoured and bleached commodity cotton fibers and bonded by point-bond calendering. The total weight of the web was 160 g/m^2. The blend levels were 50/50, 65/35, 75/25 and 85/15, cotton and polyester, respectively. Those fibers were opened separately and blended just before carding. Three temperatures of 408, 422 and 436 K for diamond-patterned (25% of surface area) rolls, were selected. These were just above the melting temperature of the polyester (403 K). The temperature of the smooth roll was fixed at 394 K. Three different crush edge pressures were selected - 65, 130 and 195 kN/m - and two different feed roll speeds were examined - 10 and 20 m/min. The results showed that the temperatures of rolls and delivery speed has a significant influence on the tensile behavior and flexural rigidity of blended nonwovens. The faster delivery speed resulted in reduced bonding between the base fiber, cotton, and binder fiber. The study also showed that the nip pressure changes had little effect on the strength of blended nonwovens.

Polypropylene fibers were also examined as a binder in thermally calendered cotton-containing nonwovens [72, 73]. Blends of 80/20, 70/30, 50/50 and 25/75 cotton/polypropylene nonwovens were prepared by opening and blending before carding. Nip pressure (28 kN/m) and delivery speed (100 ft/min) were held constant. Different weights were prepared - 40, 60 and 80 g/m^2 - and different bonding temperatures were selected to seek an optimum bonding condition in the range of 130 to 165 °C. As the cotton content was increased, it was necessary to raise the bonding temperature. Higher bonding temperatures resulted in higher breaking strength, while higher web weight resulted in greater stiffness. In a given processing condition, an increase in cotton content resulted in a decrease in strength, but an increase in hand (less stiffness). Also, cotton blends provided a greater cover factor, faster wicking (greater absorbency), biodegradability, comfort and consumer appeal.

The possibility of using bicomponent fibers in thermal calendering of cotton blends has also been studied [35]. Two sheath/core bicomponent fibers, polyethylene/polyester and polyester/polyester, were blended and calendered with cotton fibers to fabricate 160 g/m^2 nonwoven fabrics with a blend ratio of 50/50. The effects of several processing factors and sample preparation methods on the physical properties of the nonwoven fabrics were evaluated. The results indicated that the physical properties of the nonwoven fabrics were highly dependent on bonding temperatures. In addition, the sample preparation method, the outside layering of thermoplastic fibers with cotton sandwiched in-between was more effective in the bonding behavior when compared with homogeneous blending.

A more complete study [52] evaluated nine physical properties of thermobonded nonwoven fabrics made from blends of cotton and 20 synthetic binder fibers, including nylon, polyester, polypropylene, bicomponent fibers and biconstituent fibers. A basis weight of 60 g/m^2, a blend ratio of 70/30 cotton/synthetic fibers, a constant nip pressure of 41.52 N/mm and a constant production speed of 30 mm/s were used for fabrication. Several statistical methods, such as nested analyses of variance, univariate-mean-comparison tests, a multiresponse optimization procedure, regression analyses and least-squares mean-comparison tests, were employed to examine the effects of bonding temperatures, to determine the optimal bonding temperatures and to identify relationships among variables. The results indicated that all the physical properties were significantly influenced by the bonding temperatures. For each binder-fiber type, an optimal bonding temperature was determined. Also, there were significant relationships between machine direction and cross direction of breaking strengths, elongations, and stiffness. In addition, polypropylene fibers and bicomponent fibers with a polypropylene sheath were found to be the best binder fibers in nonwoven fabrics containing cotton base fiber.

A well bonded fabric can be produced usually by increasing temperatures of rolls and/or crush nip pressure, while decreasing delivery speed. However, there are several limits to this general pattern. The trends, at some point, actually reverse. Stiffness increases and strength may drop. Lower delivery speeds are detrimental to production rate [31, 32, 73, 77]. At higher temperatures, the degree of bonding is normally enhanced, and the complete melting of thermoplastic fibers is possible. The bonding area becomes film-like, which results in harsh hand, stiffness and even a decrease in tensile strength. Also, since the cotton fibers become brittle at temperatures above 200°C, there is an optimum temperature range.

Slower calendering speeds may result in crushed fibers and a brittle bonding point. This results in weaker fabrics due to the longer contact time and heating period [31, 73]. The brittle bonding sites fail easily under loading as a result of the difficulty in tensile load sharing throughout the fabrics. Nip pressure that is too high may result in a reduction of strength [77]. Since the fibers are completely flattened and the directions are intensively changed, the sharp edges at the borders of the bonding point fail under strain. Light weight webs tend to bond more completely due to complete heat transfer. However, incomplete bonding can occur in medium or heavy weight webs due to low heat transfer throughout the web. This is especially in the middle of the web thickness. An increase in the amount of binder fiber in nonwovens from fiber blends results in an increased degree of bonding. However, the soft hand and the unique properties of the base fiber would be sacrificed. For all practical purposes, there should be optimum process conditions and fiber blend to produce a fabric with a special end-use by controlling the degree of bonding, stiffness, strength, web weight and blend ratio.

To produce a well bonded nonwoven fabric without sacrificing softness and strength, pre-heating or solvent-pretreatment has been suggested. The usefulness of pre-heating has been predicted by a one-dimensional finite element model [31]. There is a limitation to increasing the temperature of calendering rolls due to the brittleness of cotton fibers at high temperatures. For high cotton content or heavy weight webs, pre-heating the webs before calendering increases its starting or ambient temperature, which helps complete the heat transfer throughout the webs. One possible method suggested is the circulation of hot air (60°C and above for cotton/polypropylene webs) through the webs before they reach the calendering rolls.

SOLVENT BONDING

SOLVENT-POLYMER INTERACTION

The strength of the intermolecular forces between polymer molecules is an important factor in determining physical properties of polymers such as glass transition temperatures, which is related to cohesive energy density (CED), the molar energy of vaporization per volume [90]. The CED values are used to predict solubility (δ). The interaction between polymer and solvent is determined by the ability of the solvent to break the interactive forces between polymer molecules, and replace them with polymer-solvent interactions. The interactive forces between polymer molecules are dispersion (London) forces, polar forces, induction forces, and hydrogen bonding [90, 122].

Solution Processes

The solution process is a slow process that essentially occurs in two steps [15, 90]. The first step is a slow swelling process, called solvation, in which solvent molecules diffuse into the polymer molecules to produce a swollen gel based on their solubility values. This stage of solution process is applicable mainly for polymers having high intermolecular forces such as crosslinking, crystallinity and strong hydrogen bonding. In the second step, polymers, such as branched polymers, dissolve and the swollen gel disintegrates into a solution.

The solution process in polymer systems can be quite complicated due to the size differences between polymer and solvent molecules, the viscosity of the polymer, the effects of the topology and molecular weight of polymers, and the conditions of the solution process, such as the types of solvents and temperatures [15]. In general, crosslinked polymers swell rather than dissolve by solvents. Polymers with high crystallinity, especially nonpolar ones such as polyethylene and polytetrafluroethylene, do not dissolve under normal conditions, but they can dissolve at temperatures near their melting points. In the presence of solvents, melting temperatures are actually depressed. Branched polymers are more easily soluble than linear polymers having the same chemical type and molecular weight. The potential energy differences of polymers, from the ordered crystalline state to the solvate state, is much higher than from the noncrystalline state to the solvate state. Semicrystalline and oriented polymers, such as fibers, swell by solvents rather than dissolve because of the inhibited segmental motion and limited network chain separation [122]. The effect of temperature can be described by the Flory temperature θ [15, 90], where the θ temperature is the lowest temperature at which a polymer is completely miscible with a solvent, and the chain is in an unperturbed dimension. Therefore, the polymer chain extends above the θ temperature.

Solubility Parameter Theory

A solution process, governed by the free energy of mixing, occurs when the free energy of mixing (ΔG), the driving force in the process, is negative. The Gibbs free energy equation for constant temperature is as follows:

$$\Delta G = \Delta H - T\Delta S$$

where ΔH = change in enthalpy

ΔS = change in entropy

and ΔG = change in free energy.

ΔG is negative for a spontaneous process, and the entropy of mixing ΔS is always positive due to the increased polymer chain mobility of the polymer-solvent system. Therefore, the negativity of ΔG depends on the sign and magnitude of the enthalpy term ΔH [15, 16, 90, 122]. For example, when the entropy term increases in a solution process, a negative or lower value of enthalpy term is required for solution to progress.

Hildebrand and Scott [45], in 1950, defined the solubility parameter δ as the square root of the cohesive energy density, and the enthalpy term ΔH, the heat of mixing per unit volume, was approximated by the relationship as [39],

$$\Delta H = V(\delta_1 - \delta_2)^2\phi_1\phi_2$$

where V = total volume of the mixture

δ_1, δ_2 = solubility parameters of solute and solvent

and ϕ_1, ϕ_2 = volume fractions of the two components in the mixture.

Since the cohesive energy density, i.e., the energy required to overcome all the molecular forces in one mol of the substance, is defined as the heat of vaporization per unit volume, then the solubility parameter can be written as follows:

$$\delta = (\Delta E/V)^{1/2}$$

where $\Delta E/V$ = cohesive energy density, the heat of vaporization per unit volume

ΔE = energy of evaporation

and V = molar volume.

Therefore, the Hildebrand heat-of-mixing term can be rewritten as follows:

$$\Delta H = V[(\Delta E_1/V_1)^{1/2} - (\Delta E_2/V_2)^{1/2}]^2\phi_1\phi_2$$

where V = total volume of the mixture

ΔE_1, ΔE_2 = energies of evaporation of the two components

V_1, V_2 = molar volumes of the two components

and ϕ_1, ϕ_2 = volume fractions of the two components in the mixture.

Since the heat of mixing a solute and a solvent is proportional to the square of the differences in solubility parameters, if the differences are close to zero, the enthalpy term approaches zero and the solution process would be expected to proceed. This solubility parameter concept predicts the heat of mixing for liquids and nonpolar amorphous polymers. In addition, Hildebrand suggested that any nonpolar amorphous polymer would dissolve in a liquid or a mixture of liquids having the differences of their solubility parameters less than 1.8 Hildebrands (1 H = 1 $(\text{cal}\cdot\text{cm}^{-3})^{0.5}$) [45, 90].

For polar polymers, the solution process is expected, also, based on the similarity of the solubility parameters and the degree of hydrogen bonding (low, medium and poor) of the polymers and solvents [16]. Hydrogen bonding capacities are also classified in the Polymer Handbook [16] as poor (δ_p), moderate (δ_m) and strong (δ_s) hydrogen bonding solvents. Another consideration of the polymer-solvent systems was suggested by Hansen in while the cohesive energy arises from dispersive (d), polar (p, permanent dipole-dipole interactions) and hydrogen bonding (h). Hansen's solubility parameters of liquids are based on the following equation [16, 122],

$$\delta^2 = \delta_d^{\,2} + \delta_p^{\,2} + \delta_h^{\,2}.$$

Polymer-Solvent Interaction Parameter Theory

Another thermodynamical approach to predict the interaction between polymers and solvents was developed by Flory [38] and Huggins [51]. The Flory-Huggins theory for the free energy of mixing a polymer solution is based on the equation,

$$\Delta G = RT(N_1 \ln\phi_1 + N_2 \ln\phi_2 + \mathcal{X}_1 N_1 \phi_2)$$

where N_1, N_2 = number of moles of solvent and polymer

ϕ_1, ϕ_2 = volume fractions of solvent and polymer

and $\mathcal{X}_1$ = Flory interaction parameter.

The value $\mathcal{X}_1$ is a measure of the interaction energy between solvent and polymer.

Also, Huggins calculated the enthalpy term of χ_H, and χ_1 can be obtained from the solubility parameters [15, 16, 122].

$$\chi_1 = \chi_S + \chi_H$$

where χ_S = polymer-solvent interaction of an entropy term

χ_H = polymer-solvent interaction for an enthalpy term, $V_1/RT(\delta_1^2 - \delta_2^2)^2$.

For a nonpolar system, $\chi_S = 0.34$. Consequently, $\chi_1 = 0.34 + V_1/RT(\delta_1^2 - \delta_2^2)^2$.

To predict the dissolution of a polymer by a given solvent, the Flory-Higgins equation was derived with respect to ϕ_2 equal zero. This approach led to a critical value of the interaction parameter, χ_c which could be obtained from the molecular weight value, M, of the polymer. This critical value of the interaction parameter is,

$$\chi_c = ½\,(1 + 1/M^{1/2})^2.$$

For the polymer-solvent miscibility, χ_1 should be less than the χ_c value. Since the χ_c value for very high molecular weight polymer is 0.5, χ_1 should be less than 0.5 for complete miscibility of a polymer and a solvent over the entire composition range.

Diffusion Theory

The kinetics of diffusion of a solvent into a polymer can be divided into two types based on differences in diffusion behavior [122]. The first one is the Fickian diffusion of a small penetrant molecule having low thermodynamical interaction with the polymer. The rate of diffusion depends on the free volume, segmental mobility and topology of polymers. The second diffusion process is non-Fickian, or anomalous, diffusion for large penetrant molecules with a strong interaction with the polymer. After the highly interactive solvent diffuses into the polymer, the segmental mobility of the polymer is increased, and in some cases the polymer structure is rearranged. This anomalous diffusion is a time-dependent polymer behavior, in which the solvent concentration equilibrium in the polymer is achieved at a finite rate rather than instaneously. One study of the kinetics of n-hexane vapor sorption on polystyrene film showed the time-dependent diffusion sorption behavior [47]. The result indicated a sharp penetration of the rubbery and outer domain of the polymer at constant velocity at the beginning stage of diffusion, and the weight gain of the polymer, which was linear with time over the first considerable penetration range. The relaxation of stresses produced from swelling is the rate-determining factor in this diffusion.

SOLVENT EFFECTS ON FIBERS

Effects on Fiber Structure

Effects on Melting Temperature

Effects of solvent on melting temperature depend on the degree of interaction between the polymer and the solvent, as well as the amount of solvent [122]. The melting temperature of the polymer is depressed by the solvent. A quantitative study of the melting temperature of a semicrystalline polymer and the amount of diluent in the amorphous phase was done by Flory [37]. The following equation is based on the assumption that the diluent does not penetrate the crystalline phase of semicrystalline fibers.

$$1/T_m - 1/T_m^o = RV_2/\Delta H_2 V_1(\phi_1 - \chi_1\phi_1^2)$$

where T_m^o = melting temperature without diluent

V_1, V_2 = molar volumes of diluent and polymer repeat unit

χ_1 = Flory-Huggins interaction parameter

ϕ_1 = Volume fraction of diluent

and ΔH_2 = heat of fusion per mol of repeat unit of the polymer.

Effects on Glass Transition Temperature

The glass transition temperature of the polymer with a solvent is depressed since the solvent penetrates into the noncrystalline domains of the polymer as a plasticizer, breaks the intermolecular bonds by providing a lubrication action, and then results in enhanced segmental mobility of the polymer [122]. One study of the depressing of glass transition temperature by diluent was based on diluent uptake by the polymer. The new glass transition temperature became [41],

$$T_g = (KW_1T_{g1} + W_2T_{g2})/(KW_1 + W_2)$$

where W_1, W_2 = weight fractions of the polymer and solvent

T_{g1}, T_{g2} = glass transition temperatures of the two components and $K = \Delta\beta_2/\Delta\beta_1$, where β = expansion coefficients of melt and glass of the two components.

Solvent Induced Crystallization (SINC)

In the presence of a solvent, crystallization occurs due to the increased segmental mobility of the polymer in a swollen state. During solvent-induced crystallization, the nucleation rate of the polymer is very high resulting in high concentrations of small crystallites in the unoriented, amorphous phase [62, 122, 123].

Solvent Cavitation

The solvent may cause cavitation, externally or internally, of the polymer due to anisotropic swelling forces, solvent stress cracking or crazing, and localized spherulite formation during solvent induced crystallization [122]. Solvent-induced surface cavitation results in high surface to volume ratios, which can be useful in applications that require improved bonding adhesion, moisture transport, and slow decomposition properties [40, 122].

Shrinkage

In the presence of a solvent, the glass transition temperature of a polymer is lowered because of the increased segmental mobility of the polymer which leads to the molecular relaxation of orientational strains in the direction of the principal axis of a fiber. The degrees of swelling and shrinkage induced by a solvent depend on the solubility parameter, an indicator of the degree of polymer and solvent interaction [57, 85, 86, 122].

Effects on Fiber Property

An absorption and swelling process usually occurs for textile fibers of semicrystalline and oriented polymers rather than complete solubilization, unless highly interactive solvents are involved. The penetration of solvent into the amorphous region of a polymer results in swelling, a breakdown of intermolecular bonds, and an increased segmental mobility of the polymer chain. It is this while leads to the lowering of the glass transition temperature [87, 122].

The effects on the physical properties of swollen polyester fibers by different solvents have been studied [69, 70, 123]. From the load-elongation curves after solvent treatments, the breaking force was relatively unaffected by those solvent treatments. However, Young's modulus and yield stress of the solvent-swollen fibers were sharply lowered in the highly swollen structures, resulting in a rubbery behavior in the initial deformation. Elongations of those fibers depended on the types of solvents, but overall elongations increased by solvent treatments. The study of polyester fibers with dimethylformamide (DMF) indicated that the swelling by the solvent resulted in the relaxation of built-in stresses and in fiber shrinkage [86]. These also resulted melting and recrystallinization of imperfect crystalline domains, low initial modulus and yield stress, and high extensibility. In addition, an open structure of microvoids and voids formed upon removal of the solvent, indicating that the structure did not completely collapse.

In summary, solvent treatments did not affect values in breaking strength, while the extensibility values varied widely. The swollen fiber structures resulted in a rubbery behavior in the initial deformation range, and the disorientation of the noncrystalline domains resulted in fiber shrinkage. Upon removal of the solvent, the original properties of polyester yarns were partially restored.

Solvent Pretreatment in Dyeing and Finishing

To improve processability, solvents have been utilized as carriers in the dying and finishing processes. Due to the lowering of the glass transition temperatures by increased segmental mobility of the polymeric materials, and due to the open structures by void formation, dye and chemical diffusion kinetics are improved [122]. For example, Nomex® fibers with high T_g and high crystallinity can be dyed after solvent treatments. The solvent pretreatments broke down hydrogen bonds and increased the mobility of polymer segments, which led to the loosening of structure. The improvements in dyeability can be explained by the easy dye penetration due to the loss of polymer chain orientation and due to void formation [70, 71]. The effect of the solvent-polymer interaction causes not only swelling, disorientation and shrinkage, but also crystalline growth in the solvent swollen state [46]. Therefore, an increased dye diffusion rate is due to the opening of fiber structure by the large crystals upon removal of the solvent [46]. The mechanical properties of solvent-treated Nomex® yarns resulted in a decrease in initial modulus and tenacity at break, with little change in elongation at break. This is attributed to the high degree of disorientation and freedom of movement in polymer chains.

SOLVENT BONDING IN NONWOVENS

To produce a well bonded nonwoven fabric without sacrificing softness and strength, a solvent-pretreatment can be suggested based on the idea of solvent bonding. By using a solvent for the binder fibers, a strong bond can be formed without imparting stiffness to thermally bonded nonwovens by calendering at a relatively low temperature. According to the INDA definition [6], in solvent bonding, solvents can be used to partially dissolve the surfaces of susceptible fibers, resulting in adhesive characteristics. Upon removal of the solvent, the fiber surface is resolidified, creating bonding at the crossover points. This solvent bonding technique is one of the autogenic bonding methods in which the fibers in a web are partially dissolved in close contact using either heat , pressure or solvents and then resolidified to form polymer bonds [28].

There are numerous studies, mainly patents, about the utilization of solvents in nonwoven fabrications, and several selected procedures were reviewed. The initial idea about solvent bonding was introduced by R. Reed in 1942 [106, 107]. Solvents that can dissolve or soften thermoplastic binder fibers were used as a plasticizer. The softening agents for cellulose acetate (CA) fibers were found to have latent adhesive or coalescent characteristics. There are many solvents for cellulose acetate fibers such as acetone, methyl alcohol. methyl cellosolve, propylene oxide, methyl acetate, ethyl acetate, acetic acid. diacetone, chlorobenzene, chloroform, etc. In Reed's studies, carded webs of cotton and cellulose acetate fibers were bonded by heat or by the softening agents for the CA fibers. The softening agents were acetone or solvent mixtures, which made it possible for CA to adhere or to bond with cotton fibers. For binder fibers, various cellulose esters, cellulose ethers, vinyl polymers and polyamides were suggested, while for base fibers, cotton, wool, silk and rayon were suitable. The claims were that the use of plasticizers increased fiber-to-fiber bond. One example is the use of a plasticizer, Santicizer, for cellulose acetate fibers. Santicizer is chemically identified as ethyl-phthalyl-ethyl-glycolate [106]. The cellulose acetate fibers containing 30 % of the plasticizer could be softened sufficiently to bond with other fibers by calendering at a temperature of from 177 to 191°C. It is also claimed that the use of plasticizers minimized the heat required for hot calendering.

There are several studies of the use of solvents in nonwoven bonding processes [28]. One example is the solvent treatment of cotton fibers for an autogeneous bonding. Cellulose fibers were found to become gelatinous after treating with concentrated H_2SO_4, or other cellulose solvents followed by quenching and washing. Other examples are webs of viscose rayon fibers treated with 8-10 wt % NaOH. The addition of aqueous solutions of certain salts such as LiCl, and KCNS to nylon, acrylic or polyester fiber webs has also shown promise.

The usefulness of latent solvents for the binder fibers in nonwoven manufacturing has also been demonstrated [88, 89]. First, fibers have been coated with a latent solvent, normally inert to the fiber and does not dissolve fiber or fiber surface at room temperature. Then, the solvent coating can be activated by heat and then attacks the fibers. As a result, a spot-welding effect at fiber intersections can be produced. As an application, the latent solvent, sulpholane (cyclic-tetramethylene-sulphone) was applied to opened and blended staple fibers, e.g. acrylic or cellulose acetate fibers, before carding. The amount of the solvent add-on was in the range of 10 to 15% by weight. In the curing stage, the solvent on the fibers was activated by heat, and cellulose acetate fibers were bonded at temperatures between 90 and 140°C, below the softening points of CA fibers. The article claimed that solvent bonding provided better handle than did adhesive bonding. At low levels of bonding the handle was as soft as that of unbonded fibers. Also, the solvent bonded fabrics showed excellent strength and resilience.

The use of HCl gas on polyamide fibers has been intensively studied [109, 111, 113, 114]. Webs of polyamide fibers, mainly Nylon, were treated with an activating gas, preferably hydrogen chloride. Bonding occurred autogeneously at filament crossover points with or without an additional calendering process. The postpressing process, when the gas treatment occurred before calendering, imparted greater strength to the polyamide nonwovens [111]. For good drape and hand properties, a pattern bonding of polyamide fibers followed by HCl activating gas treatment was suggested [114]. In this system, the concentration and the temperature of the gas must be carefully controlled and monitored. A stable process was suggested, wherein the concentration of the acid gas is maintained in the range of 20 to 30wt % and the temperature range is 21 to 52°C [113].

The use of another strong acid compound, sulfuric acid, for polyamide fibers has been studied [110]. The polyamide fibers were softened and swollen by the concentrated acid, and bonded to each other when subjected to pressure. One procedure was the use of fiber blends such as nylon with polyester and polypropylene fibers. The web was dipped in 40% by weight of 96% sulfuric acid solution, and then excessive solution was squeezed out. The softened web was coagulated by being immersed into a water bath. Before drying, the bonded webs were neutralized in 10% by weight ammonium hydroxide solution.

Another study, related to the use of latent solvents, suggested the possibility of using several different aqueous solvent solutions for the fabrication of nonwovens from acetate, polyester, modacrylic, polyamide and polyvinyl alcohol fibers [108]. The synthetic fibers were bonded together by solvents upon removal of the other component of solution, the evaporation of water. Several chemical groups of solvents were explored to determine bond efficiency with the fibers. These solvents included hydroxyaliphatic acids, aliphatic and aromatic polycarboxylic acids, polyhydroxy phenols, and monohydric monocyclic phenols. The webs were impregnated with latent solvent solutions with 1-20% concentrations and dried at elevated temperatures in the range of 250 to 350°F. The use of latent solvents in this procedure produced very strong fiber-to-fiber bonds for the fabrication of durable nonwovens.

Another study showed a process for solvent bonding blended nonwoven fabrics [115]. A controlled amount of solvent, e.g., 75% ethylene carbonate/25% propylene carbonate, was applied to webs of acrylic, modacrylic and polyester fibers, and then by the application of heat and pressure to give bonding at crossover points. The bonding resulted from the plasiticizing effects of solvents on the filaments which were attacked by the solvent coating upon activation by heat. The uniqueness of this method lies in the application method of solvents. Solvents in an aqueous solution in the concentration range of 10 to 20wt % were spayed onto the fabric surface. Improved elongation and teat strength were claimed by this aqueous solvent mist method.

Another use of solvents having solubility parameters close to those of fibers such as polyester and nylon fibers was examined [116]. This bonding techniques involved the use of both a polar solvent such as benzyl alcohol, nitro benzene, m-cresol and trichloroacetic acid, and microwave energy. The fibers which did not react to microwave energy, at first, were coated with polar solvents having sufficient polarity to be heated by microwave radiation. The bonding occurred when the webs were heated by microwave radiation.

CELLULOSE ACETATE AS A BINDER FIBER

Fiber Structure

Acetate fiber is the generic name for a fiber that is a partially acetylated cellulose such as wood pulp and cotton linters. The fiber is also known as (secondary) cellulose acetate. In the manufacturing process of cellulose acetate (CA) fiber, an acetic-acid ester derivative of cellulose is formed by treating swollen cellulose with acetic acid (HOAc) and sulfuric acid (H_2SO_4). By the addition of acetic anhydride, a triacetate derivative is formed with the acetylation of 3 hydroxyl groups per glucose unit of cellulose. The CA is obtained by hydrolysis of the triacetate to an average degree of substitution of 2.4 acetyl groups per anhydroglucose unit (a combined acetic acid value of 55%). In general, the lower the degree of substitution the greater is the solubility in polar solvents. The CA fiber is produced by a dry spinning method, in which an acetate-acetone dope, consisting of a 30% concentrated solution of acetate polymer in acetone, is extruded into to a hot-air drying column. The degree of polymerization (DP) of cellulose acetate is around 300, which is considerably lower than that of the starting cellulosic materials [2, 22, 65, 103].

The CA fiber has low crystallinity and orientation. The specific gravity is about 1.32. The crystalline portion of the CA is smaller than that of regenerated cellulose such as rayon [75, 83]. The reasons for the reduction in crystallinity are the bulky acetyl groups, the irregular chain structure with acetyl and hydroxyl groups, and the weak intermolecular forces. All these prevent the formation of the regular crystalline order [75].

Fiber Property

The uniqueness of cellulose acetate fibers lies in the cellulosic, and the thermoplastic and heat-fusible characteristics. Cellulose acetate is hydrophilic and absorbent. It has excellent wicking and comfort characteristics. Furthermore, CA can be thermally bonded, ultrasonically welded and thermally embossed due to its thermoplastic properties [95]. The melting temperature of cellulose acetate fiber is around 260°C (500°F), which is considered high compared with commercially available binder fibers, such as low melting polyesters and polypropylene. However, its advantage for thermal bonding as a binder fiber is its low softening or sticking temperature of 180-205°C [1, 103].

The tenacity of cellulose acetate fiber are in the ranges of 1.2 to 1.4 g/d (0.13 N/tex) [1, 103]. The elongation-at-break is in the range of 25 to 45 %. Although the CA fibers are weaker than viscose rayon fibers, they are more extensible and resilient [22, 75]. Therefore, the fiber is soft and supple with low modulus and high extensibility. This can produce less stiff nonwovens compared with many other binder fibers.

Unlike other binder fibers such as polypropylene and polyester, cellulose acetate fibers provide good absorbency and comfort. Moisture regain and content values are 6.3 and 5.0 %, respectively. The degree of swelling of CA fiber by absorption of water are 1% in length and 10% in diameter. Easy wettability, good liquid transport and high moisture uptake are enhanced by irregular cross-sectional shape. This makes cellulose acetate nonwovens useful in filtration applications. Another consumer appeal lies in the excellent coloring due to the strong affinity toward dyestuff and pigments [1, 2, 65, 95].

Cellulose acetate fibers are hypoallergenic and can be used in skin-contact and food-contact applications. The fibers are easy to sterilize by common treatments with ethylene oxide or gamma radiation, and they have the ability to neutralize ammonia by forming ammonia acetate with the acetyl groups. Therefore, cellulose acetate nonwovens can safely be used in diapers, sanitary products and medical applications [1, 95].

Solubility of Cellulose Acetate

Cellulose acetate fiber, containing acetyl and hydroxyl groups, can react with certain organic chemicals and can be made soluble in many common solvents such as acetone, low boiling ketones, phenols, selected aldehydes, methylene chloride, ethylene glycol ethers, dioxane, diethanolamine, pyridine and aniline [1, 16, 65, 95]. It can be chemically modified and plasticized with liquid plasticizers such as dimethyl diethyl and dibutylphthalates [2]. Like many cellulose derivatives, CA can be dissolved in its solvents to form anisotropic solutions like liquid crystalline. The polymer-solvent interaction determines the concentration needed to form the anisotropic polymer solution. CA is soluble in 60% or higher concentrations of acetone-water at 25 °C [65].

The solubility parameters of CA and acetone, a common solvent for CA, are represented by the degree of hydrogen bonding and Hansen's solubility parameter. Typical values are presented in Table 1 [16]. For complete miscibility, hydrogen-bonding capacities and solubility parameters of polymer and solvent should be similar. Acetone, one of ketones, is classified as a moderate hydrogen-bonding solvent. A complete solution of CA and acetone would be expected from the similar solubility parameters (δ_m =9.9-14.7 and 9.9, δ(single value) =10.9 and 9.9, and δ =19.56 and 20.10, CA and acetone, respectively) of the two components.

Table 1. Solubility Parameters of Cellulose Acetate and Acetone [16].

Solvent Hydrogen Bonding in $(cal/cm^3)^{1/2}$				
	poor (δ_p)	moderate (δ_m)	strong (δ_s)	single value
CA	11.2-12.7	9.9-14.7	-	10.9
Acetone	-	9.9	-	9.9

Hansen's Solubility Parameter in $(MPa)^{1/2}$				
	δ_D	δ_P	δ_H	δ
CA	15.55	-	11.87	19.56
Acetone	15.5	10.4	7.0	20.10

* $\delta^2 = (\delta_D)^2 + (\delta_P)^2 + (\delta_H)^2$

CHAPTER 3

METHODS

FIBER SELECTION AND PROPERTIES

The cotton selected for this study was supplied by Cotton Incorporated. This scoured and bleached commodity cotton has a reported moisture content of 5.2%, a micronaire value of 5.4, and upper-half-mean fiber length of 24.4 mm (0.96 inch). It is the same cotton on which the previous cotton/thermal binder fiber blended nonwovens studies [33, 34] have been based. The scoured fiber, whereby the natural wax surface coating was removed, has long been known to provide an improved bonding surface to binder fibers of many types.

The cellulose acetate staple fibers which have comparable denier and fiber lengths to those of cotton were supplied by Celanese Corporation in Charlotte, NC. It is a modified cellulosic fiber having easy wettability, good liquid transport, and high moisture uptake [1]. A major advantage is that acetate is made from renewable sources - like wood pulp and cotton linters. Because it is a thermoplastic fiber with a softening temperature of 180°C, it can be used as a binder fiber which can be used to produce cotton/cellulose acetate nonwovens by thermal calendering, thereby eliminating the use of any petroleum-based synthetic fiber or chemical binder.

In addition, the chemical modification of cellulose acetate by using plasticizing agents to lower the melting temperature allows lower temperatures of processing [105, 106].

BIODEGRADATION TESTS

Two standard test methods, *AATCC 30-1988: Antifungal Activity, Assessment on Textile Materials: Mildew and Rot Resistance of Textile Materials* [7] and *ASTM D5209-91: Standard Test Method for Determining the Aerobic Biodegradation of Plastic Materials in the Presence of Municipal Sewage Sludge* [10], were selected and adopted, to determine the biodegradability of cellulose acetate fibers.

AATCC SOIL BURIAL TEST

The main experimental procedures, based on the AATCC method, consisted of the following steps.

1. A soil bed was prepared by mixing garden and potting soils in the ratio of approximately 1:1. A moisture content in the range of 20-30% of the soil mixture was controlled by adding distilled water.
2. Five replicated samples each of 100% cotton, 50/50 cotton/CA blend, and 100% CA fabric were of a size 1" x 7", and placed into the soil bed.

3. An incubation temperature in the range of 25-30°C was controlled by a garden lamp.

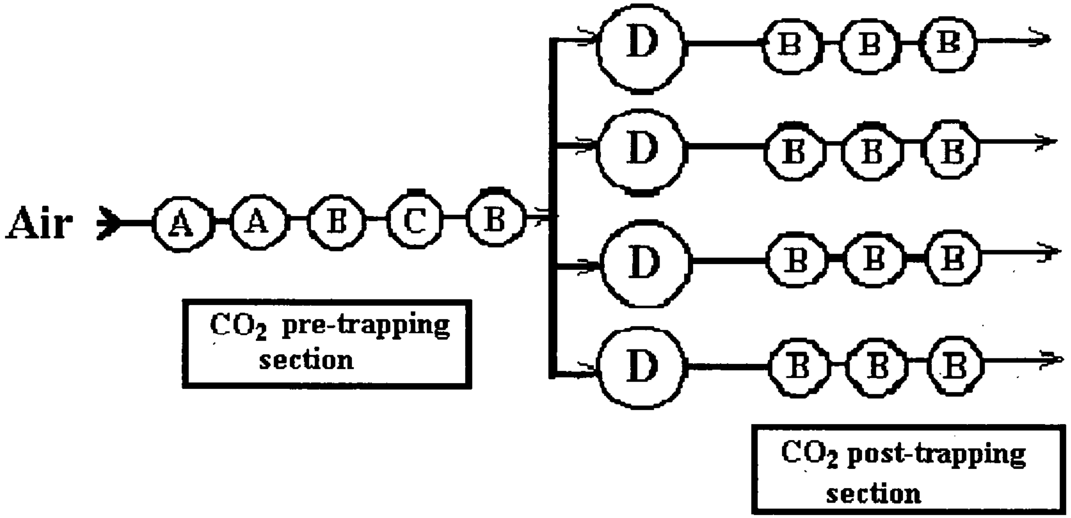
4. Moisture content was readjusted by the spraying of water each week, and fabric samples were examined visually from the soil bed. The cotton samples were only visually examined in this soil bed test, but the extent of biodegradability of the CA fabric was monitored instrumentally by ASTM strength tests.

ASTM AEROBIC SLUDGE TEST

Setting-up apparatus

The experimental scheme is illustrated in Figure 1. It physically consisted of connecting a series of Erlenmeyer flasks to one another by flexible tubing in such a way as to provide carbon dioxide scrubbing, bioreactor and carbon dioxide trapping stages. A controlled flow rate of air was continuously provided though the series of flasks which were sealed with rubber stoppers.

The carbon dioxide scrubbing components consisted of three flasks in series: the first flask contained 700 mL of 10 N sodium hydroxide solution and the second flask contained 700 mL of 0.025 N barium hydroxide solution. The third remained empty and was included to prevent accidental overflow into the bioreactors that followed. The bioreactors were connected in parallel, and each bioreactor contained a 1% inoculum prepared from sludge and medium stock solution. One of those included cellulose acetate fibers and another contained cotton fiber as a known biodegradable



A = NaOH
B = Ba(OH)$_2$
C = Empty
D = Bioreactors for test fibers and blank

Fig. 1. Experimental Scheme for ASTM Aerobic Sludge Test.

control against which the cellulose acetate was to be compared. The flask without a fiber sample acted as a check basis of carbon dioxide generation by the sludge alone. All bioreactors were placed on magnetic stirrers to provide proper oxygen and mixing. They were followed by carbon dioxide trapping units comprising a series of 100 mL flasks, each containing 100 mL of 0.025 N barium hydroxide.

Preparing Inoculum

Sludge containing activated microorganisms was obtained from Kawahee Wastewater Plant in Knoxville, TN. After aeration in the laboratory for four hours, the 500 mL sludge sample was homogenized for 2 minutes in a blender. Settling for 30 minutes followed to avoid suspended particles in the sludge. Enough supernatant, 15 mL for each bioreactor, was taken out to be used for preparing the 1 % inoculum for each 2-L bioreactor. The 1% inoculum were prepared from medium stock solution, sludge inoculum and high quality water. The actual mixture is as follows:

1. 1235 mL of high quality water
2. 13.5 mL medium stock solute consisting of 1.5 mL of 0.001% magnesium sulfate, 1.5 mL of 0.001% calcium chloride, 1.5 mL of 0.001% ammonium sulfate, 3 mL of 0.002% phosphate buffer made of potassium phosphate dibasic, potassium phosphate monobasic, sodium phosphate dibasic and ammonium chloride, and 6 mL of 0.004% ferric chloride
3. 15 mL activated sludge inoculum

The total 1263.5 mL solution added to each bioreactor as described above. The whole system was aerated for 24 hours, prior to filling the CO_2 trapping flasks with $Ba(OH)_2$ solution.

Testing and Analyzing CO_2 Evolution

Samples with enough carbon content, not exceeding a 40/1 weight ratio of C/N based on stock solution, were prepared. Chopped samples of approximately 5 mm in length of 500 mg of cellulose acetate and 500 mg of cotton fibers were added to each of two bioreactors. The cotton fibers, which are known to be biodegradable, were used as a positive standard. One bioreactor remained blank to be monitored. A small amount (15mL) of solution was removed from each bioreactor to measure initial pH and Total Organic Carbon (TOC) content.

The test was initiated by setting the air flow rate at 50 to 100 mL/min (approximately 1 to 2 bubbles/sec in the bioreactors). Once the $Ba(OH)_2$ solution in the trapping flasks began absorbing evolved $CO_{2,}$ the white precipitation of barium carbonate could be observed. Every few days, the CO_2 absorbers (trapping flasks) nearest each bioreactor were removed to determine carbon dioxide content by titration with HCl solution. One hundred milliliters of 0.025 N $Ba(OH)_2$ solution was titrated with 0.06 N HCl solution to a phenolphthalein end-point by color change from clear to pink or red. The amount of evolved from the test specimens was adjusted downward by the resulting CO_2 from the blank test.

The theoretical (Y) and actual (Y') amount of CO_2 evolved were calculated by following steps:

C (carbon content of the test material, X mg) + $\mathbf{O_2}$ = $\mathbf{CO_2}$

$\mathbf{Ba(OH)_2}$ + $\mathbf{CO_2}$ = $\mathbf{BaCO_3}\downarrow$ + H_2O

$\mathbf{Ba(OH)_2}$ + **2HCl** = $BaCl_2$ + $2H_2O$

Y mg of CO_2 evolved (theoretical) = **X mg of carbon x 44/12**

Y' mg of CO_2 evolved (actual) = **44/2 x 0.06 N x Z mL of HCl**

= 1.32 x Z ml of HCl

The percentage of carbon dioxide evolved (Y ÷Y' x 100) was obtained by the equation,

$$\textbf{\% CO}_\mathbf{2}\textbf{ evolved} = \frac{1.32 \times Z}{{}^{44}\!/_{12} \times X} \times 100$$

where Z = the amount(mL) of HCl solution used for titration

X = the carbon content(mg) of 500 mg of fibers,

222 mg and 246 mg for cotton and cellulose acetate fibers, respectively.

The testing was continued until the CO_2 evolution reached a plateau. Throughout the experiment, the temperature remained in the range of 25 ± 5 °C. Insoluble or solid matter and biomass that remained in the bioreactors were filtered by ASTM 40-60 type crucible holders and 0.2 μm CA membrane filters. A small amount (10mL) of solution was removed to measure final pH and TOC content. The TOC contents were obtained by a Dohrmann Carbon Analyzer, in which the concentration of oxidizable carbon matters, such as insoluble or soluble organic carbons, were measured.

PROCESSING STEPS

OPENING, BLENDING AND CARDING

Each of the fiber components were prepared by separate mechanical opening. The blending of two fibers with selected blend ratios (50/50 and 75/25, 85/15 cotton/cellulose acetate) was done by hand mixing the two fibers. For each of the blends, a total of 50 grams of blended fibers were weighed for carding to form a web using a modified Hollingsworth card. The resulting carded web gave a basis weight of 160 g/m^2.

THERMAL CALENDERING

The carded webs were thermally bonded using a Ramisch Kleinewefers 600-mm (23.6-inch) wide, five-roll calender. The effect of varying the processing variables of temperature, feed speed, and nip roll pressure on the effectiveness of bonding was recognized as important and a choice of operational parameters was selected to give reasonably optimum conditions. The operational parameters were selected based on results found in the previous literature [32, 33, 34, 35]. The roll speed was 10 m/min, and the nip roll pressure was 100 kN. The temperatures of the patterned roll were selected to be slightly above the softening temperature of the cellulose acetate fiber, and the temperature of the smooth roll was selected to be close to the softening temperature of the fiber.

SOLVENT PRETREATMENT

The lowering of CA softening temperatures and the enhancement of heat transfer in the calendering process were considered in optimizing both process and product properties. The possibility of using acetone solvent was investigated both by spaying acetone onto the carded webs before or after calendering, and by placing the carded webs in a closed container of saturated acetone vapor prior to calendering. An atomized spray gun or a mist spray gun was to be used for applying acetone solvent. For solvent vapor treatment, an acetone concentrate was to be poured into large enough containers in which the webs could be placed on a perforated rack above the liquid reservoir of acetone. There is no liquid/fabric contact. After a specified time for saturation, the webs were to be removed and immediately calendered. In the solvent assisted calendering procedure , the calendering temperatures were set at lower temperatures than the normal softening temperatures of CA fibers. The solvent assisted calendering procedure, thermal calendering process and solvent pretreatment experimental setup are presented in Figures 2, 3 and 4.

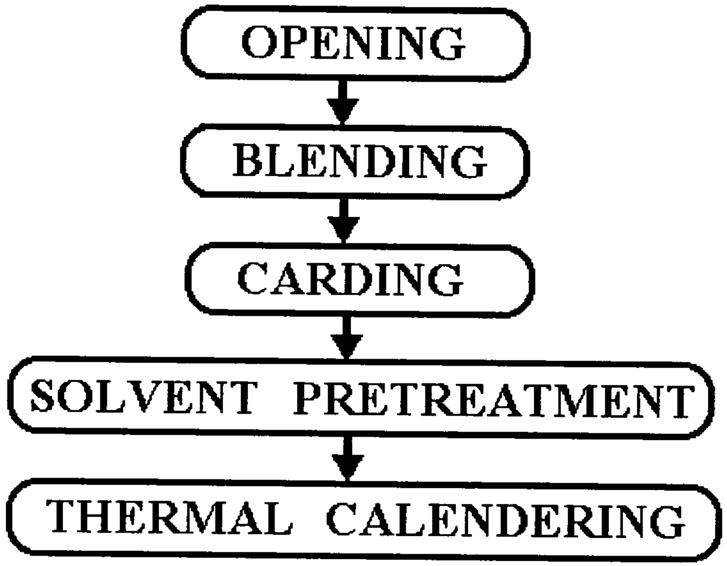


Fig. 2. Experimental Scheme for Solvent-Assisted Thermal Calendering.

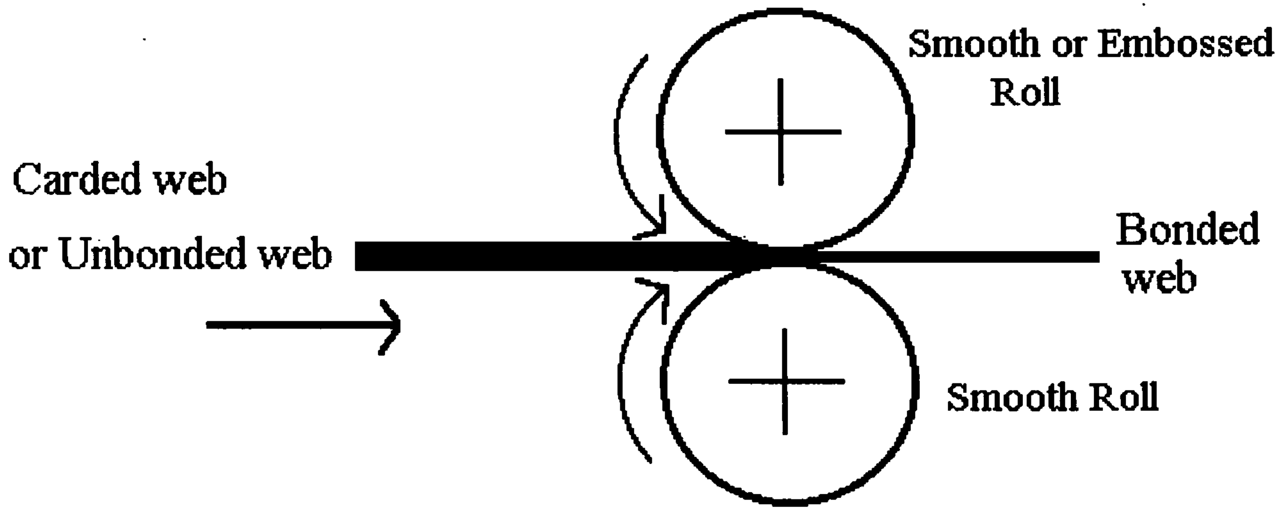


Fig. 3. Thermal Calendering Process

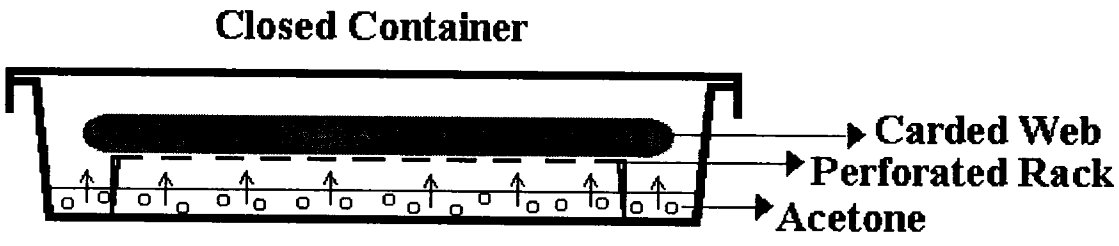


Fig. 4. Experimental Setup for Solvent Pretreatment.

CHARACTERIZATION OF NONWOVEN FABRICS

The nonwoven fabrics produced from different conditions were evaluated. Initially, the tensile properties were evaluated according to ASTM or INDA Standards. Other performance properties such as bending rigidity and air permeability were examined, and also, to understand the bonding behavior of cellulose acetate, Scanning Electron Microscopy (SEM) pictures were taken.

Because of the hydrophilic nature of cotton and cellulose acetate fibers, the samples were conditioned and tested under a standard atmospheric conditions to avoid the temperature and relative humidity effects on the physical properties of the fabrics. Based on *ASTM D 1776-90 Standard Practice for Conditioning Textiles for Testing*, the temperature of 70 ± 2 °F (21 ± 1 °C) and the relative humidity of 65 ± 2 percent were used as the standard atmosphere for testing textiles.

BASIS WEIGHT

The conditioned nonwoven samples were cut and weighed as directed by *INDA Standard Test (IST) 130.1-92 Standard Test Method for the Mass Per Unit Area of Nonwoven Fabrics*. Two of 7" x 7" samples were cut from each nonwoven fabric, and weighed. The average values of weight measurements were calculated and converted to the mass per unit area in g/m^2.

THICKNESS

As directed in *ASTM D 1777-64 Standard Test Method for Measuring Thickness of Textile Materials*, ten thicknesses were obtained for each nonwoven type and an average value calculated. A TMI 49-70-00 Micrometer, which measured the thickness with automatic preset loading was used.

AIR PERMEABILITY

Air permeability, according to ASTM definition, is defined as the rate of air flow through a material under a differential pressure between the two fabric surfaces. Air permeability, an important performance property, is directly related to filtration efficiency and is an indication of breathability of fabrics. A Frazier Differential Pressure Air Permeability Machine was used, as directed in *ASTM D 737-75 Standard Test Method for Air Permeability of Textile Fabrics*. The rate of air flow passing through the test area of the fabric was determined from the level of oil in a vertical nanometer and the use of conversion tables for selected nozzles. An average value ($ft^3/ft^2 \cdot min$) of five observations was obtained for each fabric, and converted to metric units ($cm^3/cm^2 \cdot s$) by a conversion factor of 0.508.

STIFFNESS

Stiffness or flexural rigidity, according to *ASTM D 1388-64 Standard Test Methods for Stiffness of Fabrics*, is defined as the resistance to bending of a fabric. The single cantilever test method was selected and performed by a TMI FRL Cantilever Bending Tester, in which the length of overhang (O) was measured when the tip of a fabric strip fell to an angle of 41.5 degree below horizontal. Four 1" x 7" fabric strips were tested each in machine and cross directions, and average values in cm, were obtained as overhanging lengths. In this test method, bending length (c), a measure of the interaction between fabric weight and fabric stiffness, was calculated by dividing the length of overhang by two. Then, the flexural rigidity (G) in mg·cm was calculated by the equation,

$$G = W \times (O/2)^3 = W \times c^3$$

where W = weight per unit area, mg/cm^2.

An over-all flexural rigidity (G_o), the geometric mean of the flexural rigidity values(G_m and G_c) of the machine and cross directions was calculated by the equation,

$$G_o = (G_m G_c)^{1/2}.$$

TENSILE PROPERTIES

The tensile test procedure was followed by *ASTM D 1117-80 Standard Test Method for Tensile Testing of Nonwoven Fabrics*. Five 1" x 7" strips in each MD and CD were tested using a United tensile tester with a constant-rate-of-elongation, 5"/min. The peak strength, specific stress and elongation, yield point and initial modulus were reported.

The breaking strength or load (Kg) is defined as the maximum load to break a fabric. This can be converted to newtons by multiplying by 9.8. The specific strength or tenacity (N·m/Kg) is obtained from the load divided by mass/unit length, or from stress (N/m^2) divided by density. Without a known density value of the web, the specific strength is calculated from the load, fabric width and basis weight values as follows.

$$\text{Specific stress} = \text{load(N)} / \{\text{width(m)} \times \text{basis weight}(g/m^2)\}$$

The conversion factors for denier units are,

$$1\ \text{N·m/g} \equiv 1\ \text{mN/tex} \equiv 10^3\ \text{N·m/Kg}.$$

The initial modulus is the slope of the stress-strain curve, and the reciprocal of the initial modulus is referred to as compliance. The importance of this lies in the fact that lower the modulus, the easier is the extension of the fabrics. The yield point is an additional important characteristic of the fabric. The value indicates the degree of resistance to load and influences the handle of fabric [75].

STATISTICAL METHODS

FACTORIAL DESIGN

The experiments were designed for a combination of one set of a full factorial design and the other set of a 3-way fractional factorial design. The full set of experiments was a 3-way factorial design (3x3x8) with 3 classes(factors): bonding temperatures, blend ratios and solvent pretreatment times. There were three levels for each temperature and blend factors. The levels for temperature were 150, 170 and 190 centigrade, and the levels for cotton blend with CA were 50, 75 and 85 percentage. The solvent pretreatment time factor consisted of 8 levels (0, 30, 45, 69, 75, 90, 105 and 120 minutes). Table 2 summarizes the full design.

To reduce the total number of experiments to be carried out, a fractional factorial design was employed. A one third of the full factorial design is presented in Table 3. The combinations of processing parameters were selected to equalize the observations across all levels of the full factorial design. The confirmed two-way frequency distribution is presented in Appendix A-1. The 24 selected observations from the factorial design were chosen for error term instead of adding a whole replicated set (72). The total number of observations in data set was 96, i.e., 72+24.

Table 2. Class Level Information for 3-Way Factorial Design.

Class	Levels	Values
Temperature(T)	3	150, 170, 190
Blend(B)	3	50, 75, 85
Solvent Treatment Time(S)	8	0, 30, 45, 60, 75, 90, 105, 120

Table 3. Processing Conditions and Blend Ratios for One-Third Factorial Design.

Observations	T	B	S
1	190	85/15	75
2	190	85/15	45
3	190	85/15	30
4	170	85/15	75
5	170	85/15	60
6	150	85/15	105
7	150	85/15	60
8	190	75/25	120
9	190	75/25	90
10	190	75/25	60
11	170	75/25	120
12	170	75/25	45
13	170	75/25	30
14	150	75/25	90
15	150	75/25	30
16	150	75/25	0
17	190	50/50	105
18	190	50/50	0
19	170	50/50	105
20	170	50/50	90
21	170	50/50	0
22	150	50/50	120
23	150	50/50	75
24	150	50/50	45

ANALYSIS OF VARIANCE (ANOVA)

Analysis of variance (ANOVA) was employed for all eighteen test properties for the effects of the bonding temperatures, the solvent pretreatment times and the blend ratios on each of the individual physical properties.

ANOVA is a versatile technique for studying the effects of independent variables on dependent variables and the relationship between the two types of variables without assumptions about the nature of the statistical relationship. Within this context, a regression analysis is often used to determine the quantitative analysis of the independent variable [79]. In a multi-factor study, two or more factors (independent variables) are used to compare population means. The values of these factors are called levels [69, 79]. In addition to the above main effects, there are two types of interaction effects. If there is no interaction among the factors, the factor effects are additive. If the treatment mean curves for the different factor levels in a graph are not parallel, the factor effects are interacting [79].

The ANOVA model for the 3-way factorial experiment, without 3-way interaction is,

$$Y_{ijkm} = \mu + \alpha_i + \beta_j + \gamma_k + \alpha\beta_{ij} + \alpha\gamma_{ik} + \beta\gamma_{jk} + \varepsilon_{ijkm}$$

where Y_{ijkm} = the response or the each physical property measured

μ = an overall mean, a constant

$\alpha_i, \beta_j, \gamma_k$ = the effects due to the bonding temperature i, the blend ratio j and the solvent pretreatment time k, all constants

$\alpha\beta_{ij}$, $\alpha\gamma_{ik}$, $\beta\gamma_{jk}$ = the 2-way interaction effects, all constants

and ε_{ijkm} = a random error associated the response.

The null hypothesis of the ANOVA implies that there is no difference among the treatment means. This can be written as,

H_0: all $\alpha_i = 0$, or all $\beta_j = 0$, or all $\alpha\beta_{ij} = 0$

H_a: all $\alpha_i \neq 0$, or all $\beta_j \neq 0$, or all $\alpha\beta_{ij} \neq 0$

The test statistic of ANOVA is a F-test where between-sample variation (between treatments) compared to within-sample variation (within treatments, error). This F-statistic can be written as:

$$F = \frac{MST}{MSE}$$

where MST = a mean square for treatments

and MSE = a mean square for error.

For a large F-value, the null hypothesis is rejected since the variation between treatments exceed the variation within treatments.

The General Linear Model Procedure (GLM) is used in most unbalanced experimental designs like a fractional factorial design [5]. From the preliminary analysis, the 3-way interactions in all physical properties were found to be always insignificant, the degrees of freedom and the sums of squares of the interactions were combined as those of error terms. The degrees of freedom for the ANOVA are summarized in Table 4.

Table 4. The Degrees of Freedom for ANOVA.

	Degrees of Freedom
Source terms	43 (2+2+7+4+14+14)
Error	52 (28+24)
Total	95

LEAST-SQUARE-MEAN COMPARISON

The Least-Square-Mean (LSM) comparison technique was employed to discriminate between the effects of the independent variable levels on each physical property. Among the multi-mean comparison techniques, the LSM comparison was selected for this study due to the fractional experimental design being unbalanced, i.e., with missed data points.

The LSMs are estimators of the class (factor) or subclass (level) marginal means that would be expected from the balanced design involving the class variable with all covariates at their mean value [5]. From the GLM procedure in SAS, the LSMEANS statement computed the LSMs and the standard error for the LSMs, and listed the probabilities form the t-tests of two LSMs.

The null hypothesis and the t-test statistic, to compare two LSMs of the factor levels,

H_0: LSM (i) = LSM (j)

$$t = \frac{LSM(1) - LSM(2)}{S_p\sqrt{1/n_1 + 1/n_2}}$$

where n_1 and n_2 = the degrees of freedom for the two levels

$$S_p = \sqrt{\frac{(n_1 - 1)S_1^2 + (n_2 - 1)S_2^2}{n_1 + n_2 - 2}}$$

The probability above $|t|$ values of any two LSMs in each factor were provided from the SAS output, and the null hypothesis would be rejected for a large t-statistic indicates a significant difference in the mean values.

RESPONSE SURFACE METHODOLOGY

Response Surface Methodology (RSM) is a technique for approximating complex response surfaces by using polynomial response functions. The main purposes of RSM are to provide the response pattern in an experimental region and to locate the region of optimal response, i.e., maximum or minimum points [79]. Generally, RSM is carried out in three steps: the design of experiments for the response surface study, the determination of a mathematical model with fitted data, and the determination of optimal response conditions [56, 79].

The fractional factorial experiments including a set of complete data and a set of one-third fractional data was used in this response surface study. For the eighteen physical properties, the experimental design had multiresponse functions. In addition, the response functions were designed to be simultaneously optimized for the four selected properties, MD strength, MD elongation, overall flexural rigidity and air permeability.

For each physical property, a polynomial regression analysis was employed to investigate the cause-and-effect relationships between the independent variables and the response or the dependent variable, as well as to establish a fitted mathematical model for the response for further analysis of the response system. From a canonical analysis, the response surface of two independent variables were generated and the optimal region and the shape of the response surface were predicted.

Model Fitting - Regression Analysis

Polynomial (curvilinear) regression models are extensions of general linear regression models requiring higher degrees of polynomial response functions [79]. In this study, the regression models selected were 2nd-order polynomial equations with linear, quadratic and interaction (crossproduct) effects. The response function for the regression model is,

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{33} x_3^2 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3$$

where β_0 = the mean response of Y at x = 0

β_1, β_2, β_3 = linear effect coefficients

β_{11}, β_{22}, β_{33} = quadratic effect coefficients

β_{12}, β_{13}, β_{23} = interaction effects of coefficients

and x_1, x_2, x_3 = the independent variables.

The significance of the regression model relies on two criteria: R-squares and F-test results. The R-square is the coefficient of multiple determination, which is the portion of the total variation in y associated with and explained by the set of x variables [69, 79]. The lack of fit test from the F-statistic is the procedure for determining whether the response model is appropriate and significant. The null hypothesis and the test statistic of the F-test are [82],

H_0: the regression model is appropriate.

$$F = \frac{MS_{LACK}}{MSP_{EXP}}$$

where MS_{LACK} = mean squares due to lack of fit

MSP_{EXP} = mean squares due to pure experimental error (from ANOVA)

and $SSE = SS_{LACK} + SSP_{EXP}$ (a sum of squares due to regression error).

For a model with a poor fit, the F-ratio increases, as a result of the large value of the mean square due to lack of fit. Therefore, the null hypothesis would be rejected for large F value, and otherwise the model would be significant from the small value of F-ratio, the large *P*-value. Another method to prove the appropriateness of the model is to check the normality of the residual plots against the response y, which provide the normality of the random errors [56, 79, 82].

From the appropriate regression models of the physical properties, relationships between independent and dependent variables were quantitatively characterized, and further analysis by RSM would be possible to define the shape of each response function and to find out the optimal possessing conditions for each property.

Canonical Analysis

The second and main task of RSM is to determine the optimal condition from the characterized and estimated response surface. From the fitted 2nd-order polynomial models, contour plots of estimated responses can be generated. Based on the shape of the curves, there are several kinds of stationary points at which the slope of the response surfaces is zero [56]. A maximum (minimum) point is a point where response values are decreasing (increasing). If the maximum point is in the experimental region, there is no need to changing processing conditions. Another type of stationary points is a saddle point or minimax of the surface. In this case, the optimal response could be obtained by additional processing selections. Other types are a rising (falling) ridge surface and a stationary ridge, which may require additional experiments to define the optimal conditions. The optimal conditions in the experimental region can be simply extrapolated along the line of principle axis of surface [56, 79].

From SAS, the RSREG procedures provide a determination of the conditions of optimal response as well as an estimate and fit for the complete quadratic response surfaces. A canonical analysis was used to investigate the shape of the predicted response surface and to determine the type of the estimated stationary points [5].

From the canonical analysis, eigenvalues and eigenvectors in the matrix of the quadratic parameters are obtained. These characterize the shape of the response surface. The signs of the eigenvector indicate the direction of the principle axis of orientation, and the signs and magnitudes of the eigenvalues are related to the slope of the response surface. A contour plot of the predicted values by two factors is created by plotting of the surface. In addition, the critical values of the factor levels at the stationary points are provided as optimal conditions in the experiments. The main purpose of this analysis is to obtain a unique optimum combination of factor values in the experimental region. Otherwise, further analysis such as ridge analysis should be performed to search for the region of optimum response.

MULTIOPTIMIZATIONS BY BLEND RATIO

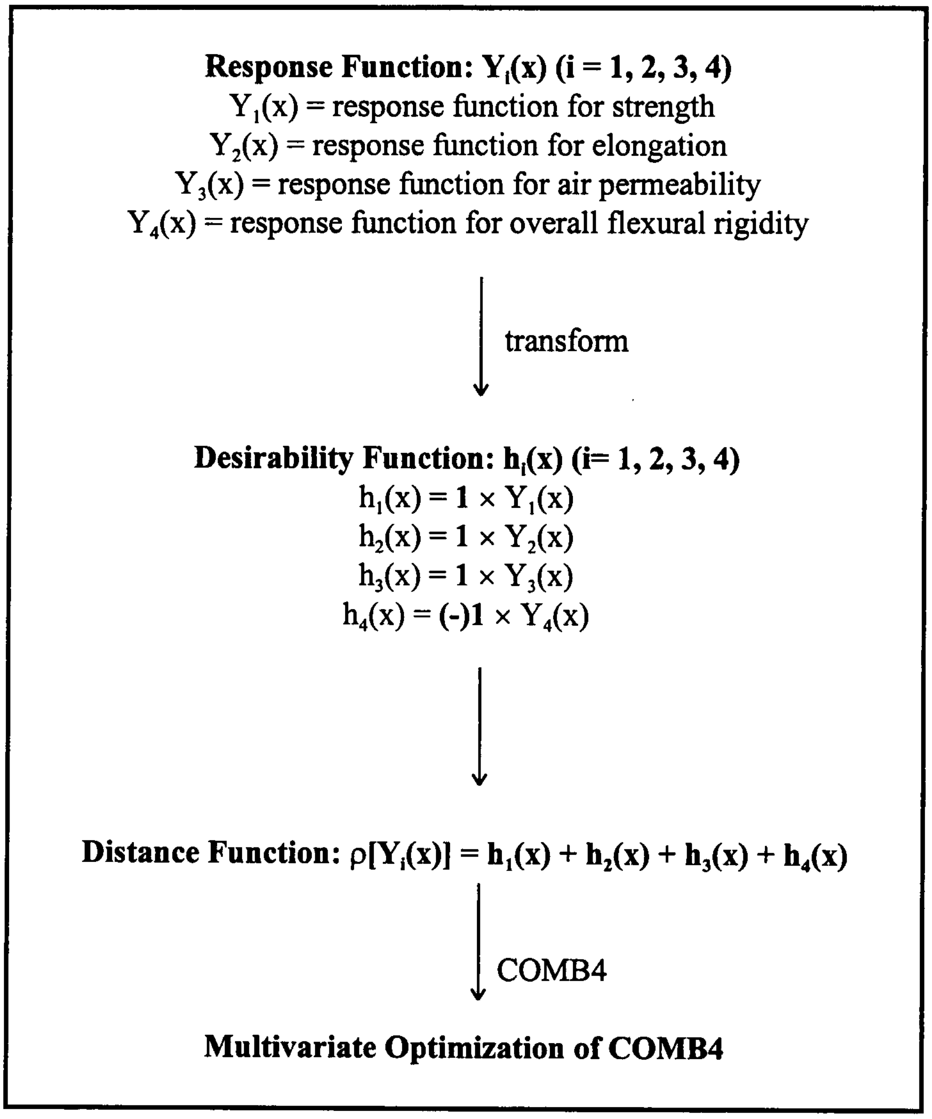
Since the blend ratio of the two fibers is a given value rather than a processing condition of the nonwoven fabrication, the RSM by blend ratio might be a more practical choice for suggesting the optimal processing values for each property. In addition, the selected properties considered as the most important ones for the end-use of the nonwovens can be simultaneously optimized for the best processing conditions for fabrication.

A multiresponse and multivariate experiment like this study is complicated, yet often leads to the need for a combined optimization of several response variables. A simple solution would be obtained by superimposing the contour plots of all response variables considered and then by locating a region of optimal conditions for the responses. However, this technique does not identify the definite optimal processing conditions very well. In this study, the simultaneous optimization of four response variables was accomplished by using a *desirability function* and *generalized distance* approach [56].

The multi-optimization technique using the desirability function approach is based on the concept of utility or desirability of a given property related to its response function. Each predicted response function of a property, $\hat{Y}_i$ (i = 1, 2, $\cdots$ r ; r = the number of responses), is transformed into a user's own desirability function, h_i. The transformation is handled by selected settings of desirability, specified by maximum and minimum acceptable parameters for each response. Then, an overall desirability function is calculated from the geometric mean of the values of h_i, $[h_1 \times h_2 \cdots \times h_r]^{1/r}$. The overall desirability function is used as an input variable, and then maximized over the experimental region by a univariate optimization procedure. This approach is very simple, the variances of the responses and the correlations among the responses are not considered [56].

The generalized distance approach for a multiresponse experiment is based on the assumption that all response functions depend on the same set of input variables (x_1, x_2, $\cdots$ x_k ; k = the number of factors) and that polynomial regression models of the same degree within the experimental region can be used. To find a near optimum for all responses, compromising conditions are placed on the input variables. Initially, a distance function $\rho[\hat{Y}(x), \phi]$ is selceted, based on the distance between the predicted equation of individual response ($\hat{Y}(x)$) and a point in the r-dimensional Euclidean space for the vector of individual optima, ϕ. The distance function can be obtained in a numerous ways, and the multiresponse optimization for the near optimum conditions on the input variables is performed by minimizing the distance function over the experimental region.

In this study, the desirability function was selected to determine the near optimal processing conditions to produce strong, extensible, breathable and flexible nonwovens. The actual data consisting four responses were used for the transformation instead of the predicted response functions. Then, a distance function to be optimized was chosen rather than calculating the overall desirability function using a geometric means. The desirability functions of data points for strength, elongation, air permeability and overall flexural rigidity were 1, 1, 1, (-)1, respectively. The distance function for the transformed data by the desirability function was the combination of the four transformed individual data points from the responses. The combined values were then optimized over the experimental region using univariate optimization techniques. The optimal bonding temperature and the optimal solvent treatment time for each blend ratio are obtained for the higher values for the properties but the lower value for the flexural rigidity value (higher softness). The summarized steps involved for the multi-response optimization are in Figure 5. The combined optimization technique was simple in this experiment because the same input variables and the desirability functions were employed without any weight for the particular responses.



Response Function: $Y_i(x)$ ($i = 1, 2, 3, 4$)

$Y_1(x)$ = response function for strength

$Y_2(x)$ = response function for elongation

$Y_3(x)$ = response function for air permeability

$Y_4(x)$ = response function for overall flexural rigidity

transform

Desirability Function: $h_i(x)$ ($i = 1, 2, 3, 4$)

$h_1(x) = 1 \times Y_1(x)$

$h_2(x) = 1 \times Y_2(x)$

$h_3(x) = 1 \times Y_3(x)$

$h_4(x) = (-)1 \times Y_4(x)$

Distance Function: $\rho[Y_i(x)] = h_1(x) + h_2(x) + h_3(x) + h_4(x)$

COMB4

Multivariate Optimization of COMB4

Figure 5. Summarized Steps for Multioptimization by Response Surface.

CORRELATION

Pearson correlation coefficients (Rho, ρ) were estimated from the data points of the predicted response surfaces to measure the strengths of liner relationship among the physical properties and especially between the properties in MD and in CD. The Pearson correlation coefficient is the measurement of a linear relationship between X and Y variables, and is defined by the following equation [69]:

$$\rho = \frac{Cov(X,Y)}{\sqrt{(VarX)(VarY)}} \quad .$$

The value of 1 or -1 indicates perfect positive or negative relationship, and the value of 0 signifies no linear relationship between X and Y variables.

The number of observations from the regression models for each property were 3171, and *t*-tests were conducted to verify linear relationships among the properties. The null hypothesis was followed,

H_0: Rho = 0.

In the case of the existence of linear relationship between two variables, the strength of the relationship was arbitrarily categorized according to strong ($0.7 < |\rho|$), moderate ($0.3 < |\rho| < 0.7$) and unrelated ($|\rho| < 0.3$).

CHAPTER 4

RESULTS AND DISCUSSION

BIODEGRADABILITY

AATCC SOIL BURIAL TEST

After two weeks, the 100% greige cotton fabric indicated degradation by showing holes in the fabric. There was total degradation or disappearance after 6 weeks in the soil bed. The 50/50 cotton/CA blend began to show degradation after four weeks. After 6 weeks only cellulose acetate fibers were intact. For the 100% cellulose acetate fabric, no visual degradation was observed for 12 weeks, but the white CA fabric was severely contaminated by the soil even after rinsing the fabric with distilled water. Since the cotton and the blend fabric could not be recovered after 12 weeks in the soil bed, only the CA fabric was evaluated for strength retention. The breaking load values for the untreated and the treated acetate fabrics were 18.68 kg and 13.64 kg, respectively. Therefore, evidence of microbial attack on the CA fabric was obtained on the basis of a 27% strength loss. Weight loss measurement was not possible due to the contamination of soil, which might have led to weight gain.

This result does not support the conclusion by Siu [93, 94] which claimed microbial resistance of CA fabrics with a DS above 1.0. One reason might be that the test in those studies was based strictly on weight loss of the CA fabric.

ASTM AEROBIC SLUDGE TEST

These tests included in three separate experiments. In the first experiment, 100% cotton and 100% CA fibers were examined separately to confirm the biodegradability of these fibers. In the second and third experiments, blends of fibers with different blend ratios were tested for possible synergistic action of the enzymes responsible for microbial degradation of cotton and CA fibers.

Biodegradation of Cotton and CA Fibers

Visual observations were made throughout the experiment for qualitative analysis. After two days, cotton fibers began to dissolve. After 10 days no fiber structure was observed, and there was significant carbon dioxide evolution. After 14 days, the solution in the standard bioreactor containing cotton fibers became clear of any solid matter. A growth of algae was observed after two months. These results confirmed the activity of microorganisms in the test procedures, and showed that they were comparable to results from the soil burial test when there was severe degradation of cotton fiber after two weeks.

As for biodegradability of CA fibers, there was breakdown and dissolving after 20 days. A growth of algae was observed after three months. Throughout the experiment, the blank bioreactor did not show any visual change in terms of its color, clarity or sign of algae growth. The cumulative percentage of carbon dioxide evolution over time is shown in Figure 6.

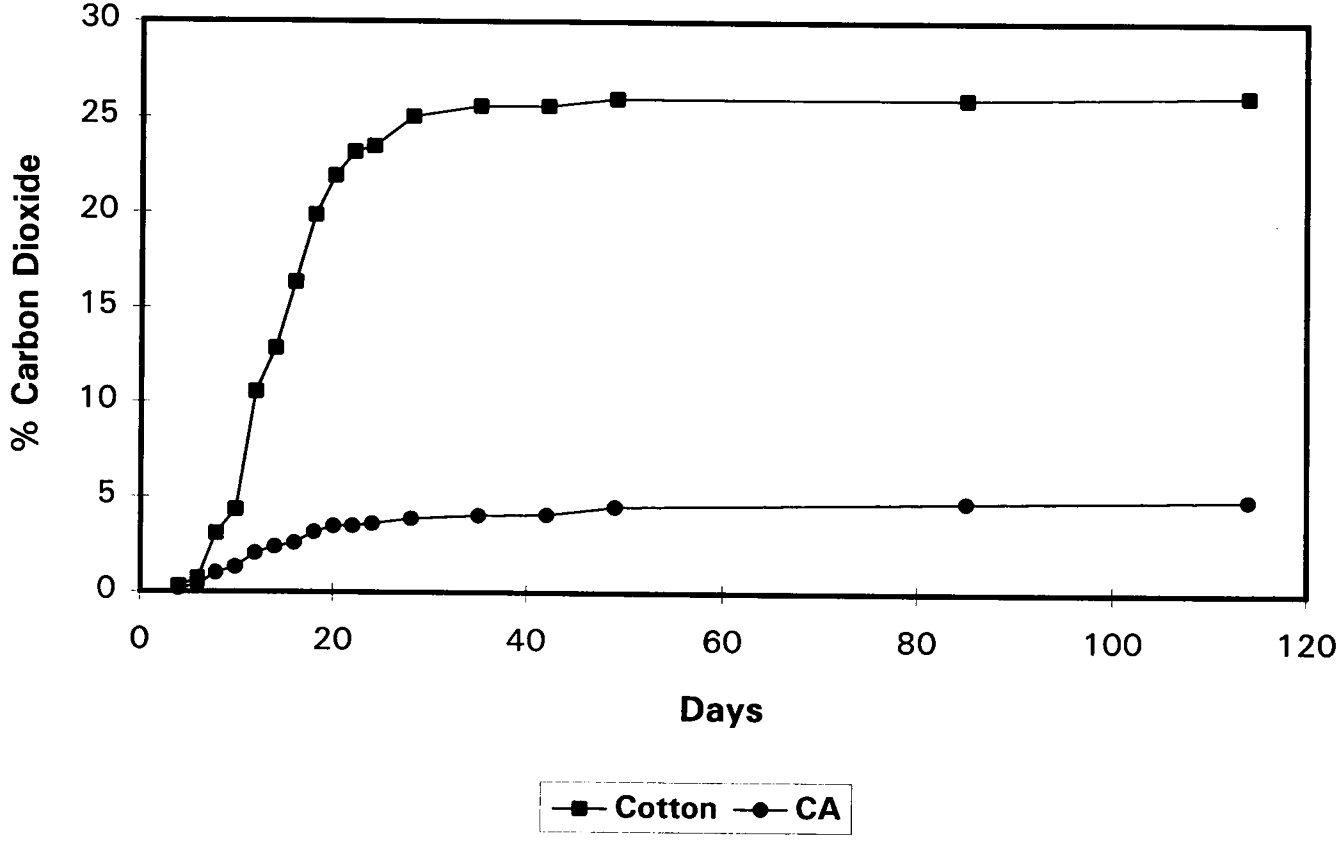


Fig. 6. Carbon Dioxide Evolution of Cotton and Cellulose Acetate Fibers.

For the cotton fiber alone, a total 26.1% of carbon dioxide was evolved after 114 days. Most of this carbon dioxide was produced within 20 days, a period of time comparable to essentially complete degradation in the soil burial test. The result was an indication of sufficient microbial activity of the bioreactors. Even though one of the criteria for the standardized procedure is that more than 70% carbon dioxide be evolved to denote degradation for positive control materials such as soluble cellulose and starch, it seemed impossible to obtain such high percentage of CO_2 evolution from cotton fibers. This attributed to the high degree of polymerization, high crystallinity and/or orientation values.

Although the biodegradability of cellulose - including cotton fabrics - has been intensively studied, most studies have been based almost solely on the weight or strength loss of cotton fabrics. Cotton fibers easily disintegrate from microbial attack resulting in 100% weight or strength loss within the first 20 days. However, 100% CO_2 evolution of cotton fibers is effectively impossible, mainly because of its large amount of crystalline microfibrils [14, 92]. Crystalline cellulose is highly resistant to enzymatic attack due to constricted action of the cellulase, especially endo-glucanase [92]. In addition, CO_2 evolution is an indication of mineralization of the polymeric chains, and oligomers and soluble cellobiose, which are also degraded products, should be taken into account. The final result depends on how one defines the biodegradability of polymeric materials. Another possible mechanism contributing to the biodegradation of cotton fiber is the limited action of β-glucosidases responsible for cellobiose elimination and rate of biodegradation. Therefore, complete conversion of cotton fibers to glucose was not obtained, due possibly in part to the large amount of cellobiose accumulation which inhibits the action of both exo- and endo-glucanases.

The total percentage of carbon dioxide evolved from cellulose acetate fibers was 4.93% over 114 days. This was approximately one fifth of that of cotton fibers. Although CA fiber does not degrade as rapidly as cotton, there is little doubt that there is significant microbial activity producing esterase enzymes which contribute to its degradation.

The final results of pH change, total carbon dioxide evolved, TOC change and weight loss or remaining solid matters were significant (Table 5). Furthermore, there was increased acidification of the solutions. This is explained both by the increase in the amount of H^+ ion generated by carbonic acid, H_2CO_3, which is formed from CO_2 dissolved water, and by the increase in the amount of degraded fragments such as lactic acid and acetic acid. In addition, the increase in TOC from both the cotton and the CA bioreactors could be an indication of the increase in carbon content in solution solely from the test samples. No cotton fibers remained in the bioreactors, resulting in 100% weight loss. A large amount of algae was filtered out. For the CA fibers, weight loss could not be measured due to the inability to separate solid CA fractions and algae.

Table 5. Biodegradation Results of Cotton and Cellulose Acetate(CA) Fibers.

Sample (500 mg)	**Cotton**	**Cellulose Acetate**
Total Carbon Source	222 mg	246 mg
pH (From 7.9)	6.50	6.80
Total CO_2 Evolved	26.1 %	4.93 %
TOC content	19.8 ppm	1.866 ppm
Remaining Biomass/Fibers	96 mg	470 mg

Biodegradation of 50/50 cotton/CA Fibers

Throughout the second experiment, similar visual observations were made on the cotton and CA bioreactors. In the case of the bioreactor containing the 50/50 blend fibers, the solution began to clear, but still contained yellow fibrous materials after 9 days. The cumulative percentage of carbon dioxide evolution over time is shown in Figure 7. For the cotton and cellulose acetate fibers, total values of 27.04% and 9.18% carbon dioxide, respectively, were evolved at 45 days. This result provides further confirmation of the biodegradability of cotton and CA fibers and, also, demonstrates the reproducible microbial activity in the test method. The final results in pH and TOC changes, and the amount of biomass and remaining materials are shown in Table 6. In the experiment, 0.2 μm membrane filters were used for the complete filtration of microorganisms in the bioreactors. This resulted in an increase in the amount of biomass and remaining materials, and a decrease in the TOC changes.

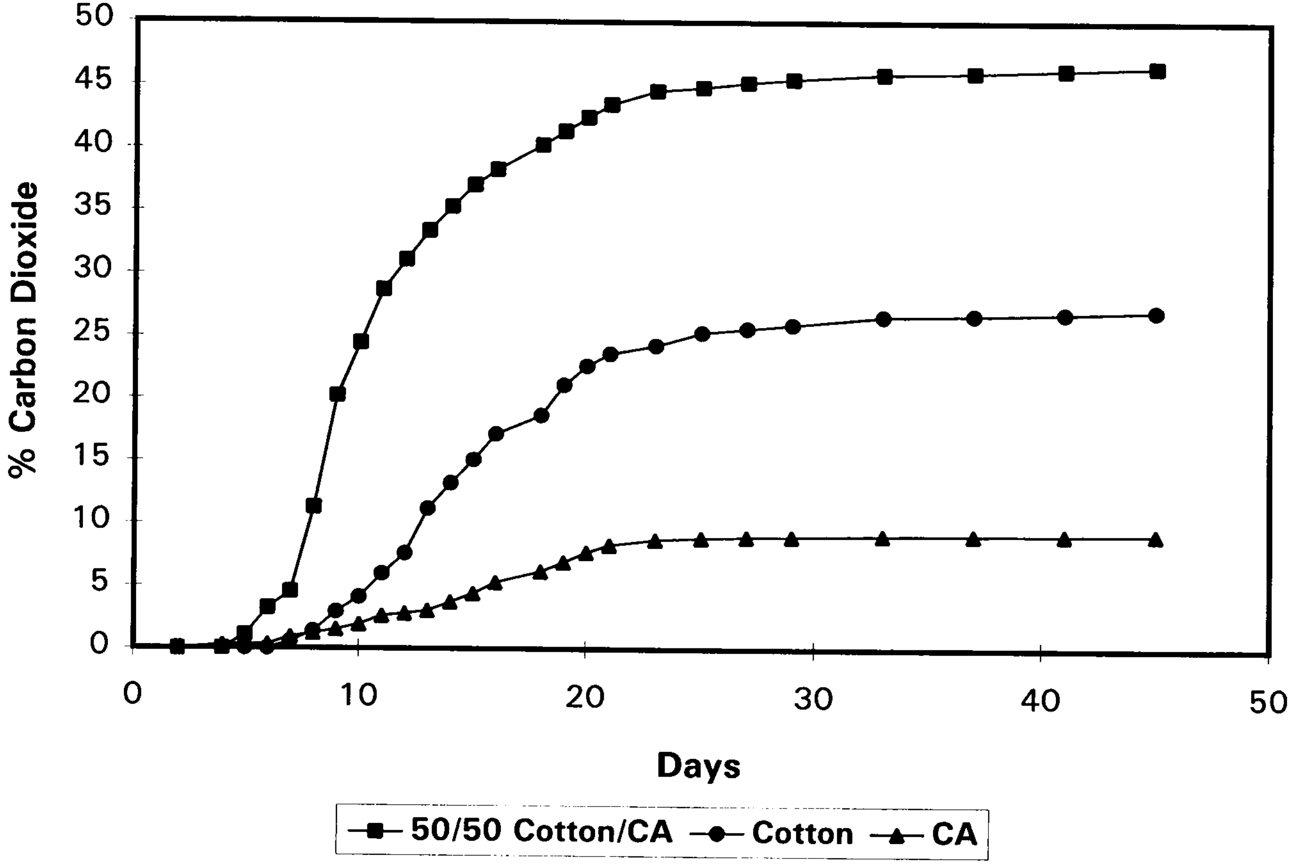


Fig. 7. Carbon Dioxide Evolution of 50/50 Cotton/CA, Cotton and CA Fibers.

The total percentage of carbon dioxide evolution for the cotton/CA blend was 46.5% over 45 days. This unexpected value was much greater than that of 100% cotton fibers. In addition, the rate of degradation was much faster than that of cotton fibers alone. This result suggests possible synergistic effects of esterase and cellulase enzymes, as well as the reduction of the cellobiose cumulation by increased activity of glucosidases. Therefore, the possible and important conclusion is that the greater amount of esterases and cellulases is induced in the presence of the two blended fibers.

Table 6. Biodegradation Results of 50/50 Cotton/CA, Cotton and CA Fibers.

Sample (500 mg)	**Cotton**	**50/50 Cotton/CA**	**CA**
Total Carbon Source	222 mg	234 mg	246 mg
pH (From 7.8)	6.48	6.35	6.71
Total CO_2 Evolved	27.04 %	46.5 %	9.18 %
TOC content	6.760 ppm	6.770 ppm	3.910 ppm
Remaining Biomass/Fibers	204.8 mg	245.2 mg	465.2 mg

Biodegradation of 75/25 and 25/75 Cotton/CA fibers

To begin to understand the synergistic effect of esterase and cellulase enzymes, cotton/ CA fibers with different blend ratios, 75/25 and 25/75, were tested along with 50/50 cotton/CA fibers as a positive control. Since the molecular weight and chemical structure of cotton and CA fibers are similar, the carbon content of each bioreactor containing 500 mg of fibers was in the same range. Therefore, there should be no effective difference in carbon source available for microbial activity. The cumulative percentage of carbon dioxide evolution over time and the final analyses are shown in Figure 8 and Table 7. For 50/50 cotton/CA blends, a total of 41.7% of carbon dioxide was evolved after 40 days. Also, the pH and TOC changes, and biomass and remaining material showed a similar trend to the results from the second test of 50/50 blend fibers.

The data from different blends of fibers indicated that the higher the cotton content the greater amount of carbon dioxide evolved. The qualitative results were 55.5, 41.7 and 30.5%, respectively, for the 75/25, 50/50 and 25/75 cotton/CA blends. Also, the pH and TOC changes were greater in the bioreactor solution of high cotton content. The amount of carbon dioxide evolved from the bioreactors containing the fiber blends, regardless of blend ratio, was greater than that of the individual fiber. This confirmed the likely synergistic effect of esterase and cellulase enzymes. Moreover, the greater carbon dioxide evolution and the faster rate of biodegradation of fiber blends with a higher cotton content imply that the cellulase enzymes were favorably induced over esterase.

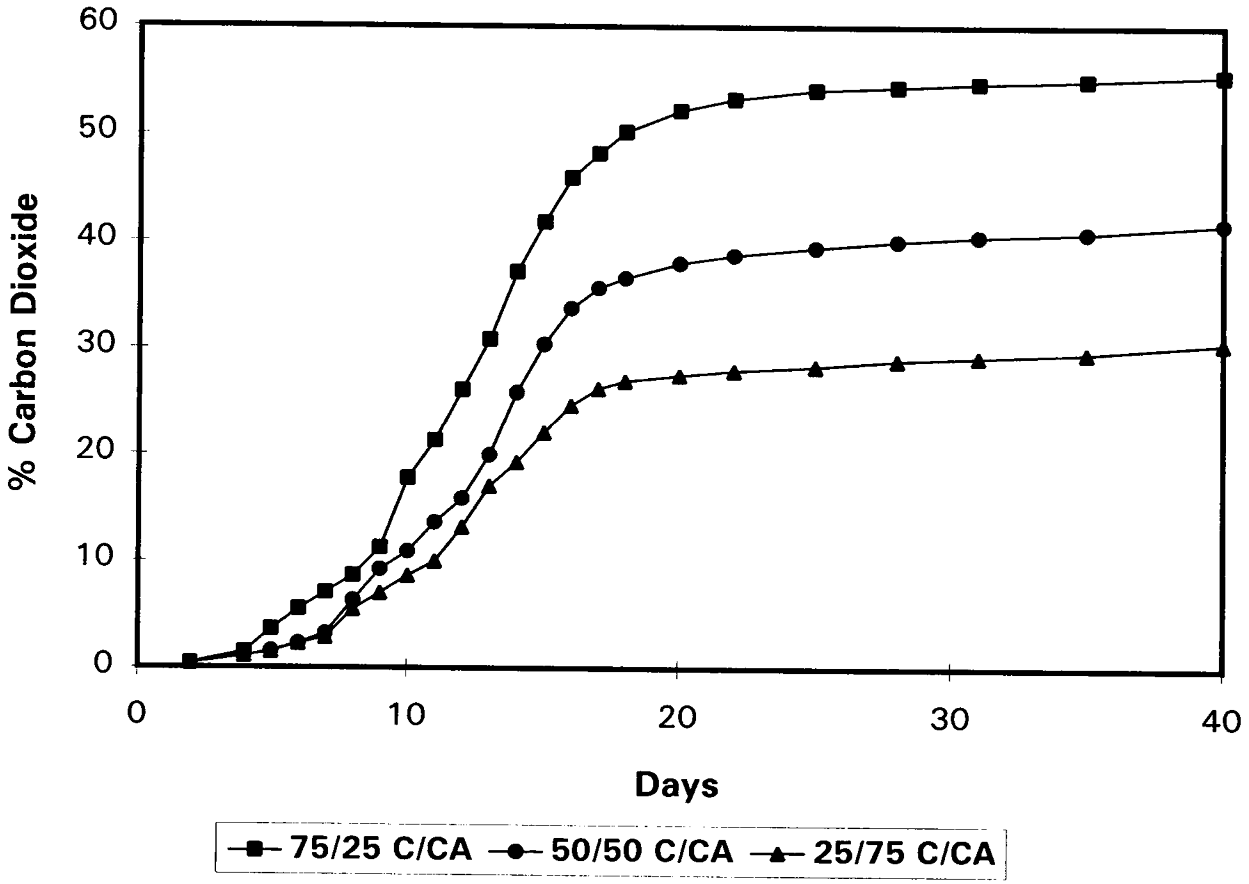


Fig. 8. Carbon Dioxide Evolution of 75/25, 50/50 and 25/75 Cotton/CA Blend Fibers.

Table 7. Biodegradation Results of 75/25, 50/50 and 25/75 Cotton and CA Blend Fibers

Sample (500 mg)	**75/25 Cotton/CA**	**50/50 Cotton/CA**	**25/75 Cotton/CA**
Total Carbon Source	228 mg	234 mg	240 mg
pH (From 7.8)	6.38	6.52	6.51
Total CO_2 Evolved	55.49 %	41.66 %	30.53 %
TOC content	6.794 ppm	6.693 ppm	6.038 ppm
Remaining Biomass/Fibers	217.5 mg	271.1 mg	316.7 mg

The biodegradation results provide a convincing view on the qualitative and quantitative biodegradability of cellulose acetate fibers. From activated sludge tests, reproducible biodegradability of fibers can be obtained through microstructural degradation and possibly enzymatic levels by carefully measuring carbon dioxide evolution. The data regarding the biodegradability of CA fibers and the enhanced biodegradability of cotton/ CA blends due to synergistic effects suggest a promising method in an effort to produce biodegradable/compostable fabrics.

PROCESS CHARACTERIZATION

SOLVENT-ASSISTED THERMAL CALENDERING

Carded webs of 75/25 cotton/CA fibers were thermally bonded at different temperature combinations for patterned (top) and smooth (bottom) rolls. The carded webs were first calendered without a solvent pretreatment at bonding temperatures in a range of 170 to 240°C to observe the effect of thermal conditions on the bonding properties of cellulose acetate to itself and to cotton fibers. The results of the tensile tests (Figures 9 and 10) showed that tensile strengths in both machine direction (MD) and cross direction (CD) increased with temperature as expected. There was a sharp rise in strength for the 230°C roll temperatures. MD strengths did not exceed 10 mN/tex until roll temperatures exceeded 20°C beyond the softening temperatures of cellulose acetate fibers (180-205 °C). The results of CD strengths showed a similar trend to those of the MD strengths. There was a general increase in strength with temperature, especially for bonding temperatures over 200°C. Except for temperatures over 200°C, CD strengths were less than 3 mN/tex.

The tensile behavior of thermally bonded nonwovens with solvent pretreatment is shown in Figures 11 and 12. The solvent pretreatments were carried out under different saturation times ranging from 30 minutes to 2 hours, with 30 minute intervals. Three combinations of bonding temperatures - 100, 170 and 180°C - were selected, which were lower than both the softening temperatures of cellulose acetate

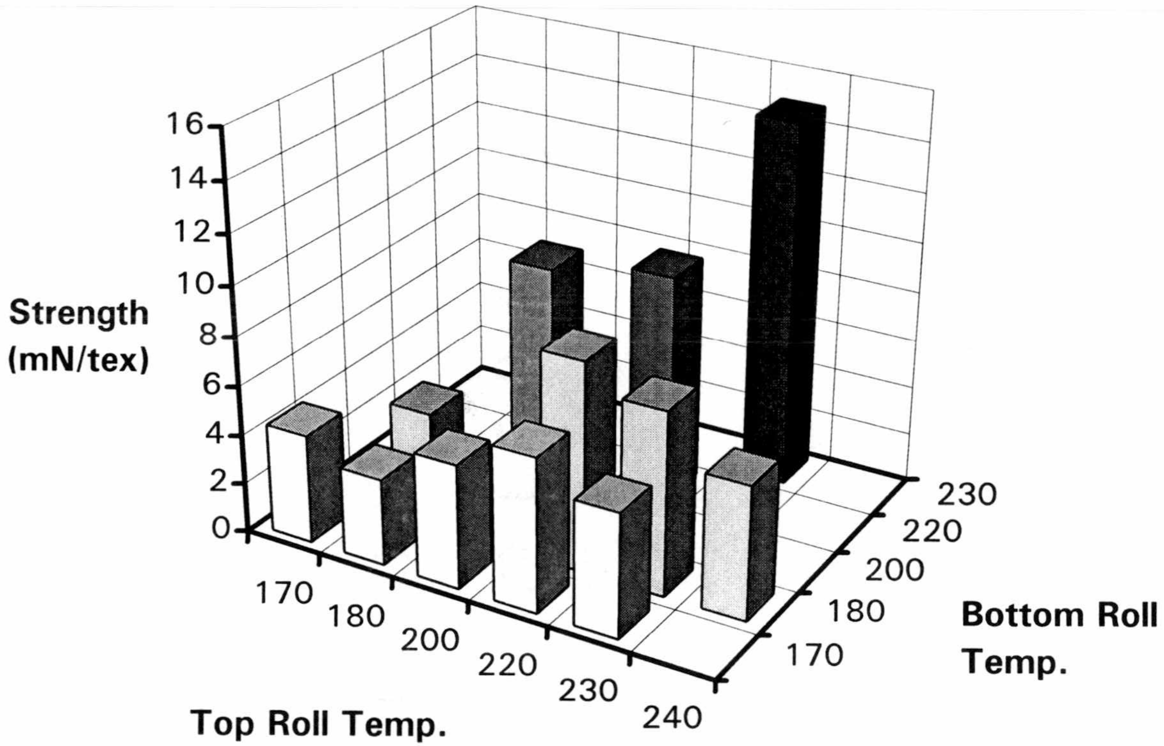


Fig. 9. Effects of Bonding Temperatures (^{o}C) on MD Strength.

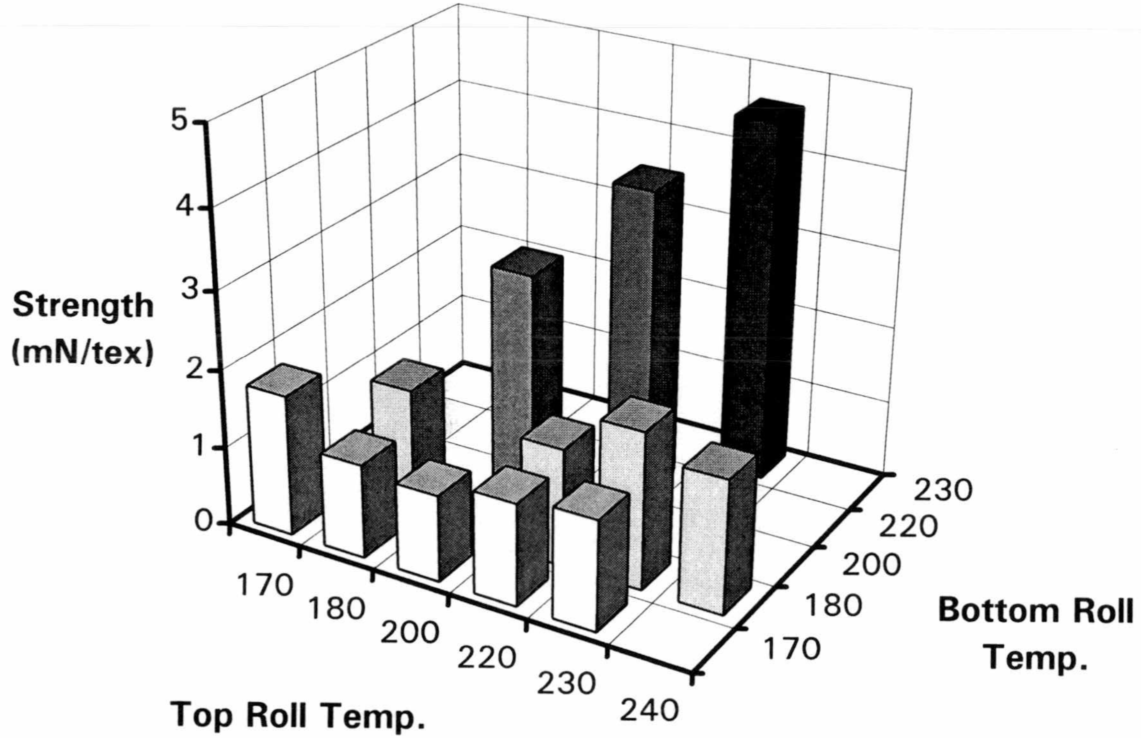


Fig. 10. Effects of Bonding Temperatures (^{o}C) on CD Strength.

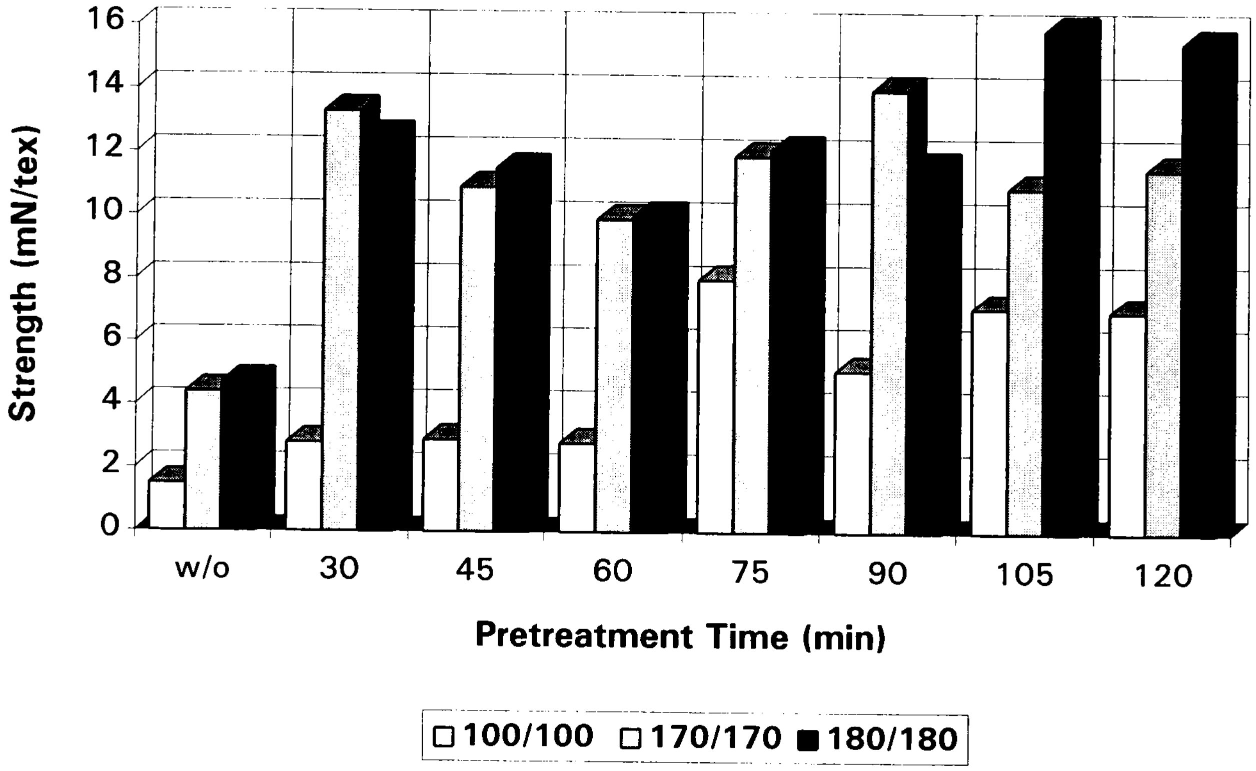


Fig. 11. Effects of Solvent Pretreatment Times on MD Strength.

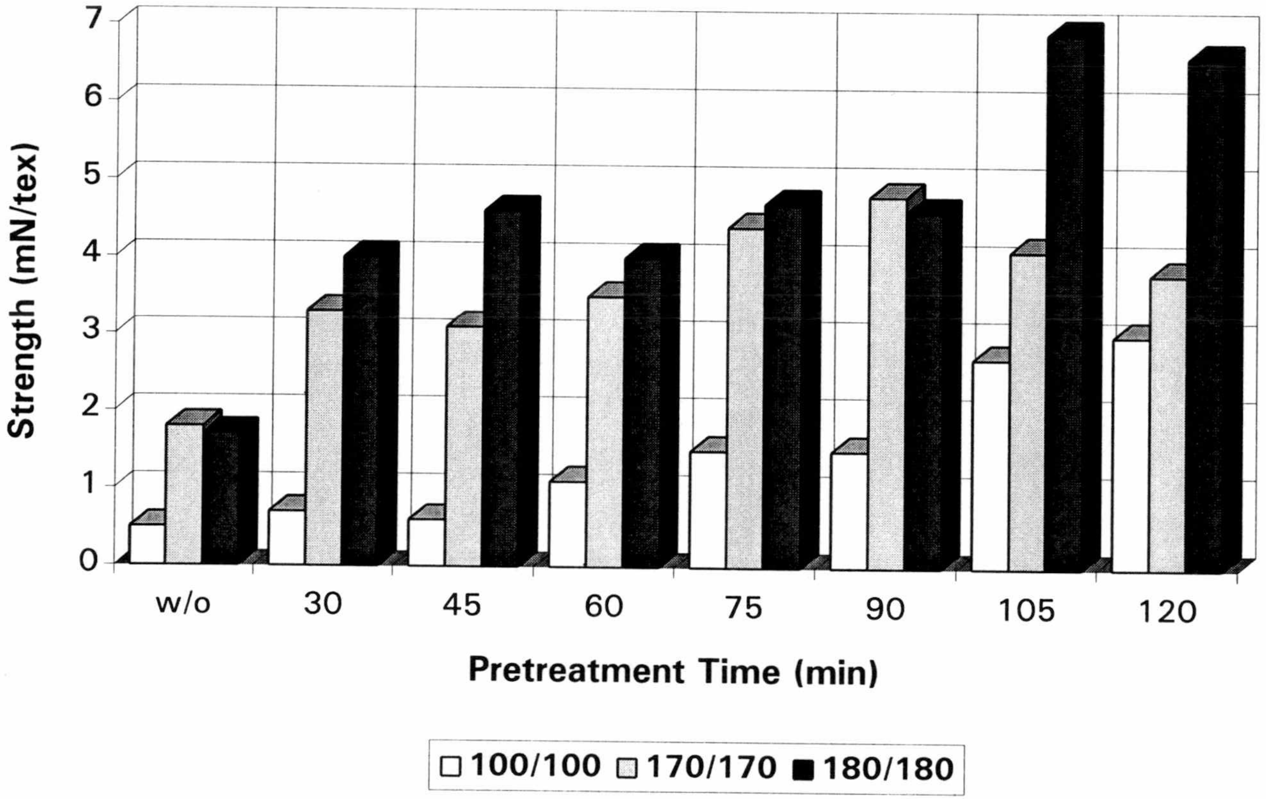


Fig. 12. Effects of Solvent Pretreatment Times on CD Strength.

and the selected temperatures for non-solvent studies. The results indicated that higher temperatures and longer pretreatment times provided greater strengths. Compared with non-treatment, the solvent pretreatment provided remarkable enhancement in tensile properties of MD and CD strengths. Most fabrics bonded at 170°C and 180°C with solvent pretreatments resulted in MD strengths exceeding 10 mN/tex and CD strength around 4 mN/tex, similar to those results obtained from nonwovens bonded at 230°C without solvent treatments. Even nonwovens bonded at 100°C showed increased strengths with increased solvent pretreatment times.

A remarkable strength enhancement occurred with nonwovens exposed to the acetone vapor for thirty minutes. The result probably means that surface softening is sufficient to activate a mechanism that raises the strength of the calendered fabric by a factor of nearly three, while doing so at low temperatures. Another possible explanation can be based on the mechanism of fiber-solvent interactions, in which solvents lower the softening or glass transition temperatures of fibers by the modification of amorphous regions. In any case, the short saturation pretreatment time was enough to modify the CA fiber on the surface or in the amorphous regions. That led to the enhanced bonding adhesion at surface temperatures lower than the original softening temperatures of CA fibers. The above effect could be extremely beneficial from an energy standpoint and from the knowledge that cotton fibers become brittle and weak when processed at temperatures much above 200°C.

PHYSICAL PROPERTIES

The raw data and the graphs of the eighteen physical properties of nonwoven fabrics produced by different processing conditions and blend ratios are located in Appendices A-2 and A-3. The effects of three processing factors on all the physical properties are examined in detail in the next section. The SEM photograph (Fig. 13) of solvent treated carded web before thermal calendering showed the softening of cellulose acetate fiber solely by the acetone vapor treatment. Selected SEM photographs of bonding region in fabrics with the lowest and highest strengths by blend ratio showed the bonding, or melting, behavior of the CA fibers (Fig. 14).

ANALYSIS OF VARIANCE (ANOVA)

The R-square values are the indicator of the strength of the relationship between variables. Especially in ANOVA, the indicator of degree of variation is explained by all factors. The results presented in Tables 8 and 9 showed that all physical properties were well explained by the model (all R-square values above 0.6).

The compilation of results show that eighteen dependent variables were tested for the effects of bonding temperatures, blend ratios, solvent pretreatment times and their two-way interactions. Overall, the physical variables were significantly affected by the processing conditions. Also some complicated interactions among the processing conditions showed up.

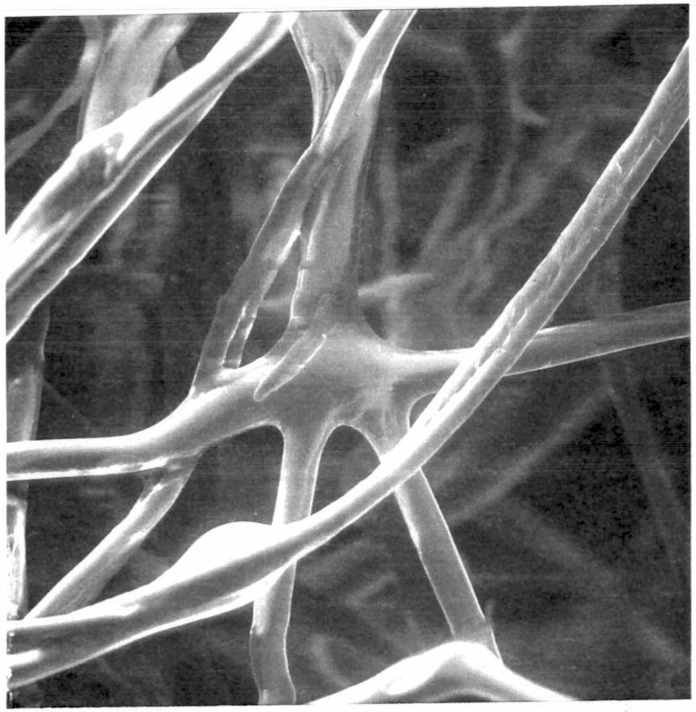
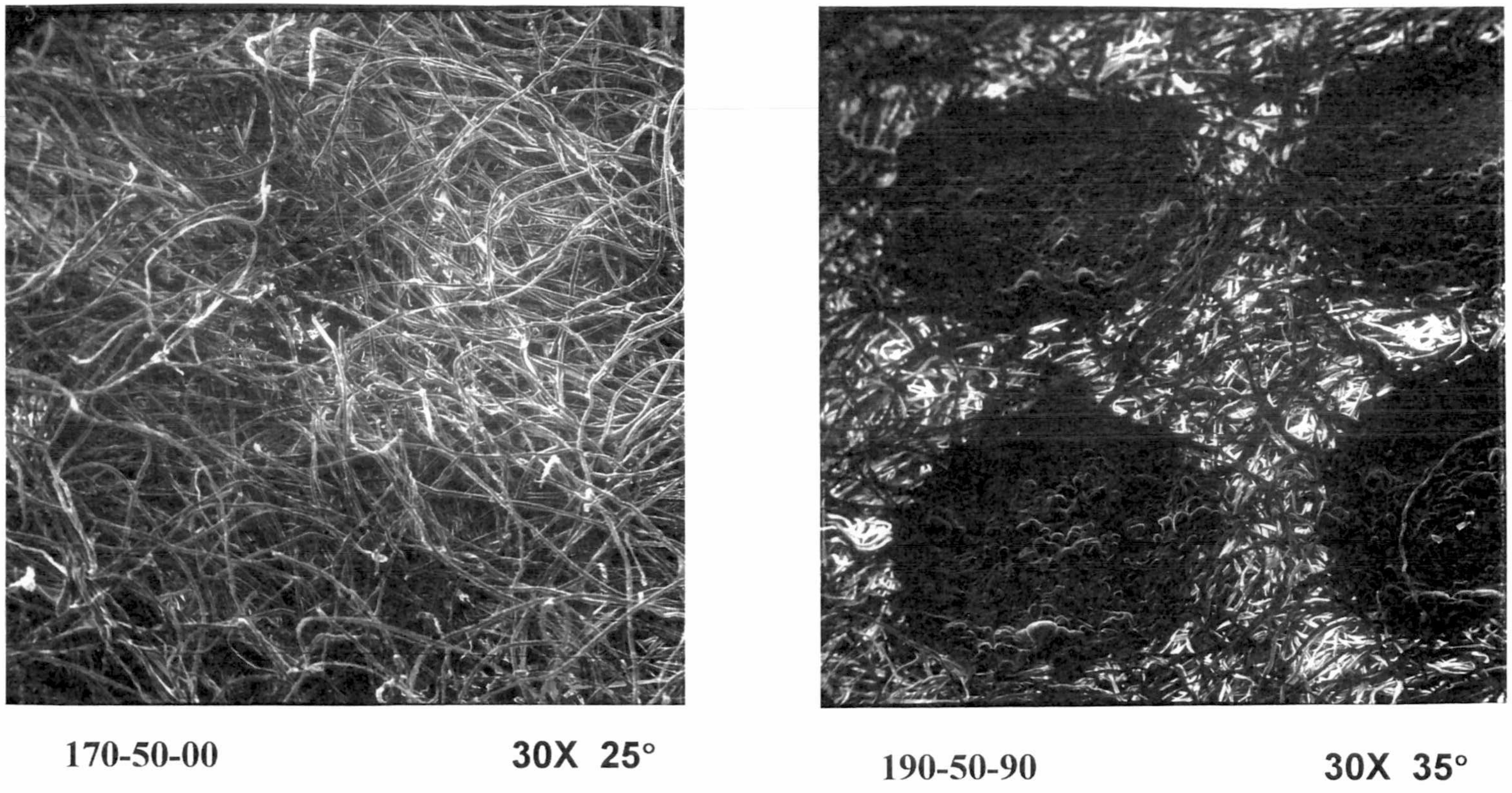


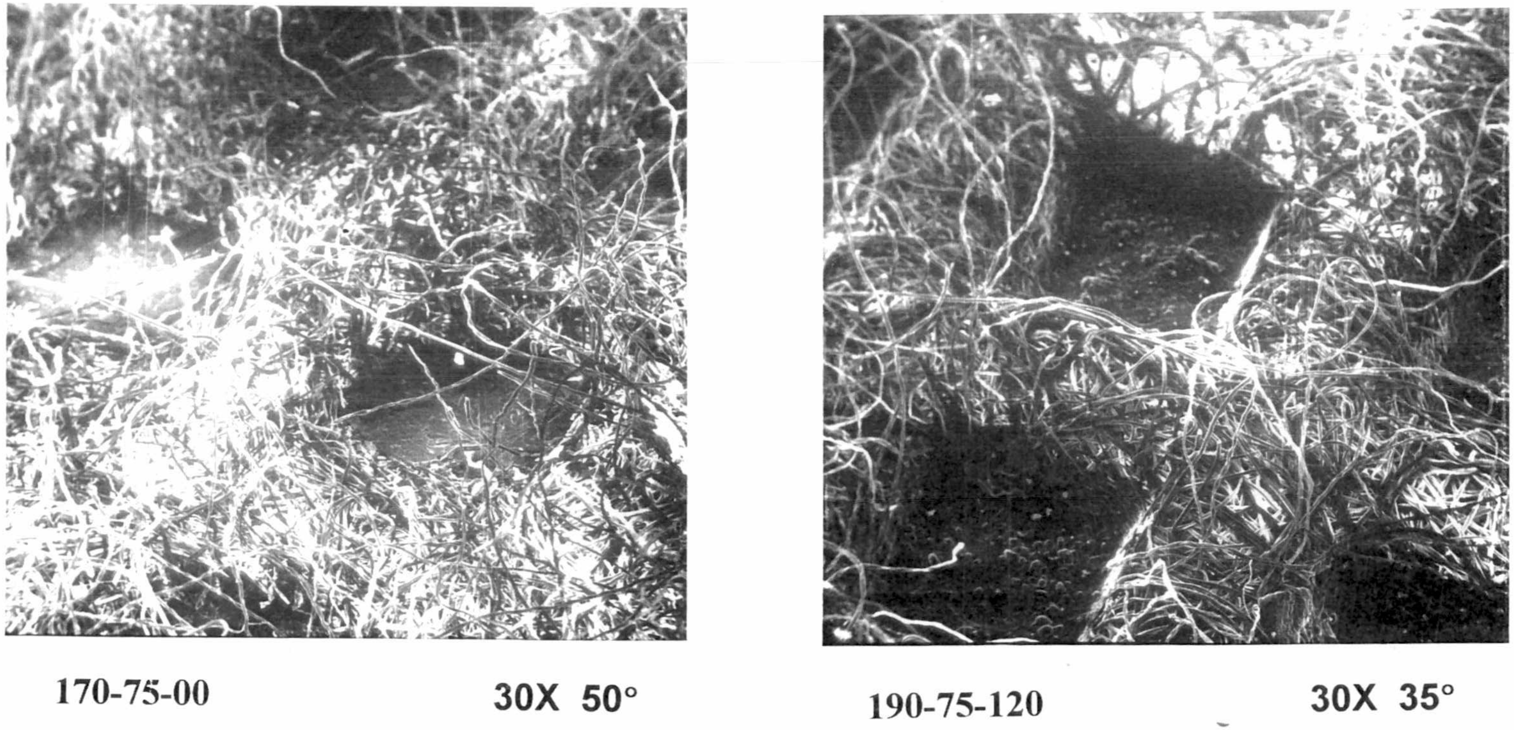
Fig. 13. SEM Photograph of Solvent Treated Carded Web from 75/25 Cotton/CA Blends.



(a) 50/50 cotton/CA, bonded at 170°.

(b) 50/50 cotton/CA, bonded at 190°
with 90 min solvent treatment

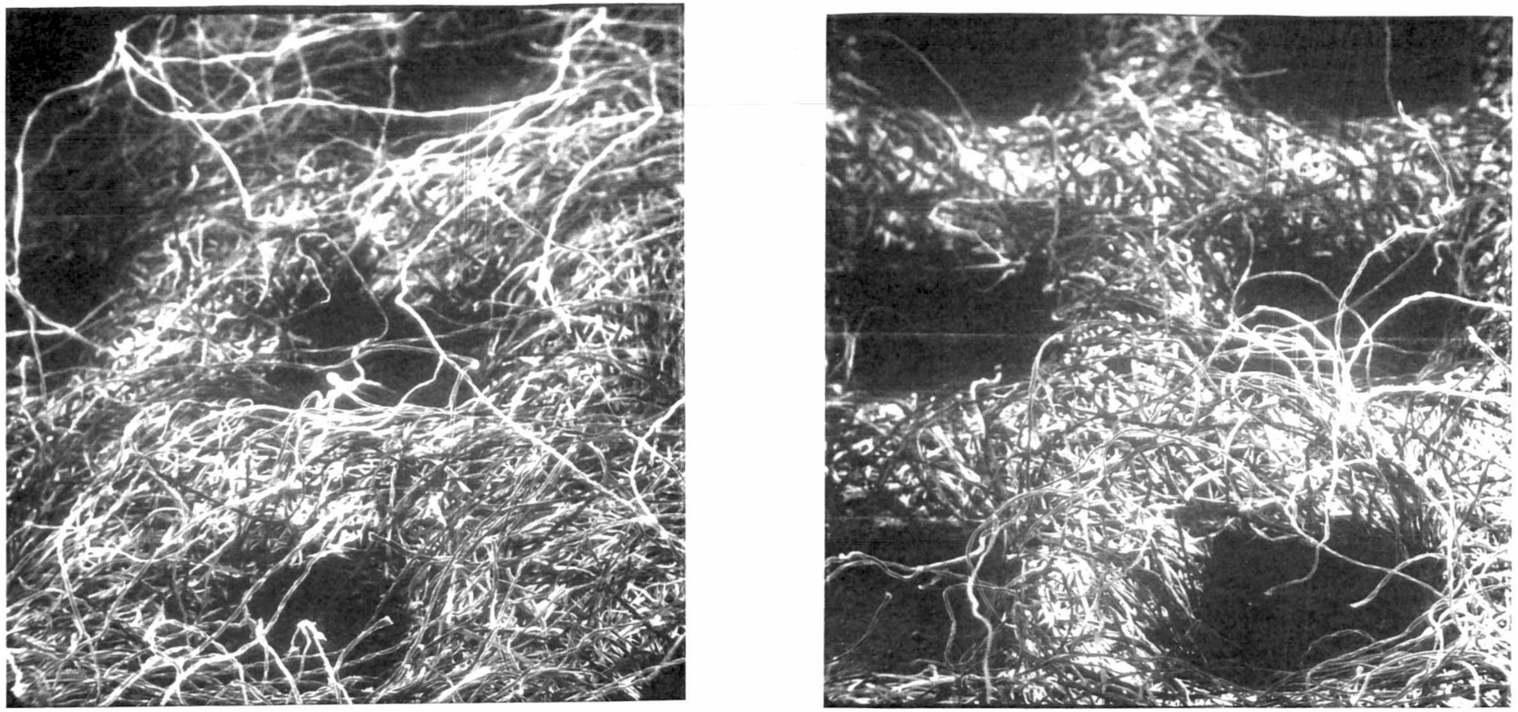
Fig. 14. SEM Photographs of Nonwovens from Different Blend Ratios.



(c) 75/25 cotton/CA, bonded at 170°.

(d) 75/25 cotton/CA, bonded at 170° with 120 min solvent treatment

Fig. 14. (continued)



170-85-00 **30X 50°**

(e) 85/15 cotton/CA, bonded at 170°.

170-85-45 **30X 50°**

(f) 85/15 cotton/CA, bonded at 170°
with 45 min solvent treatment

Fig. 14. (continued)

Table 8. F-Test Results from ANOVA.

Source (DF)	Basiswt	Thick	Airper	BendMD	BendCD	SstrsMD
T (2)	0.3095	0.0001	0.0001	0.0001	0.0001	0.0001
B (2)	0.0183	0.3718	0.1055	0.0001	0.0001	0.0001
TB (4)	0.1329	0.3366	0.0624	0.2126	0.2497	0.0061
S (7)	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001
TS (14)	0.9541	0.9281	0.8871	0.8190	0.8778	0.1735
BS (14)	0.7210	0.632	0.0176	0.0179	0.0102	0.0001
R-square	0.6166	0.8033	0.7747	0.9084	0.8724	0.9276

Source (DF)	SstrsCD	ElongMD	ElongCD	StrthMD	StrthCD	Flexall
T (2)	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001
B (2)	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001
TB (4)	0.0005	0.0001	0.0028	0.0134	0.0010	0.1824
S (7)	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001
TS (14)	0.2996	0.2455	0.0407	0.3338	0.4562	0.9210
BS (14)	0.0026	0.0001	0.0001	0.0017	0.0163	0.3406
R-square	0.9013	0.9120	0.9461	0.9069	0.8800	0.8303

Source (DF)	FlexMD	FlexCD	YstrsMD	YstrsCD	ImodMD	ImodCD
T (2)	0.0001	0.0001	0.0001	0.0001	0.0001	0.0003
B (2)	0.0001	0.0005	0.0001	0.0001	0.8179	0.5038
TB (4)	0.1519	0.2513	0.0093	0.0005	0.4049	0.5135
S (7)	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001
TS (14)	0.8089	0.8980	0.2985	0.3075	0.4618	0.7693
BS (14)	0.4103	0.3666	0.0003	0.0023	0.0125	0.1100
R-square	0.8460	0.7859	0.9210	0.9011	0.8189	0.7566

Table 9. Effects of Factors and Interactions on Physical Properties.
(* Significant differences among mean values due to the source variables with p<0.05)

	T	B	S	TB	TS	BS
Basiswt		*	*			
Thick	*		*			
Airper	*		*			*
BendMD	*	*	*			*
BendCD	*	*	*			*
SstrsMD	*	*	*	*		*
SstrsCD	*	*	*	*		*
ElongMD	*	*	*	*		*
ElongCD	*	*	*	*	*	*
StrthMD	*	*	*	*		*
StrthCD	*	*	*	*		*
Flexall	*	*	*			
FlexMD	*	*	*			
FlexCD	*	*	*			
YstrsMD	*	*	*	*		*
YstrsCD	*	*	*	*		*
ImodMD	*		*			*
ImodCD	*		*			

Especially, noteworthy was the observation that all physical properties were significantly affected by among solvent pretreatment times. Most of the physical properties, except for basis weight, showed significant differences in their mean values due to bonding temperature. In addition, blend ratio was an important factor affecting most of the properties except for thickness, air permeability, and initial modulus in MD and CD. Therefore, the results confirmed that bonding temperature, blend ratio and solvent treatment time were important factors in determining the physical properties of nonwovens.

In this study, interaction effects were significant factors of influence on the physical properties of nonwovens. Especially, the interactions between blend ratios and solvent pretreatment times, and between blend ratios and bonding temperatures were significant in some cases. Most properties except for basis weight, thickness, flexural rigidities, and initial modulus in CD were influenced by the interaction between the blend ratios and the solvent pretreatment times. In addition, the interactions between bonding temperatures and blend ratios influenced differences in mean values of specific stress in both MD and CD, elongation in MD and in CD, strength in MD and in CD, and yield stress in MD and in CD.

However, the interaction between bonding temperature and solvent pretreatment time showed only in CD elongation. No interaction between two variables indicates only an additive effects of the two factors (126). It means that changes in the levels of bonding temperature on physical properties result in the same difference between mean responses, irrespective of solvent treatment time. Therefore, the parallelism between bonding temperature and solvent treatment time provided the possible prediction of the effect of the next level of bonding temperature on each physical property for all levels of solvent treatment, since the differences of mean values of the two levels of the temperatures were known.

Interactions between blend ratio and bonding temperature, and blend ratio and solvent treatment time made it difficult to interpret the effects of the processing conditions on physical properties. This is possibly attributed to the uneven proportioning in the levels of blend ratios. Differences in mean values of some physical properties from the two blend ratios between 50/50 and 75/25 cotton/CA compared to those between 75/25 and 85/15 cotton/CA were not equal for all bonding temperatures or for all solvent treatment times. To avoid the complex interaction effects, analysis by blend ratios is more suitable, even though the number of data are tripled.

For basis weight, only blend ratio and solvent treatment time, rather than bonding temperature, was significantly important. This can be understood by the fact that the increase in basis weight for increased solvent treatment time possibly results from the shrinkage and compactness of the cellulose acetate fibers by the solvent. Furthermore, basis weight increased with a decrease in cellulose acetate content within a blend ratio. This is possibly due to the dissolving of cellulose acetate fibers. In addition, the lower basis weight values of 50/50 cotton/CA fabricated without solvent pretreatment indicated that cellulose acetate fibers were lost during the carding process.

Bonding temperature and solvent treatment time, not blend ratio, were significantly important factors in fabric thickness. It is well known that the thickness of thermally bonded nonwovens decreases with the increase in bonding adhesion.

There were significant differences in mean values attributed to the bonding temperatures, the solvent treatment times, blend ratios, and interactions between the temperatures and the blend ratios and between the solvent treatment times and the blend ratios, for most mechanical properties such as specific stress in MD and in CD, elongation in MD, strength in MD and in CD and yield stress in MD and in CD. For elongation in CD, all factors, i.e., main and interaction terms, affected mean values of the property. The implication is that the elongation in CD is difficult to predict by changing the processing conditions.

Significance differences appeared in MD and CD bending length among bonding temperature, among blend ratio, and among solvent treatment time. The interaction between blend ratio and solvent treatment time, also, played a role. However, differences in flexural rigidities were attributed only to the main factors and not to an interaction. The reason is probably that flexural rigidity values were obtained from normalization by basis weight values, which resulted in significant differences by blend ratio and solvent treatment time.

There were significant differences among the mean values of the initial modulus in MD and in CD for bonding temperature and solvent treatment time, but not for blend ratio.

Results from ANOVA demonstrated the importance in processing conditions and blend ratio on the physical properties of the cotton/CA nonwovens in this study, and that the factors influencing the differences in mean values of each property in MD, except for initial modulus, were the same as those in CD.

LEAST-SQUARE-MEANS (LSM) COMPARISON

The least square means (LSM), which are balanced means, were compared to discriminate and to identify the effects of the factor level on each physical property. The results of ANOVA showed that most properties were significantly affected by the main factors. The effect of the interaction terms was more difficult to interpret and differentiate. The main factors like bonding temperature, blend ratio and solvent treatment time were evaluated by the multiple-comparison procedure. The rank of sample LSmeans was assigned from lowest LSmean value (letter 'a') to highest (letter 'c').

Levels of Bonding Temperatures

Most of the physical properties showed significant differences ($p < 0.05$) in LSmeans among the temperature levels, 150, 170 and 190°C (Table 10). For basis weight, however, there were no significant differences among the LSmeans in temperature levels (a, a, a), although the actual LSmeans of basis weight were higher at a higher bonding temperature. Like the results from ANOVA, the bonding temperatures did not affect the basis weight of the nonwovens.

Elongation in MD and initial modulus in CD showed no significant differences in their LSmean values for bonding temperatures between 150 and 170°C. However, the LSmeans of elongation in MD and initial modulus in CD at the bonding temperature of 190°C were significantly greater than those from lower bonding temperatures.

Thickness and air permeability values were significantly lower for an increase in the level of bonding temperatures. An increase of 20°C of bonding temperatures had a significant influence on the fabrication of thinner and less breathable nonwovens from cotton/CA fibers.

Most of the mechanical properties like bending length in MD and in CD, specific stress in MD and CD, elongation in CD, strength in MD and in CD, overall flexural rigidity, flexural rigidity in MD and in CD, yield stress in MD and in CD and initial modulus in MD, showed significant increases in their LSmean values with an increase in the each level of bonding temperatures. Therefore, the conclusion is that the selection of bong temperatures is one of the important processing parameters determining the physical properties of nonwovens.

Levels of Blend Ratios

Like results from ANOVA, thickness, air permeability and initial modulus in MD and in CD showed insignificant differences in all levels of blend ratio (a, a, a) (Table

10), although the higher cotton content produced a greater actual LSmeans of thickness and a less actual LSmeans of air permeability. This implies a bulkier and breathable nonwoven. The initial modulus values did not have any specific trend.

The results of LSM comparison indicated that the basis weight of 50/50 cotton/CA nonwovens was significantly less than that of 85/15, but not different from that of 75/25 cotton/CA nonwovens. Weight loss of cellulose acetate fibers during carding and/or during solvent pretreatment, and/or shrinkage of cellulose acetate fibers during solvent pretreatment and/or during thermal calendering might be reasons for lower basis weight of the nonwovens having higher cellulose acetate content.

In addition, the results of bending length in MD and in CD, overall flexural rigidity and flexural rigidity in MD and in CD, indicated that the LSmean values from 50/50 cotton/CA nonwovens were significantly less than those from 75/25 or 85/15 cotton/CA nonwovens (a, b, b). The higher cotton content blends resulted in less flexible nonwovens.

The LSmeans of specific stresses in MD and in CD, elongations in MD and in CD, strengths in MD and in CD, and yield stresses in MD and in CD showed significant differences in their values for each level of blend ratio (c, b, a). The higher cellulose acetate content nonwovens produced tougher thermally bonded nonwovens. For those physical properties, the choice of blend ratios was the most important factor in the fabrication of nonwovens from cotton/CA blend fibers.

Table 10. Results from Least-Square-Mean Comparison ($p>0.05$) for Bonding Temperatures and Blend Ratio.
(from a ; lowest value to c ; highest value, and same letters indicating the insignificant difference in the LSmean values)

Dependent Variable	**Bonding Temperature** **150 170 190**	**Blend Ratio** **50 75 85**
Basiswt	a < a < a *	a < a b < b *
Thick	c > b > a	a < a < a *
Airper	c > b > a	a > a > a *
BendMD	a < b < c	a < b < b *
BendCD	a < b < c	a < b < b *
SstrsMD	a < b < c	c > b > a
SstrsCD	a < b < c	c > b > a
ElongMD	a > a < b *	c > b > a
ElongCD	a < b < c	c > b > a
StrthMD	a < b < c	c > b > a
StrthCD	a < b < c	c > b > a
Flexall	a < b < c	a < b < b *
FlexMD	a < b < c	a < b < b *
FlexCD	a < b < c	a < b < b *
YstrsMD	a < b < c	c > b > a
YstrsCD	a < b < c	c > b > a
ImodMD	a < b < c	a < a > a *
ImodCD	a < a < b *	a < a > a *

* at least one of the LSmean values are not significantly different from the other LSmean values with $p=0.05$.

Levels of Solvent Pretreatment Time

Solvent pretreatment times as influences on physical properties were ranked from lowest to highest values of LSmeans of each dependent variable (Table 11). The LSmean values of most of the physical properties increased with time of solvent treatment. However, all physical properties showed the significant differences between untreated nonwovens and the treated fabric over 30 to 120 minutes of pretreatment times.

The LSmeans of air permeability, and the elongations in MD and in CD did not show significant differences in solvent pretreated samples for any solvent pretreatment time levels. In addition, the elongations in MD and in CD showed an inconsistent ranking of LSmeans with the extent of solvent treatment time. The greater the solvent treatment time the stronger, the less extensible were the nonwovens. It is difficult to predict elongation properties of the nonwovens by the amount of the solvent pretreatment time.

The LS means of basis weight from 30 to 90 minutes of solvent pretreatment time were significantly less than that at 120 minutes. Also, the 30 minute solvent treatment resulted in a significantly lower basis weight than compared to that from a 90 minute treatment. The LSmeans of fabric thickness were lower for solvent pretreatment times of 105 to 120 minutes as compared to that of 30 minute treatment. The longer the solvent pretreatment, the thinner and the heavier the nonwovens, even though only 105 and 120 minute treatments are really significantly different from 30 minute treatments.

Table 11. Results of Least-Square-Mean Comparison ($p < 0.05$) for Solvent Treatment Times.
(******* indicating the insignificant differences in their LSMs)

<table>
<tr><th rowspan="2">Dependent Variable</th><th colspan="8">Solvent Pretreatment Time</th></tr>
<tr><th colspan="8">Lowest ←——————→ Highest</th></tr>
<tr><td rowspan="4">Basiswt</td><td>0</td><td>30</td><td>60</td><td>45</td><td>90</td><td>75</td><td>105</td><td>120</td></tr>
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<tr><td></td><td></td><td></td><td colspan="5">***********************</td></tr>
<tr><td rowspan="3">Thick</td><td>105</td><td>90</td><td>120</td><td>75</td><td>60</td><td>45</td><td>30</td><td>0</td></tr>
<tr><td colspan="6">****************************</td><td></td><td></td></tr>
<tr><td></td><td></td><td></td><td colspan="4">****************</td><td></td></tr>
<tr><td rowspan="2">Airper</td><td>120</td><td>90</td><td>75</td><td>105</td><td>45</td><td>60</td><td>30</td><td>0</td></tr>
<tr><td colspan="7">*********************************</td><td></td></tr>
<tr><td rowspan="4">BendMD</td><td>0</td><td>30</td><td>45</td><td>60</td><td>75</td><td>90</td><td>105</td><td>120</td></tr>
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<tr><td rowspan="4">BendCD</td><td>0</td><td>45</td><td>30</td><td>60</td><td>75</td><td>90</td><td>105</td><td>120</td></tr>
<tr><td></td><td colspan="3">***********</td><td></td><td></td><td></td><td></td></tr>
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<tr><td></td><td></td><td></td><td></td><td colspan="4">******************</td></tr>
<tr><td rowspan="5">SstrsMD</td><td>0</td><td>30</td><td>45</td><td>60</td><td>75</td><td>90</td><td>120</td><td>105</td></tr>
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<tr><td rowspan="5">SstrsCD</td><td>0</td><td>30</td><td>45</td><td>60</td><td>75</td><td>90</td><td>105</td><td>120</td></tr>
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<tr><td></td><td></td><td></td><td></td><td></td><td colspan="3">*************</td></tr>
<tr><td rowspan="2">ElongMD</td><td>0</td><td>90</td><td>120</td><td>75</td><td>105</td><td>60</td><td>45</td><td>30</td></tr>
<tr><td></td><td colspan="7">**********************************</td></tr>
<tr><td rowspan="2">ElongCD</td><td>0</td><td>105</td><td>75</td><td>30</td><td>90</td><td>120</td><td>60</td><td>45</td></tr>
<tr><td></td><td colspan="7">**********************************</td></tr>
<tr><td rowspan="4">StrthMD</td><td>0</td><td>30</td><td>45</td><td>60</td><td>75</td><td>90</td><td>105</td><td>120</td></tr>
<tr><td></td><td colspan="3">***********</td><td></td><td></td><td></td><td></td></tr>
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Table 11. (continued)

<table>
<tr><th rowspan="2">Dependent Variable</th><th colspan="8">Solvent Pretreatment Time</th></tr>
<tr><th colspan="8">Lowest ⟵⟶ Highest</th></tr>
<tr><td rowspan="5">StrthCD</td><td>0</td><td>30</td><td>45</td><td>60</td><td>75</td><td>90</td><td>105</td><td>120</td></tr>
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<tr><td></td><td></td><td></td><td></td><td></td><td colspan="3">*************</td></tr>
<tr><td rowspan="5">Flexall</td><td>0</td><td>30</td><td>45</td><td>60</td><td>75</td><td>90</td><td>105</td><td>120</td></tr>
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<tr><td rowspan="5">FlexMD</td><td>0</td><td>30</td><td>45</td><td>60</td><td>75</td><td>90</td><td>105</td><td>120</td></tr>
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<tr><td rowspan="4">FlexCD</td><td>0</td><td>45</td><td>30</td><td>60</td><td>75</td><td>90</td><td>105</td><td>120</td></tr>
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<tr><td rowspan="4">YstrsMD</td><td>0</td><td>30</td><td>45</td><td>60</td><td>75</td><td>90</td><td>105</td><td>120</td></tr>
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<tr><td rowspan="4">ImodMD</td><td>0</td><td>30</td><td>45</td><td>60</td><td>75</td><td>90</td><td>105</td><td>120</td></tr>
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<tr><td rowspan="2">ImodCD</td><td>0</td><td>45</td><td>30</td><td>60</td><td>75</td><td>90</td><td>105</td><td>120</td></tr>
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The results of the multi-mean comparisons for bending lengths in MD and in CD, specific stresses in MD and in CD, strengths in MD and in CD, flexural rigidities, yield stresses in MD and in CD and initial moduli in MD and in CD provided similar trends in their LSmean differences with solvent pretreatment times. The LSmeans of the above properties from treatment times of 30 to 60 minutes were significantly less than those of 90 to 120 minutes. There were no differences in the values from 90 to 120 minutes. Therefore, there may be no gain in pretreating the carded webs more than 90 minutes to achieve the higher values in those above properties.

In more detail, bending lengths in MD and in CD of the nonwovens with the solvent treatment times of 30 and 45 minutes were significantly less than those of 75 minutes. The specific stress values in MD from 30 and 45 minutes were less than those from 75 minutes, which were less than those of 105 minutes. The LSmeans of specific stress in CD showed that the 30 and 75 minute treatment times resulted in lower values than those of 75 and 120 minute pretreatment times. The same result was obtained for strength in CD. However, the strength in MD showed that only values from solvent treatment times of 30 to 45 minutes were less than those of 75 minutes.

Detailed results of the overall flexural rigidity showed that the LSmeans for 30 and 45 minute solvent treatments were less than those obtained from 75 minutes. The latter were less than those of 120 minutes. The LSmean values of flexural rigidity in MD for the solvent treatment times of 30 minutes were significantly less than those from 75 minutes, and the values from 60 and 75 minutes were less than those of 120 minutes. Also, the LSmeans of the flexural rigidity in CD from the solvent treatment times of 30 and 45 minutes were significantly less than those of 75 minutes.

For yield stress in MD, the values from solvent treatment times of 30 and 45 minutes were significantly less than those of 75 minutes. Also, the LS mean values of yield stress in CD from the treatment times of 30 minutes were significantly less than those of 75 minutes, and the latter were less than those of 120 minutes. For the initial modulus in MD, the values from treatment times of 30 to 60 minutes were significantly less than those of 90 minutes.

Overall, the solvent pretreatment significantly affect all of the physical properties. In addition, the physical properties - except for air permeability and elongations in MD and in CD - were significantly different for the levels of the solvent treatment times. However, the above properties did not show the significant improvement by increasing the solvent treatment times from 90 to 120 minutes. Also, the properties at shorter treatment times, such as 30 and 45 minutes, were significantly improved by increasing the amount of treatment time to 60, 75 and 90 minutes. Therefore, change in solvent pretreatment times was necessary to affect physical properties and most of the properties were improved by the increase in the treatment times. Longer treatment times such as 120 minutes were not necessary to achieve the highest values of each property.

REGRESSION MODELS

Regression Model

Regression analysis is a very useful technique for describing cause-effect relationships between independent and dependent variables. As a part of response surfaces, the regression model of the physical properties were first estimated. This was necessary to generate adequate response surfaces and to optimize each property with processing conditions and blend ratios. All physical properties were well characterized by full 2-nd order polynomial regression models, the model included linear (B, T, S), quadratic (B^2, T^2, S^2), and crossproduct (TB, SB, ST) terms. In addition, the correlation (R-square) between the independent variables - blend ratios (B), bonding temperatures (T) and solvent treatment times (S), and the dependent variables. The relationships and the results of lack of fit test are listed in Table 12.

The total regression R-squares, adding linear, quadratic and crossproduct correlations, ranged from 0.4496 to 0.8448. The lack of fit F-tests determined the appropriateness of fitness of the models, with $p > 0.05$. Higher p values denote a more appropriate model. All physical properties, except elongations in MD (p>F: 0.0003) and in CD (p>F: 0.0004), showed that the 2-nd order equations were well fitted. Probably the higher order equations would be more suitable to describe the elongation models when considering the complicated elongation mechanisms of nonwovens. Also, the full 2-nd order equations could be simplified by elimination methods such as stepwise processes. However, the regression equations presented were used for the

Table 12. Regression Models for Physical Properties (* not significant with $\alpha = 0.05$).

Dependent Variable	**Regression Model**
Basiswt	$= 105.82 + 0.0861\mathbf{B} + 0.3446\mathbf{T} + 0.3839\mathbf{S}$ $+ 0.00165\mathbf{B}^2 - 0.000595\mathbf{T}^2 - 0.000646\mathbf{S}^2$ $- 0.000564\mathbf{TB} - 0.00157\mathbf{SB} - 0.000581^{\mathbf{ST}}$
	$R^2 = 0.4496$ Lack of Fit (P>F) = 0.9951
Thick	$= 1.477 - 0.00375\mathbf{B} - 0.00338\mathbf{T} - 0.00463\mathbf{S}$ $+ 0.0000216\mathbf{B}^2 + 0.00000574\mathbf{T}^2 + 0.0000124\mathbf{S}^2$ $- 0.00000187\mathbf{TB} + 0.0000222\mathbf{SB} + 0.00000478\mathbf{ST}$
	$R^2 = 0.7066$ Lack of Fit (P>F) = 0.6026
Airper	$= 90.39 - 0.2975\mathbf{B} - 0.3728\mathbf{T} - 0.5375\mathbf{S}$ $- 0.00281\mathbf{B}^2 + 0.0000348\mathbf{T}^2 + 0.00109\mathbf{S}^2$ $+ 0.002805\mathbf{TB} + 0.002465\mathbf{SB} + 0.000979\mathbf{ST}$
	$R^2 = 0.6515$ Lack of Fit (P>F) = 0.9749
BendMD	$= - 9.387 + 0.0961\mathbf{B} + 0.093\mathbf{T} + 0.06147\mathbf{S}$ $- 0.000324\mathbf{B}^2 - 0.000199\mathbf{T}^2 - 0.000201\mathbf{S}^2$ $- 0.0000784\mathbf{TB} - 0.00297\mathbf{SB} + 0.000000231^{\mathbf{ST}}$
	$R^2 = 0.8319$ Lack of Fit (P>F) = 0.2372
BendCD	$= - 8.433 + 0.0540\mathbf{B} + 0.08956\mathbf{T} + 0.04878\mathbf{S}$ $- 0.0000577\mathbf{B}^2 - 0.000197\mathbf{T}^2 - 0.000113\mathbf{S}^2$ $- 0.0000907\mathbf{TB} - 0.000262\mathbf{SB} - 0.0000302\mathbf{ST}$
	$R^2 = 0.7797$ Lack of Fit (P>F) = 0.6957
SstrsMD	$= - 59.03 + 0.5568\mathbf{B} + 0.4166\mathbf{T} + 0.3516\mathbf{S}$ $- 0.002883\mathbf{B}^2 - 0.000813\mathbf{T}^2 - 0.00114\mathbf{S}^2$ $- 0.000802\mathbf{TB} - 0.00251\mathbf{SB} + 0.000290\mathbf{ST}$
	$R^2 = 0.8448$ Lack of Fit (P>F) = 0.3734
SstrsCD	$= - 19.33 + 0.2342\mathbf{B} + 0.1113\mathbf{T} + 0.1011\mathbf{S}$ $- 0.00120\mathbf{B}^2 - 0.000176\mathbf{T}^2 - 0.000310\mathbf{S}^2$ $- 0.000304\mathbf{TB} - 0.001097\mathbf{SB} + 0.000287\mathbf{ST}$
	$R^2 = 0.8026$ Lack of Fit (P>F) = 0.6076
ElongMD	$= 126.98 - 0.5573\mathbf{B} - 1.2047\mathbf{T} + 0.2746\mathbf{S}$ $- 0.001166\mathbf{B}^2 + 0.003115\mathbf{T}^2 - 0.001223\mathbf{S}^2$ $+ 0.003051\mathbf{TB} - 0.000583\mathbf{SB} - 0.000218\mathbf{ST}$
	$R^2 = 0.6563$ Lack of Fit (P>F) = 0.0003*
ElongCD	$= - 25.45 + 0.1109\mathbf{B} + 0.3197\mathbf{T} + 0.4224\mathbf{S}$ $- 0.000807\mathbf{B}^2 - 0.000589\mathbf{T}^2 - 0.001443\mathbf{S}^2$ $- 0.000741\mathbf{TB} - 0.002246\mathbf{SB} - 0.000196\mathbf{ST}$
	$R^2 = 0.8073$ Lack of Fit (P>F) = 0.0004*

Table 12. (continued)

Dependent Variable	**Regression Model**
StrthMD	$= -253.54 + 2.349\mathbf{B} + 1.792\mathbf{T} + 1.463\mathbf{S}$ $- 0.01281\mathbf{B}^2 - 0.003624\mathbf{T}^2 - 0.004550\mathbf{S}^2$ $- 0.002786\mathbf{TB} - 0.01050\mathbf{SB} + 0.001180\mathbf{ST}$
	$R^2 = 0.8222$ Lack of Fit (P>F) = 0.6270
StrthCD	$= -80.76 + 1.0124\mathbf{B} + 0.4465\mathbf{T} + 0.4146\mathbf{S}$ $- 0.005699\mathbf{B}^2 - 0.000744\mathbf{T}^2 - 0.001173\mathbf{S}^2$ $- 0.000891\mathbf{TB} - 0.004639\mathbf{SB} + 0.001212\mathbf{ST}$
	$R^2 = 0.7763$ Lack of Fit (P>F) = 0.7949
Flexall	$= -91.31 + 0.6101\mathbf{B} + 0.6740\mathbf{T} + 0.2979\mathbf{S}$ $- 0.003292\mathbf{B}^2 - 0.001696\mathbf{T}^2 - 0.001173\mathbf{S}^2$ $+ 0.000973\mathbf{TB} - 0.002138\mathbf{SB} + 0.000980\mathbf{ST}$
	$R^2 = 0.7383$ Lack of Fit (P>F) = 0.8298
FlexMD	$= -122.43 + 1.1016\mathbf{B} + 0.7388\mathbf{T} + 0.4956\mathbf{S}$ $- 0.006586\mathbf{B}^2 - 0.001711\mathbf{T}^2 - 0.002297\mathbf{S}^2$ $+ 0.001812\mathbf{TB} - 0.002964\mathbf{SB} + 0.001616\mathbf{ST}$
	$R^2 = 0.7551$ Lack of Fit (P>F) = 0.6585
FlexCD	$= -60.96 + 0.3637\mathbf{B} + 0.4773\mathbf{T} + 0.1825\mathbf{S}$ $- 0.001707\mathbf{B}^2 - 0.001224\mathbf{T}^2 - 0.000554\mathbf{S}^2$ $+ 0.000492\mathbf{TB} - 0.00160\mathbf{SB} + 0.000598\mathbf{ST}$
	$R^2 = 0.6778$ Lack of Fit (P>F) = 0.8795
YstrsMD	$= -64.63 + 0.5207\mathbf{B} + 0.4984\mathbf{T} + 0.3571\mathbf{S}$ $- 0.003022\mathbf{B}^2 - 0.001123\mathbf{T}^2 - 0.001092\mathbf{S}^2$ $- 0.000468\mathbf{TB} - 0.002497\mathbf{SB} + 0.000210\mathbf{ST}$
	$R^2 = 0.8422$ Lack of Fit (P>F) = 0.4504
YstrsCD	$= -19.16 + 0.2360\mathbf{B} + 0.1087\mathbf{T} + 0.09987\mathbf{S}$ $- 0.001216\mathbf{B}^2 - 0.000169\mathbf{T}^2 - 0.000307\mathbf{S}^2$ $- 0.000302\mathbf{TB} - 0.001109\mathbf{SB} + 0.000296\mathbf{ST}$
	$R^2 = 0.8026$ Lack of Fit (P>F) = 0.5864
ImodMD	$= -1.112 + 0.00903\mathbf{B} + 0.00878\mathbf{T} + 0.00227\mathbf{S}$ $- 0.00002999\mathbf{B}^2 - 0.00001989\mathbf{T}^2 - 0.00000881\mathbf{S}^2$ $- 0.00001748\mathbf{TB} - 0.0000319\mathbf{SB} + 0.00001256\mathbf{ST}$
	$R^2 = 0.7111$ Lack of Fit (P>F) = 0.8365
ImodCD	$= -0.07512 + 0.00187\mathbf{B} + 0.00000156\mathbf{T} + 0.000407\mathbf{S}$ $- 0.00000559\mathbf{B}^2 + 0.00000093\mathbf{T}^2 - 0.00000156\mathbf{S}^2$ $- 0.00000312\mathbf{TB} - 0.00000815\mathbf{SB} + 0.00000379\mathbf{ST}$
	$R^2 = 0.6582$ Lack of Fit (P>F) = 0.9638

further analysis, response surface techniques. They were well described models for physical characterization properties with their high R-squares and fitness based on the results of F-tests and, in certain cases, by a normality check.

RS of the Regression Model

The results from the response surface analysis included the critical points of the independent variables and the predicted values of the dependent variables at the stationary points, and the shapes of the stationary points. Table 13 shows that the stationary points were not in or near the experimental regions. Unfortunately, most optimal conditions were not in the experimental region. Three-dimensional contour plots could not be obtained from the 3-way factorial design, which are very useful to estimate the optimal conditions in the experimental region. This makes it very difficult to estimate the maxi- and mini- points. The factorial design was too limited to determine the optimal conditions unless the optimal conditions (critical values) are in that region.

However, several predictions could be made from the maxi- and mini- stationary points. Thickness would be the minimum point for a blend ratio of 53 (cotton content) at the bonding temperature of 267 °C with the solvent treatment time of 88 minutes. Higher bonding temperature above 190 °C normally reduce the thickness values further, but higher cotton content and higher solvent treatment time will be ineffective in doing so. The bending length in MD would reach the maximum point with 78% cotton, 218°C in temperature and 96 minute solvent treatment time. Thus, only a higher bonding temperature up to 218°C would increase bending length values. Again, the cotton content and the solvent treatment time had reached the optimal conditions of bending length in MD in the experimental region.

Table 13. Results of Response Surface.

Dependent Variable	**Critical Value**			**Predicted Value**	**Stationary Point**
	Blend	**Temp**	**Soltime**		
Basiswt	67.6	195.4	127	166.8	saddle
Thick	53	266.6	88	0.723	minimum*
Airper	-493	-2623	1981	120.5	saddle
BendMD	78	217.9	95.7	7.44	maximum*
BendCD	73	202.1	104.2	5.13	saddle*
SstrsMD	-70	339.6	274.4	40.5	maximum
SstrsCD	207.9	-49.7	-228	-9.28	saddle
ElongMD	-3.33	198.3	95.4	21.5	saddle
ElongCD	848	-178	-501	-112	saddle
StrthMD	-44.9	305.5	252.3	151.9	maximum
StrthCD	225	-94.1	-317.9	-53.5	saddle
Flexall	77.7	269.7	168.8	48.4	maximum
FlexMD	93	345.1	169.3	98.2	maximum
FlexCD	-0.0016	270.8	310.7	32.0	maximum
YstrsMD	-19.3	245.6	209.2	28.9	maximum
YstrsCD	195.8	-34.7	-207.3	-8.3	saddle
ImodMD	172.1	112.6	-102.4	0.043	saddle
ImodCD	112.4	149.7	18.6	0.034	saddle

The results from strength in MD, specific stress in MD, all flexural rigidities and yield stress in MD showed that maximum values could be reached by higher bonding temperatures and/or longer solvent treatment times. To simplify the prediction of the optimal processing conditions, an analysis by blend ratios would be appropriate.

CORRELATIONS

From the above regression models, the data points (N = 3171) obtained and generated from the response surfaces were compared for relations to each other. Although the regression models for elongations in MD and in CD were not fitted, the data points were appropriate for the correlation analysis because of the high values in R-square (0.6563 and 0.8073 for MD and CD, respectively). The Pearson correlation coefficients (ρ : Rho) were tested for significance by t-test with H_o: Rho = 0. All physical properties indicated that their Pearson correlation coefficients were not zero. Therefore, all are significant. Table 14 lists the correlation coefficient values and also qualitative results of their relations based on indicatiors of strong correlation (S: $\rho > 0.7$), moderate correlation (M: $0.3 < \rho < 0.7$) and unrelated (U: $\rho < 0.3$). The signs of the coefficient indicate the positive or negative relationship.

The correlation tests are analyses of variables whether or not the linear relations exist. The lower values (< 0.3) imply no linear relation, but they do not guarantee that the variables are not related. The physical properties were tested for their linear relationships among each other and between machine and cross directions, since they could be useful in predicting physical properties based on their correlations. This is also the possibility of eliminating the number of physical testings needed to evaluate properties of nonwovens.

Table 14. Results of Correlations.

	Basiswt	Thick	Airper	BendMD	BendCD	SstrsMD
Basiswt		S (-)	S (-)	S (+)	S (+)	M (+)
Thick	-0.8281		S (+)	S (-)	S (-)	S (-)
Airper	-0.9098	0.9258		S (-)	S (-)	S (-)
BendMD	0.9777	-0.8708	-0.9296		S (+)	M (+)
BendCD	0.9845	-0.8695	-0.9403	0.9958		M (+)
SstrsMD	0.6436	-0.9286	-0.7838	0.6681	0.6688	
SstrsCD	0.6545	-0.9226	-0.7807	0.6745	0.6817	0.9122
ElongMD	0.1378	-0.5931	-0.3362	0.1637	0.1486	0.8060
ElongCD	0.1896	-0.6456	-0.4284	0.2143	0.2110	0.8592
StrthMD	0.6604	-0.9334	-0.7908	0.6825	0.6840	0.9995
StrthCD	0.6661	-0.9232	-0.7819	0.6837	0.6919	0.9887
Flexall	0.9740	-0.8489	-0.8893	0.9866	0.9855	0.6583
FlexMD	0.9698	-0.8481	-0.8868	0.9881	0.9834	0.6521
FlexCD	0.9765	-0.8469	-0.8904	0.9826	0.9859	0.6621
YstrsMD	0.6458	-0.9285	-0.7821	0.6688	0.6701	0.9997
YstrsCD	0.6489	-0.9208	-0.7776	0.6697	0.6772	0.9917
ImodMD	0.8836	-0.9586	-0.9230	0.9070	0.9145	0.8879
ImodCD	0.9190	-0.9236	-0.9128	0.9249	0.9348	0.8372

Table 14. (continued)

	SstrsCD	**ElongMD**	**ElongCD**	**StrthMD**	**StrthCD**	**Flexall**
Basiswt	M (+)	U (+)	U (+)	M (+)	M (+)	S (+)
Thick	S (-)	M (-)	M (-)	S (-)	S (-)	S (-)
Airper	S (-)	M (-)	M (-)	S (-)	S (-)	S (-)
BendMD	M (+)	U (+)	U (+)	M (+)	M (+)	S (+)
BendCD	M (+)	U (+)	U (+)	M (+)	M (+)	S (+)
SstrsMD	S (+)	S (+)	S (+)	S (+)	S (+)	M (+)
SstrsCD		S (+)	S (+)	S (+)	S (+)	M (+)
ElongMD	0.7696		S (+)	S (+)	S (+)	U (+)
ElongCD	0.8254	0.9664		S (+)	S (+)	U (+)
StrthMD	0.9940	0.7937	0.8461		S (+)	M (+)
StrthCD	0.9995	0.7568	0.8110	0.9916		M (+)
Flexall	0.6803	0.1562	0.1889	0.6758	0.6932	
FlexMD	0.6708	0.1576	0.1843	0.6692	0.6832	0.9990
FlexCD	0.6884	0.1508	0.1906	0.6801	0.7020	0.9985
YstrsMD	0.9912	0.8043	0.8578	0.9996	0.9889	0.6593
YstrsCD	0.9999	0.7714	0.8276	0.9934	0.9993	0.6756
ImodMD	0.9086	0.4585	0.5329	0.8979	0.9147	0.9115
ImodCD	0.8650	0.3789	0.4470	0.8499	0.8740	0.9367

Table 14. (continued)

	FlexMD	**FlexCD**	**YstrsMD**	**YstrsCD**	**ImodMD**	**ImodCD**
Basiswt	S (+)	S (+)	M (+)	M (+)	S (+)	S (+)
Thick	S (-)	S (-)	S (-)	S (-)	S (-)	S (-)
Airper	S (-)	S (-)	S (-)	S (-)	S (-)	S (-)
BendMD	S (+)	S (+)	M (+)	M (+)	S (+)	S (+)
BendCD	S (+)	S (+)	M (+)	M (+)	S (+)	S (+)
SstrsMD	M (+)	M (+)	S (+)	S (+)	S (+)	S (+)
SstrsCD	M (+)	M (+)	S (+)	S (+)	S (+)	S (+)
ElongMD	U (+)	U (+)	S (+)	S (+)	M (+)	M (+)
ElongCD	U (+)	U (+)	S (+)	S (+)	M (+)	M (+)
StrthMD	M (+)	M (+)	S (+)	S (+)	S (+)	S (+)
StrthCD	M (+)	M (+)	S (+)	S (+)	S (+)	S (+)
Flexall	S (+)	S (+)	M (+)	M (+)	S (+)	S (+)
FlexMD		S (+)	M (+)	M (+)	S (+)	S (+)
FlexCD	0.9952		M (+)	M (+)	S (+)	S (+)
YstrsMD	0.6527	0.6636		S (+)	S (+)	S (+)
YstrsCD	0.6660	0.6839	0.9915		S (+)	S (+)
ImodMD	0.9039	0.9180	0.8879	0.9064		S (+)
ImodCD	0.9275	0.9456	0.8377	0.8622	0.9895	

Overall, correlation coefficient values among most physical properties resulted in strong linear relationships. The results of the coefficient values and the qualitative categories are presented in Table 15 .

Correlations between MD and CD

All physical properties in MD gave similar correlation coefficients and qualitative correlations as in CD. The correlation coefficients between the properties in MD and in CD were very high, close to 1 and all positive. The correlation coefficients of machine and cross directions in bending length, specific stress, elongation, strength, flexural rigidity, yield stress and initial modulus were +0.9958, +0.9122, +0.9664, +0.9952, +0.9915 and +0.9895, respectively. Therefore, the properties of nonwovens in one direction were linearly proportional to those in the other direction.

Correlations among Physical Properties

The results of correlations among the physical properties is summarized in Table 15. Because of strong linear relations between machine and cross directions, a simple table could be produced.

Among physical properties, basis weight, bending length and flexural rigidity (overall, MD or CD) resulted in linear relations with each other. Either of the three properties could represent one of the other because of their strong linear relations as well as their same correlation trends with other properties. The properties were strongly correlated to thickness (inversely), air permeability (inversely) and initial modulus, and also moderately correlated to specific stress, strength and yield stress.

Table 15. Qualitative Linear Correlations Among Physical Properties.

	Strong	**Moderate**	**Unrelated**
Basis Weight **Bending Length** **Flexural Rigidity**	Basis Weight (+) Thickness (-) Air Permeability (-) Bending Length (+) Flexural rigidity (+) Initial Modulus (+)	Specific Stress (+) Strength (+) Yield Stress (+)	Elongation (+)
Thickness **Air permeability**	Basis Weight (-) thickness (+) Air permeability (+) Bending Length (-) Specific Stress (-) Strength (-) Flexural Rigidity (-) Yield Stress (-) Initial Modulus (-)	Elongation (-)	
Initial Modulus	Basis Weight (+) Thickness (-) Air Permeability (-) Bending Length (+) Specific Stress (+) Strength (+) Flexural Rigidity (+) Yield Stress (+) Initial Modulus (+)	Elongation (+)	
Specific Stress **Strength** **Yield Stress**	Thickness (-) Air Permeability (-) Specific Stress (+) Strength (+) Elongation (+) Yield Stress (+) Initial Modulus (+)	Basis Weight (+) Bending Length (+) Flexural Rigidity (+)	
Elongation	Specific Stress (+) Strength (+) Yield Stress (+)	Thickness (-) Air Permeability (-) Initial Modulus (+)	Basis Weight (+) Bending Length (+) Flexural Rigidity (+)

However, it was difficult to say that there was linear relationships with elongation. Therefore, the high basis weight, bending length or flexural rigidity would be linearly predicted for lower values in thickness and/or air permeability, and/or for a larger initial modulus.

Thickness, air permeability and initial modulus seemed to be important properties characterizing the nonwovens, since they showed strong linear relation with all physical properties except elongation. Among them, the strong correlations indicated that the thinner bonded webs were least permeable (lower value in air permeability), but had higher values in initial modulus. In addition, one of the above properties would lead to estimate the higher values in specific stress, bending length, strength, flexural rigidity and yield stress.

Specific stress, strength and yield stress were strongly related to each other and with other physical properties except basis weight, bending length and flexural rigidity. The thinner, less permeable nonwovens with high elongation and initial modulus values would project into the high specific stress, strength or yield stress values

Elongation was strongly correlated with specific stress, strength and yield stress, but showed no relationship with basis weight, bending length and flexural rigidity. Therefore, the stronger nonwovens had higher in elongation values. Elongations are not predictable by other properties.

Overall, the physical properties had strong linear relations with most of the other physical properties, which suggests that the number of different kinds of physical tests and separate testing for MD and CD may not be necessary to characterize the nonwovens. Another noticeable results were the positive and strong relations among basis weight, bending length and flexural rigidity, and among specific stress and strength. Bending length as linearly increased with basis weight, in this study, influences the flexural rigidity as calculated from basis weight and bending length. The same result was obtained with specific stress to strength, although specific stress is a normalized strength by basis weight.

Correlations by Blend Ratio

Since complex interactions from ANOVA resulted with blend ratios, the correlation analysis was also carried out by blend ratio to establish relations among the physical properties from changes in processing conditions, bonding temperature and solvent pretreatment time. For each blend ratio, 1057 data points generated from the total regression model were tested for correlation. The results indicated that all physical properties were strongly and linearly related each other. For the blend ratio of 50/50, all of the correlation coefficients were above 0.8 (negative values for thickness and air permeability). For the blend ratio of 75/25, the results were the same as those of 50/50. Also, for the blend ratio of 85/15, the properties were strongly related each other with the coefficients above 0.7.

Therefore, in a given blend ratio, all physical properties were strongly correlated with each other (all positively except for thickness and air permeability). Thus one of the physical values could be tested and the trend of the other values predicted.

RESPONSE SURFACE BY BLEND RATIO

Regression Models by Blend Ratio

Most regression models of the physical properties by blend ratio fitted very well with 2-nd order quadratic polynomial equations. The equations, with R-squares, and the results of the lack of fit are summarized in Tables 16, 17 and 18.

For the blend ratio of 50/50, the regression models of all physical properties except for elongation in MD (p<F : 0.0014) and in CD (p<F : 0.0366) were fitted quite well. The R-square values were in the range of 0.4817 to 0.8760. For the blend ratio of 75/25, the regression models of the physical properties resulted in high R-square values ranging from 0.6611 to 0.8960, and they all fitted well except for elongation in CD (p<F : 0.0458). For the blend ratio of 85/15, the regression models of all physical properties were fitted, but the R-square values covered a wide range. Most of the equations for the physical properties had R-squares above 0.5, except for basis weight, air permeability and elongation in MD and in CD. These R-square values were 0.2240, 0.1507, 0.3423 and 0.3655m respectively.

The models were suitable not only for further response surface analysis due to their high R-square values and/or their high p-values from F-test, but also in estimating the physical properties from processing conditions, bonding temperatures, and solvent pretreatment times. Simpler models can be obtained from further elimination techniques. The unfitted equations, elongation models, may require higher order polynomial models.

Table 16. Regression Models for 50/50 Blend Ratio.

Dependent Variable	**Regression Model**
Basiswt	$= - 168.82 + 3.5256T + 0.7682S$ $- 0.00963T^2 - 0.00119S^2 - 0.00293ST$
	$R^2 = 0.4817$ Lack of Fit (*P*>F) = 0.9723
Thick	$= 0.3121 + 0.00974T - 0.00624S$ $- 0.0000355T^2 + 0.0000186S^2 + 0.0000166ST$
	$R^2 = 0.7418$ Lack of Fit (*P*>F) = 0.4800
Airper	$= 58.41 - 0.0222T - 0.7108S$ $- 0.00082T^2 + 0.00155S^2 + 0.00242ST$
	$R^2 = 0.7004$ Lack of Fit (*P*>F) = 0.9972
BendMD	$= - 7.605 + 0.09892T + 0.0964S$ $- 0.000184T^2 - 0.000257S^2 - 0.000255ST$
	$R^2 = 0.8725$ Lack of Fit (*P*>F) = 0.6129
BendCD	$= 2.3825 - 0.02189T + 0.0638S$ $+ 0.000142T^2 - 0.000137S^2 - 0.000182ST$
	$R^2 = 0.8326$ Lack of Fit (*P*>F) = 0.9681
SstrsMD	$= - 21.94 + 0.1476T + 0.36996S$ $- 0.0000656T^2 - 0.00154S^2 - 0.00029ST$
	$R^2 = 0.8736$ Lack of Fit (P>F) = 0.7206
SstrsCD	$= - 17.589 + 0.1653T + 0.1079S$ $- 0.000341T^2 - 0.000348S^2 - 0.000054ST$
	$R^2 = 0.8753$ Lack of Fit (*P*>F) = 0.9390
ElongMD	$= 22.67 - 0.2726T + 0.6008S$ $+ 0.00102T^2 - 0.00156S^2 - 0.00229ST$
	$R^2 = 0.5838$ Lack of Fit (*P*>F) = 0.0014**
ElongCD	$= - 82.37 + 0.8944T + 0.6533S$ $- 0.002154T^2 - 0.002406S^2 - 0.00153ST$
	$R^2 = 0.8036$ Lack of Fit (*P*>F) = 0.0366**

Table 16. (continued)

Dependent Variable	**Regression Model**
StrthMD	$= -212.19 + 1.996T + 1.657S - 0.0042T^2 - 0.00617S^2 - 0.001975ST$
	$R^2 = 0.8392$ Lack of Fit (P>F) = 0.8582
StrthCD	$= -109.004 + 1.0915T + 0.4997S - 0.00255T^2 - 0.0013S^2 - 0.000587ST$
	$R^2 = 0.8368$ Lack of Fit (P>F) = 0.9814
Flexall	$= -34.233 + 0.1925T + 0.5848S + 0.00021T^2 - 0.00124S^2 - 0.00134T$
	$R^2 = 0.7497$ Lack of Fit (P>F) = 0.9198
FlexMD	$= -167.496 + 1.6176T + 1.024S - 0.00344T^2 - 0.00248S^2 - 0.00228ST$
	$R^2 = 0.7577$ Lack of Fit (P>F) = 0.6085
FlexCD	$= 28.862 - 0.4647T + 0.3278S + 0.00182T^2 - 0.000541S^2 - 0.000778ST$
	$R^2 = 0.7119$ Lack of Fit (P>F) = 0.9795
YstrsMD	$= -35.149 + 0.3027T + 0.3945S - 0.000516T^2 - 0.00147S^2 - 0.000493ST$
	$R^2 = 0.8760$ Lack of Fit (P>F) = 0.8005
YstrsCD	$= -17.745 + 0.1663T + 0.1086S - 0.000342T^2 - 0.000348S^2 - 0.0000591ST$
	$R^2 = 0.8736$ Lack of Fit (P>F) = 0.9380
ImodMD	$= -0.20897 + 0.00163T + 0.00121S - 0.00000145T^2 - 0.0000114S^2 + 0.00001091ST$
	$R^2 = 0.6941$ Lack of Fit (P>F) = 0.6928
ImodCD	$= 0.09154 - 0.00124T + 0.00024S + 0.0000043T^2 - 0.00000131S^2 + 0.00000214ST$
	$R^2 = 0.6686$ Lack of Fit (P>F) = 0.8451

Table 17. Regression Models for 75/25 Blend Ratio.

Dependent Variable	**Regression Model**
Basiswt	$= 311.725 - 1.9535T + 0.09276S$ $+ 0.00599T^2 - 0.000842S^2 + 0.000646ST$
	$R^2 = 0.6611$ Lack of Fit (P>F) = 0.8629
Thick	$= 1.1704 - 0.002217T - 0.001768S$ $+ 0.00000369T^2 + 0.0000145S^2 - 0.00000466ST$
	$R^2 = 0.8120$ Lack of Fit (P>F) = 0.9653
Airper	$= 55.138 - 0.1387T - 0.2719S$ $+ 0.0000166T^2 + 0.001294S^2 + 0.000287ST$
	$R^2 = 0.8509$ Lack of Fit (P>F) = 0.0662
BendMD	$= - 3.4734 + 0.08611T + 0.00818S$ $- 0.00022T^2 - 0.00025S^2 + 0.00023ST$
	$R^2 = 0.8853$ Lack of Fit (P>F) = 0.3724
BendCD	$= -10.875 + 0.1589T + 0.00912S$ $- 0.00044T^2 - 0.000161S^2 + 0.000137ST$
	$R^2 = 0.8303$ Lack of Fit (P>F) = 0.7745
SstrsMD	$= - 22.634 + 0.2108T + 0.06926S$ $- 0.000359T^2 - 0.0012S^2 + 0.000928ST$
	$R^2 = 0.8960$ Lack of Fit (P>F) = 0.4626
SstrsCD	$= 23.092 - 0.2912T - 0.05151S$ $+ 0.00094T^2 - 0.000401S^2 + 0.000778ST$
	$R^2 = 0.8635$ Lack of Fit (P>F) = 0.8654
ElongMD	$= 117.401 - 1.485T + 0.08428S$ $+ 0.00478T^2 - 0.00103S^2 + 0.000483ST$
	$R^2 = 0.7258$ Lack of Fit (P>F) = 0.0538
ElongCD	$= 45.378 - 0.5258T + 0.09653S$ $+ 0.001732T^2 - 0.001375S^2 + 0.000644ST$
	$R^2 = 0.6875$ Lack of Fit (P>F) = 0.0458**

Table 17. (continued)

Dependent Variable	**Regression Model**
StrthMD	$= - 21.61 + 0.00339T + 0.19725S$ $+ 0.001095T^2 - 0.004813S^2 + 0.004375ST$
	$R^2 = 0.8917$ Lack of Fit (P>F) = 0.5697
StrthCD	$= 130.964 - 1.6259T - 0.2652S$ $+ 0.005126T^2 - 0.001606S^2 + 0.00351ST$
	$R^2 = 0.8634$ Lack of Fit (P>F) = 0.9185
Flexall	$= - 71.8678 + 0.8903T - 0.2831S$ $- 0.002377T^2 - 0.0016S^2 + 0.003947T$
	$R^2 = 0.8362$ Lack of Fit (P>F) = 0.9166
FlexMD	$= - 9.1408 + 0.1338T - 0.3548S$ $+ 0.000148T^2 - 0.002996S^2 + 0.005996ST$
	$R^2 = 0.8564$ Lack of Fit (P>F) = 0.7739
FlexCD	$= -78.4319 + 0.96935T - 0.21227S$ $- 0.002762T^2 - 0.000846S^2 + 0.002597ST$
	$R^2 = 0.7714$ Lack of Fit (P>F) = 0.9079
YstrsMD	$= - 23.7333 + 0.22864T + 0.06581S$ $- 0.000427T^2 - 0.001176S^2 + 0.000928ST$
	$R^2 = 0.8889$ Lack of Fit (P>F) = 0.5593
YstrsCD	$= 23.2793 - 0.29261T - 0.05541S$ $+ 0.000942T^2 - 0.000399S^2 + 0.000798ST$
	$R^2 = 0.8656$ Lack of Fit (P>F) = 0.8680
ImodMD	$= - 0.34714 + 0.004427T - 0.001148S$ $- 0.0000116T^2 - 0.0000101S^2 + 0.0000208ST$
	$R^2 = 0.8532$ Lack of Fit (P>F) = 0.9062
ImodCD	$= 0.03605 - 0.000242T - 0.000586S$ $+ 0.000000768T^2 - 0.00000233S^2 + 0.0000068ST$
	$R^2 = 0.7248$ Lack of Fit (P>F) = 0.9511

Table 18. Regression Models for 85/15 Blend Ratio.

Dependent Variable	**Regression Model**
Basiswt	$= 203.8 - 0.4946T + 0.01664S$ $- 0.0014T^2 - 0.000188S^2 - 0.000367ST$
	$R^2 = 0.2240$* Lack of Fit (P>F) = 0.9862
Thick	$= 2.687 - 0.02033T - 0.001368S$ $+ 0.0000556T^2 + 0.00000499S^2 + 0.00000299ST$
	$R^2 = 0.7808$ Lack of Fit (P>F) = 0.8411
Airper	$= 61.31 - 0.4695T - 0.0879S$ $+ 0.001331T^2 + 0.000515S^2 + 0.00008197ST$
	$R^2 = 0.1507$* Lack of Fit (P>F) = 0.5038
BendMD	$= - 4.999 + 0.1164T + 0.0171S$ $- 0.000313T^2 - 0.000123S^2 + 0.000043ST$
	$R^2 = 0.6453$ Lack of Fit (P>F) = 0.4385
BendCD	$= - 8.982 + 0.1479T + 0.01304S$ $- 0.000409T^2 - 0.0000557S^2 - 0.000008ST$
	$R^2 = 0.5569$ Lack of Fit (P>F) = 0.0066**
SstrsMD	$= - 73.53 + 0.8844T + 0.1104S$ $- 0.002468T^2 - 0.000725S^2 + 0.0000839ST$
	$R^2 = 0.6070$ Lack of Fit (P>F) = 0.2727
SstrsCD	$= - 38.68 + 0.4661T + 0.0299S$ $- 0.001329T^2 - 0.000245S^2 + 0.000082ST$
	$R^2 = 0.5193$ Lack of Fit (P>F) = 0.1681
ElongMD	$= 89.13 - 1.0467T + 0.09999S$ $+ 0.00318T^2 - 0.000413S^2 - 0.000115ST$
	$R^2 = 0.3423$ Lack of Fit (P>F) = 0.8636
ElongCD	$= - 43.69 + 0.5759T + 0.1385S$ $- 0.00163T^2 - 0.000669S^2 - 0.000224ST$
	$R^2 = 0.3665$ Lack of Fit (P>F) = 0.2889

Table 18. (continued)

Dependent Variable	**Regression Model**
StrthMD	$= - 293.19 + 3.5296T + 0.4448S$ $- 0.00984T^2 - 0.00297S^2 + 0.000462ST$
	$R^2 = 0.6167$ Lack of Fit (P>F) = 0.1986
StrthCD	$= - 165.92 + 2.003T + 0.0985S$ $- 0.00573T^2 - 0.000951S^2 + 0.000461ST$
	$R^2 = 0.5214$ Lack of Fit (P>F) = 0.0837
Flexall	$= - 140.16 + 1.77T + 0.1333S$ $- 0.00487T^2 - 0.00102S^2 + 0.000567T$
	$R^2 = 0.5671$ Lack of Fit (P>F) = 0.3332
FlexMD	$= - 121.57 + 1.6043T + 0.211S$ $- 0.00419T^2 - 0.00199S^2 + 0.00135ST$
	$R^2 = 0.5739$ Lack of Fit (P>F) = 0.7268
FlexCD	$= - 119.84 + 1.4983T + 0.07158S$ $- 0.00421T^2 - 0.000455S^2 + 0.000219ST$
	$R^2 = 0.4948$ Lack of Fit (P>F) = 0.0530
YstrsMD	$= - 86.02 + 1.031T + 0.1075S$ $- 0.00289T^2 - 0.000692S^2 + 0.0000695ST$
	$R^2 = 0.6042$ Lack of Fit (P>F) = 0.1929
YstrsCD	$= - 38.21 + 0.4608T + 0.02719S$ $- 0.001314T^2 - 0.000242S^2 + 0.00009333ST$
	$R^2 = 0.5159$ Lack of Fit (P>F) = 0.1192
ImodMD	$= - 1.574 + 0.0194T + 0.000245S$ $- 0.0000549T^2 - 0.00000525S^2 + 0.00000465ST$
	$R^2 = 0.5910$ Lack of Fit (P>F) = 0.5549
ImodCD	$= - 0.08658 + 0.001377T - 0.000191S$ $- 0.00000405T^2 - 0.00000132S^2 + 0.00000283ST$
	$R^2 = 0.5352$ Lack of Fit (P>F) = 0.1227

Response Surface by Blend Ratio

Response surfaces of the physical properties were generated for each blend ratio. The critical values, predicted values and stationary points were obtained from canonical analysis of the response surfaces. Some of the critical points with maxi- and mini- stationary points were in the actual experimental region of bonding temperature (in the range of 150 to 190°C) and the solvent treatment times (in the range of 0 to 120 minutes). In the case of saddle points and critical points outside the experimental region, the optimal conditions were estimated from 3-D surface and contour plots based on the path of steep. The results for the three blend ratios are listed in Tables 19, 20 and 21.

Response Surface of 50/50

Basis weight and elongation in CD reached maximums in the experimental region. The optimal conditions were bonding temperatures of 165 and 119 °C, and solvent treatment times of 119 and 79 minutes, respectively. For other physical properties, the optimal conditions were estimated from the types of stationary points and the response surface plots. The estimated optimal processing conditions were for maximum physical properties, except for thickness and air permeability.

From the estimated optimal conditions and the shape of the response surface plots, suggestions can be made to improve each of the physical properties. There were several properties which could be enhanced by an increase in bonding temperatures and/or solvent treatment times. Specific stress in CD, strength in CD, overall flexural

Table 19. Results from Response Surface for 50/50 Blend Ratio.
(* estimated optimal condition, maximum in the experimental region)

Dependent Variable	**Critical Value**				**Optimal Condition**	
	TEMP	**SOLTIME**	**Predicted Value**	**Stationary Point**	**TEMP**	**SOLTIM E**
Basiswt	165	119	168	maximum	165	119
Thick	159	96.7	0.787	saddle	154* 190*	0* 95*(min)
Airper	151	112	17.1	saddle	150* 190*	0* 80*(min)
BendMD	211	82.7	6.83	maximum	190*	90*
BendCD	159	127	4.71	saddle	190*	120*
SstrsMD	1085	17.9	61.5	maximum	190*	90*
SstrsCD	231	137	8.93	maximum	190*	120*
ElongMD	186	67.9	17.7	saddle	150*	80*
ElongCD	180	78.7	23.7	maximum	180	78.7
StrthMD	214	100	84.0	maximum	190*	105*
StrthCD	197	148	35.3	maximum	190*	120*
Flexall	108	178	28.1	saddle	190*	120*
FlexMD	197	116	51.0	maximum	190*	120*
FlexCD	167	183	20.2	saddle	190*	120*
YstrsMD	249	92.4	20.8	maximum	190*	120*
YstrsCD	232	137	8.92	maximum	190*	120*
ImodMD	-947	-401	-1.22	saddle	190*	120*
ImodCD	101	174	0.05	saddle	190*	120*

Table 20. Results from Response Surface for 75/25 Blend Ratio.
(* estimated optimal condition, maximum in the experimental region)

Dependent Variable	**Critical Value**				**Optimal Condition**	
	TEMP	**SOLTIME**	**Predicted Value**	**Stationary Point**	**TEMP**	**SOLTIME**
Basiswt	157	115	164	saddle	190*	120*
Thick	376	121	0.646	minimum	150* 190*	0* 95*(min)
Airper	$1.1x10^{5}$	$-1.2x10^{4}$	$-7.7x10^{3}$	minimum	150* 190*	0* 80*(min)
BendMD	268	139	8.63	maximum	190*	105*
BendCD	198	113	5.40	maximum	190*	110*
SstrsMD	665	287	57.4	maximum	190*	105*
SstrsCD	129	61.4	2.66	saddle	190*	120*
ElongMD	152	76.4	8.01	saddle	190*	85*
ElongCD	139	67.7	12.1	saddle	190*	80*
StrthMD	-22.3	10.4	-20.6	saddle	190*	105*
StrthCD	136	66.6	11.8	saddle	190*	120*
Flexall	$-4.7x10^{3}$	$-5.9x10^{3}$	$-1.3x10^{3}$	saddle	190*	120*
FlexMD	35.1	-24.1	-2.52	saddle	190*	120*
FlexCD	401	179	64.9	maximum	190*	120*
YstrsMD	522	234	43.7	maximum	190*	100*
YstrsCD	130	60.3	2.63	saddle	190*	120*
ImodMD	$1.9x10^{3}$	$1.9x10^{3}$	2.77	maximum	190*	120*
ImodCD	95.8	13.9	0.0204	saddle	190*	120*

Table 21. Results from Response Surface for 85/15 Blend Ratio.
(* estimated optimal condition, maximum in the experimental region)

	Critical Value				**Optimal Condition**	
Dependent Variable	**TEMP**	**SOLTIME**	**Predicted Value**	**Stationary Point**	**TEMP**	**SOLTIME**
Basiswt	152	192	168	saddle	190*	120*
Thick	181	83.1	0.793	minimum	150* 181	0* 83(min)
Airper	174	71.5	17.3	minimum	150* 174	0* 72(min)
BendMD	193	104	7.12	maximum	190*	105*
BendCD	180	104	4.98	maximum	180	104
SstrsMD	181	86.6	11.1	maximum	181	86.6
SstrsCD	178	90.7	4.20	maximum	178	86.6
ElongMD	166	98	6.96	saddle	190*	95*
ElongCD	172	74.8	10.9	maximum	172	74.8
StrthMD	182	89.0	46.7	maximum	182	89.0
StrthCD	179	95.1	17.7	maximum	179	95.1
Flexall	189	118	34.5	maximum	189	118
FlexMD	212	125	61.4	maximum	190*	120*
FlexCD	181	122	20.4	maximum	180*	120*
YstrsMD	179	86.7	11.0	maximum	179	86.7
YstrsCD	179	90.4	4.16	maximum	179	86.7
ImodMD	181	103	0.194	maximum	181	103
ImodCD	231	175	0.0559	maximum	190*	120*

rigidity, flexural rigidity in CD, yield stress in MD and in CD, and initial modulus in MD still have room for improvement by harsher processing conditions

However, some physical properties have already reached their maximum points for solvent pretreatment, but could be improved by an increase in the bonding temperatures. These properties include bending length in MD and in CD, specific stress in MD, strength in MD, flexural rigidity in MD, initial modulus in CD, thickness (minimum), air permeability (minimum) and elongation in MD.

Besides basis weight and elongation in CD, most of the properties had their optimal conditions near the highest bonding temperatures (190°C) and near the longest solvent treatment times (120 minutes). These values, too, could be increased by harsher processing conditions.

Response Surface of 75/25

No physical properties reached their optimal conditions in the experimental region. All optimal conditions in the experimental region were estimated by critical values, stationary points and the response surface plots. Similar to those conditions in the analysis of the 50/50 blend ratio, some of the estimated optimal conditions were for the highest bonding temperature and the longest solvent treatment time. It seems that bonding temperature above 190°C and solvent treatment times exceeding 120 minutes may be needed for the properties to reach their maximum values.

Some properties that could be increased by the higher temperature and/or longer solvent treatment times include specific stress in CD, strength in CD, flexural rigidities, yield stresses in MD and in CD, initial modulus in MD and in CD. On the other hand, properties that already have reached their maximum solvent treatment effects, but could be improved by the higher bonding temperatures, include basis weight, thickness (minimum), air permeability (minimum), bending lengths in MD and in CD, specific stress in MD, strength in MD, and elongations in MD and in CD.

Like results from the blend ratio of 50/50, the physical properties of the nonwovens from the blend ratio of 75/25 cotton/CA fibers could be enhanced by higher bonding temperatures and/or longer solvent treatment times. This result is possibly due to the 25% cellulose acetate content, wherein more bonding adhesion to cotton fibers might be gained by harsher processing conditions of raised temperature and/or increased modification by a solvent.

Response Surface of 85/15

Unlike results from the blend ratios of 50/50 and 75/25, most of the physical properties reached their maximum points within the experimental region or, at least, near experimental region. Overall, the optimal conditions for most of the properties seemed to be a bonding temperature of around 180°C and a solvent treatment time of around 90 minutes. Those properties reaching their optimal values in the experimental region were thickness (minimum), air permeability (minimum), bending length in CD, specific stresses in MD and in CD, elongation in CD, strengths in MD and in CD, overall flexural rigidity, yield stresses in MD and in CD, and initial modulus in MD. The flexural rigidity in CD would reach its maximum value near experimental region.

The initial modulus of nonwovens from 85/15 cotton/CA blends could be improved by an increase in the bonding temperature and/or by an increase in the solvent treatment time. Only an increase in the bonding temperatures would improve some properties such as basis weight (maximum), bending length in MD, elongation in MD and flexural rigidity in MD.

Because of the small portion of cellulose acetate fibers in the nonwovens from 85/15 cotton/CA blends, a limitation exists for enhancing the bonding adhesion of cellulose acetate fibers to cotton fibers by raising bonding temperatures or increasing solvent pretreatment times.

Response Surface of Combination of Four Properties

The desirability functions were added for the four selected properties: air permeability, strength in MD, elongation in MD and overall flexural rigidity. The four properties were optimized simultaneously for strength, air permeability and flexibility with high elongation. Each data value of the four properties was multiplied by 1 for highest value except for the overall flexural rigidity while was multiplied by (-)1 for lowest value. Then the newly combined values (COMB4) were obtained by adding the four data points. New regression models, for the blend ratios, were generated from the values of COMBO4. The results are given in Table 22.

All models were fitted with quadratic equations ($p<0.05$). Except for the model of the blend ratio of 85/15 cotton/CA, the models of COMB4 gave high R-square values. Overall, the models were appropriate for further analysis and optimization by

Response Surface methods. The graphs from Response surface are shown in Figures 15, 16 and 17, and the optimal conditions are listed in Table 23.

Table 22. Regression Models for COMBO4.

Blend Ratio of Cotton/CA	**Regression Model for COMBO4**
50/50	$= -96.874 + 1.5088T + 0.9619S - 0.004219T^2 - 0.005676S^2 + 0.000225ST$
	$R^2 = 0.7808$ Lack of Fit (*P*>F) = 0.0953
75/25	$= 222.798 - 2.5406T + 0.2928S + 0.008263T^2 - 0.00295S^2 + 0.001198ST$
	$R^2 = 0.6738$ Lack of Fit (*P*>F) = 0.0562
85/15	$= -2.601 + 0.2435T + 0.3235S - 0.000459T^2 - 0.000138S^2 - 0.00185ST$
	$R^2 = 0.2229$ Lack of Fit (*P*>F) = 0.8432

Table 23. Results from Response Surface for COMBO4.

Blend Ratio of Cotton/CA	**Critical Value of COMBO4**				**Optimal Condition**	
	TEMP	**SOLTIME**	**Predicted Value**	**Stationary Point**	**TEMP**	**SOLTIME**
50/50	181	88	82.3	maximum	181	88
75/25	148	80	46.5 (67.5*)	saddle	190*	85*
85/15	253	78	40.9 (39*)	maximum	190*	80*

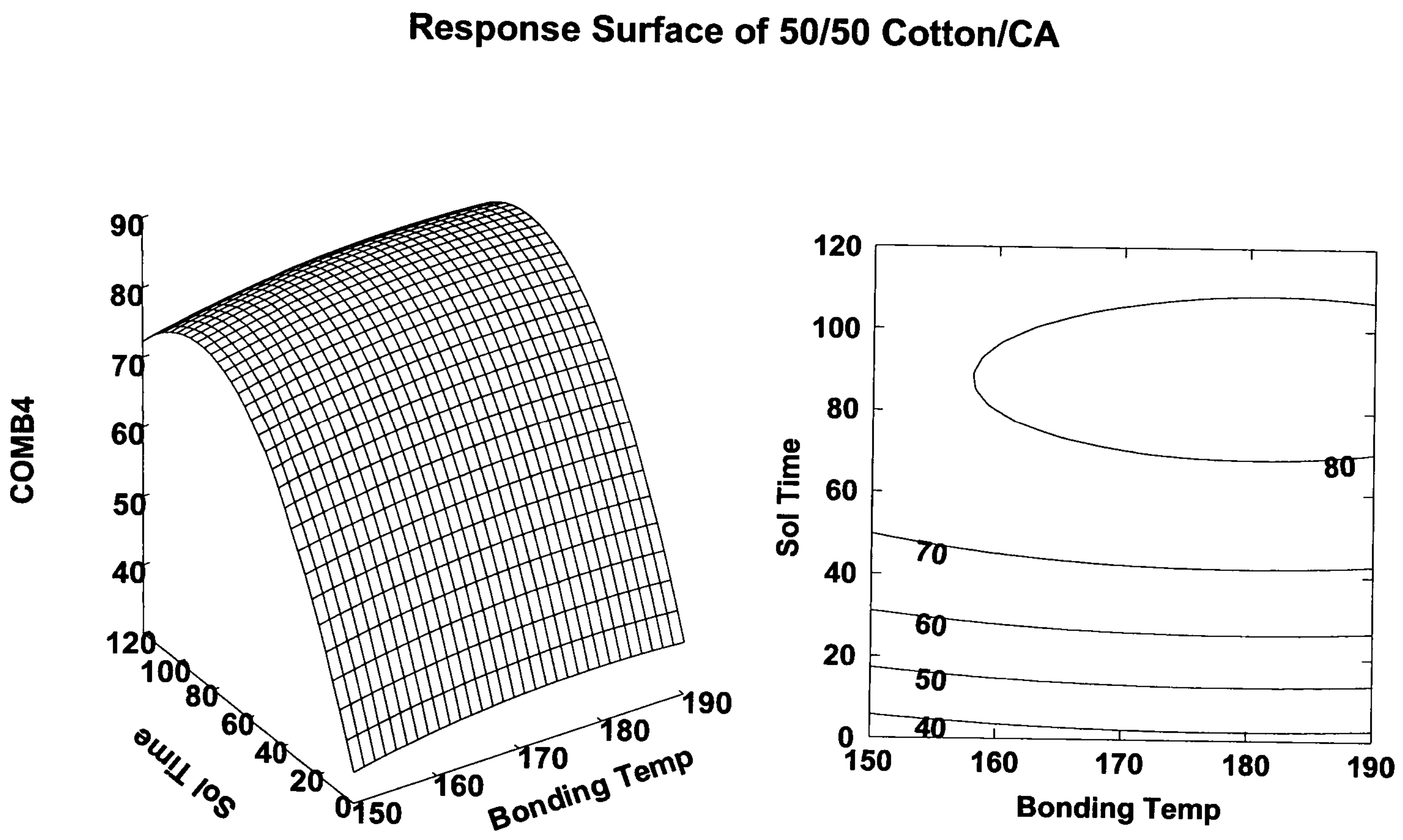

Fig. 15. 3-D and Contour Plots of COMB4 for 50/50 Cotton/CA.

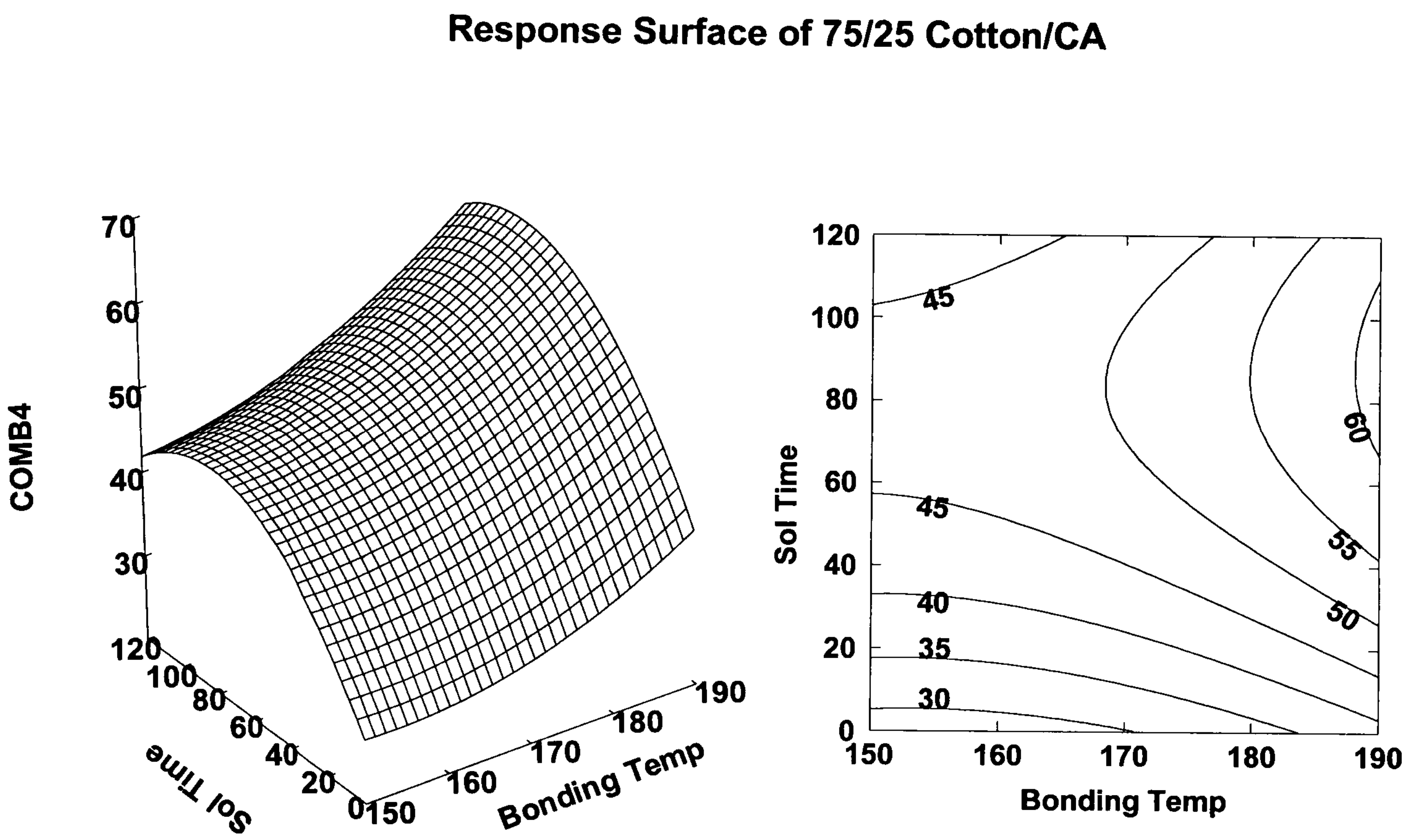

Fig. 16. 3-D and Contour Plots of COMB4 for 75/25 Cotton/CA.

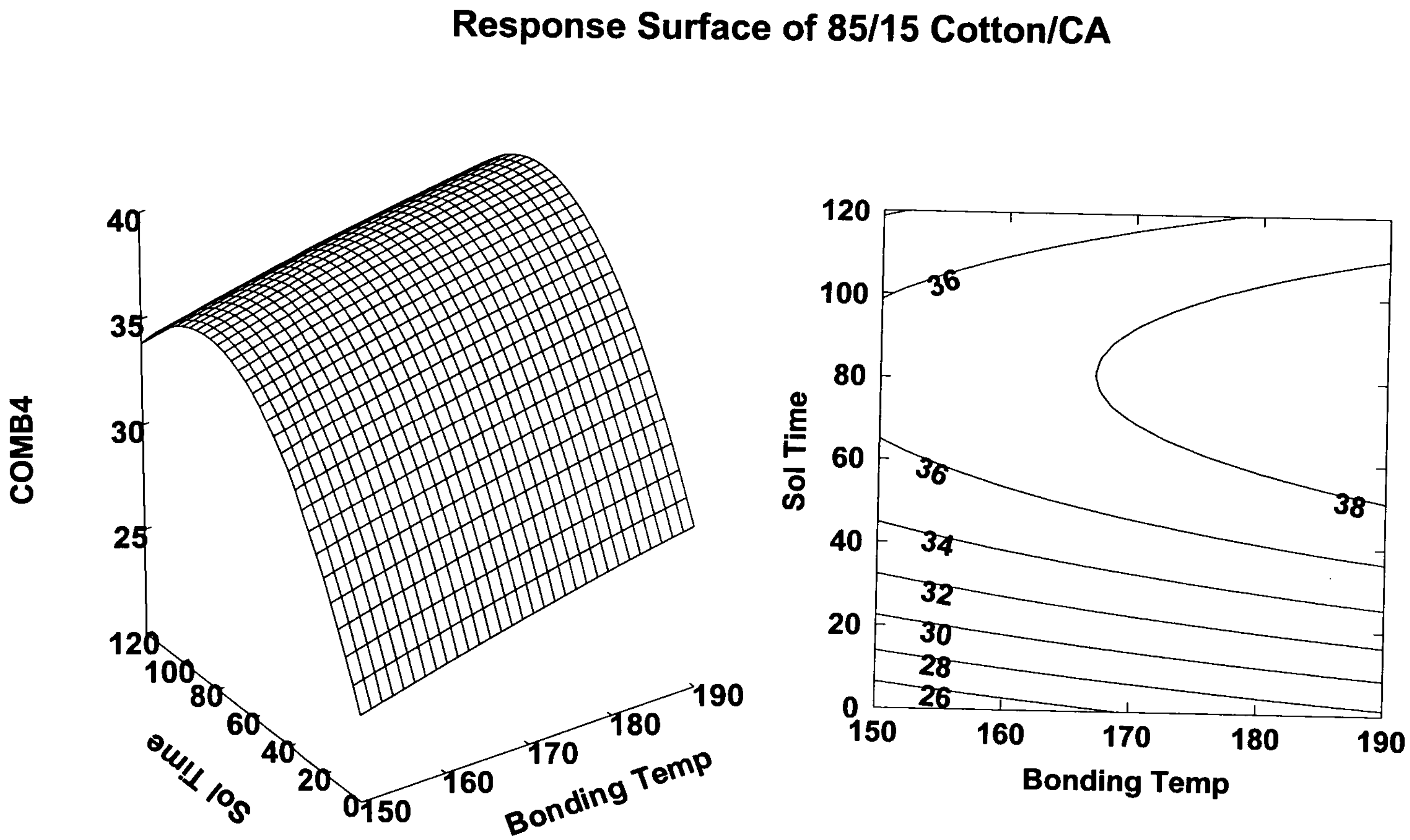

Fig. 17. 3-D and Contour Plots of COMB4 for 85/15 Cotton/CA.

Among the optimal conditions of the blend ratios, the critical value from the blend ratio of 50/50 was in the experimental region. The processing conditions for this were a the bonding temperature of 181°C and a the solvent pretreatment time of 88 minutes. Higher temperatures than 181 °C and greater time solvent pretreatment time than 88 minutes would produce nonwovens with greater strength and higher elongation values, but lower air permeability values and higher flexural rigidity values. The implication of this result is that cellulose acetate fibers probably contribute greater bonding adhesion with greater degree of the softening under hasher processing conditions. Therefore, maximum procession conditions selected in this study would not be necessary to produce stronger, breathable and flexible nonwovens from 50/50 cotton/CA with higher elongation. On the other hand, if strength and elongation (toughness) were the only desired properties, breathablility and flexibility could be traded off in 50/50 cotton/CA blends to obtain these two desired properties.

The stationary point of the 75/25 blend ratio was a saddle point, at which the critical values were a bonding temperature of 148°C and a solvent pretreatment time of 80 minutes. The estimated optimal conditions in the experimental region were a temperature of 190°C and a treatment time of 85 minutes. From the surface and contour plots, the COMBO4 value is increased with bonding temperatures above 150°C and with solvent treatment times above 80 minutes. For example, further increase in temperatures above 190°C would result in increases in COMBO4 values to give stronger, more breathable and more flexible nonwovens with higher elongation.

Therefore, nonwovens from 75/25 cotton/CA nonwovens did not reach their maximum levels in the experimental region, and a further increase in bonding temperatures would be necessary to improve the combination of the four properties of these nonwovens.

The critical values of the blend ratio of 85/15 cotton/CA nonwovens were reached at the bonding temperature of 253°C and the solvent treatment time of 78 minutes, at which the stationary point was maximum. The optimal processing conditions of COMB4 were not in the experimental region, even though the solvent treatment had reached its maximum at around 80 minutes. From the surface and contour plots, the optimal conditions in the experimental was estimated to occur at the bonding temperature of 190°C and the solvent treatment time of 80 minutes. The values of COMB4 would be increased at temperatures above 190°C up to 253°C, but not an the increase in solvent pretreatment times.

Regardless of the differences in the blend ratio, the COMB4 values had reached their maximum points by solvent pretreatment times in the range of 80 to 90 minutes. However, for blend ratios of less cellulose acetate content, 75/25 and 85/15, the values could be increased by raising calender-roll temperatures. From the plots, the notable results were the negligible effects of bonding temperatures in the region of low solvent pretreatment times below 60 minutes, for which the values were increased only by the solvent effects. For solvent treatment times above 60 minutes, the effects of bonding temperature and the solvent treatment time contributed additively to an increase the COMB4 values for all blend ratios.

In addition, the optimal values in the experimental region were 82, 68 and 39 for the highest to lowest contents of cellulose acetate in the blend. The maximum value of COMB4 of 85/15 would be 41 (at the optimal condition (254°C)), which is still half the value from 50/50 in the experimental region. Therefore, higher cellulose acetate content showed stronger, more breathable and more flexible nonwovens with higher elongation.

CHAPTER 5

SUMMARY AND CONCLUSIONS

The advantages of the choice of cellulose acetate as a binder fiber in thermally bonded nonwovens containing cotton as a base fiber are clearly reflected in terms of its biodegradability, enhanced degradation with cotton fibers and enhanced tensile strengths of the blend nonwovens by solvent modification. The benefits of using statistical methods are also demonstrated for characterization of the effects of processing conditions on the physical properties of the nonwovens from cotton/CA, and for optimization of the processing conditions in the solvent-assisted thermal calendering.

BIODEGRADABILITY

To determine a semi-quantitative measure of the biodegradation of cellulose acetate fibers, the standard test method of AATCC 30-1988: "Antifungal Activity, Assessment on Textile Materials: Mildew and Rot Resistance of Textile Materials" was selected. After a 12-week soil burial test, evidence of microbial attack on the CA fabric was obtained on the basis of 27% strength loss.

As a more reliable method for monitoring the aerobic activity of microorganisms, the ASTM test method D5209-91: "Standard Test Method for Determining the Aerobic Biodegradation of Plastic Materials in the Presence of Municipal Sewage Sludge" was adopted. From the aerobic sludge test, the amount of CO_2 evolved from the decomposition of cellulose acetate, cotton and the fibers in the blend was measured. The biodegradability of CA fibers, with a degree of substitution of 2.5, was confirmed by showing carbon dioxide evolution. In addition, the synergistic effects of multi-enzyme systems between cellulase and esterase were suggested based on the increased biodegradation rates in fiber blends.

Therefore, the biodegradability of cellulose acetate with a DS of 2.5 was confirmed through degradation by microbial attack. Strength loss and carbon dioxide evolution were the source of confirmation. The synergistic enzymatic effect of cotton/CA blend fibers provided a promising feature for environmentally friendly nonwovens of the fiber blends.

PROCESS CHARACTERIZATION

Opening, blending, carding and thermal calendering processes were used to fabricate the nonwovens from cotton/CA blends. With additional innovation toward optimizing the nonwoven fabric properties, the possibility of using acetone as a surface softening agent was investigated. Pretreatment with the solvent vapors was introduced to modify the softening temperatures of the CA and to lower the calendering temperatures. From the results of tensile tests, a remarkably enhanced strength was obtained at lower calendering temperatures with the solvent vapor pretreatment. This finding is extremely beneficial, both from an energy consumption standpoint and from the knowledge that cotton fiber becomes brittle and weak at temperatures above 200°C.

Various physical properties of nonwovens from the solvent-assisted thermal calendering, were also investigated at different bonding temperatures and with different solvent pretreatment times. The effects of the processing conditions and blend ratios were explored by various statistical methods. The experiment was designed for a 3-way factorial design (3x3x8) with the following factors: bonding temperatures, blend ratios and solvent treatment times. The effects of the 3 factors on a total number of 18 physical properties were determined by analysis of variance, least-square-means comparison, regression and correlation techniques. The results indicated that all the physical properties were significantly influenced by the bonding temperatures and the solvent treatment times. The summary of the results are as follows:

1. Most of the selected physical properties were significantly influenced by processing conditions and blend ratio. The solvent pretreatment times affected all the physical properties.

2. Most of the physical properties showed significant differences among the bonding temperatures, solvent pretreatment times and blend ratios.
3. All physical properties were well characterized by full second order polynomial regression models.
4. All physical properties in the machine direction were strongly correlated with those in the cross direction.

From multivariate and multiresponse optimization by response surface methodology, optimal bonding temperatures and solvent pretreatment times were identified and predicted for each physical property. With the desirability and distance functions of four physical properties, the optimal process and pretreatment conditions for strong, soft, breathable and extensible nonwovens were suggested. This statistical optimization technique is useful in understanding the two-way interactions of bonding temperature and solvent pretreatment times. It might well be useful in setting up a production line of a solvent-assisted thermal calendering process. The significance of this work lies in the employment and the adaptation of statistical techniques to determine the optimal processing conditions in a simple way.

IMPLICATIONS AND SUGGESTIONS

The biodegradability of cellulose acetate fibers was examined by an ASTM standard method. The method was designed for plastic materials such as films. From this study, the possibility of the use of this procedure as a test of textile fibers was confirmed. In this method, carbon dioxide evolution was monitored and the method appears appropriate as the most quantitative biodegradation test available to explore mineralization processes of textile materials. In addition, from the biodegradation results of cotton/CA fiber blends, synergistic enzymatic effects could be explored between all cellulosic materials including rayon, wood pulp and starch. Synergistic effects of different kinds of cellullase enzymes might be followed from the studies of different blended cellulosic materials.

Diversity is one of the unique characteristics of manufacturing nonwoven fabrics. In this study, a new way to produce nonwoven fabrics combining solvent and thermal bonding processes was introduced. The idea of solvent modification before thermal calendering can be applied to various thermoplastic binder fibers. A variety of solvents for these binder fibers could be employed to increase bonding adhesion and to improve physical properties of the resulting nonwoven fabrics.

The use of statistical methods for optimization procedures in thermal calendering is almost necessary and highly recommended. By using response surface methodology, multivariate and/or multiresponse experiments can be simultaneously optimized.

Most thermal calendering processes involve several processing parameters, and in most cases several physical properties are evaluated for suitable end-use applications. In this study, the use of distance and desirability functions, along with response surface methodology, was introduced. The technique can be useful in optimizing processing conditions in any production line and in optimizing physical properties of materials.

For future study, some microstructural analysis such as differential scanning calorimetry and wide-angle X-ray diffraction would be helpful in understanding bonding behavior of cellulose acetate to cotton fibers, and in examining the structural changes of cellulose acetate fibers by the solvent used in pretreatment.

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APPENDICES

APPENDICES

A-1. Frequency Tables for Fractional Factorial Design.

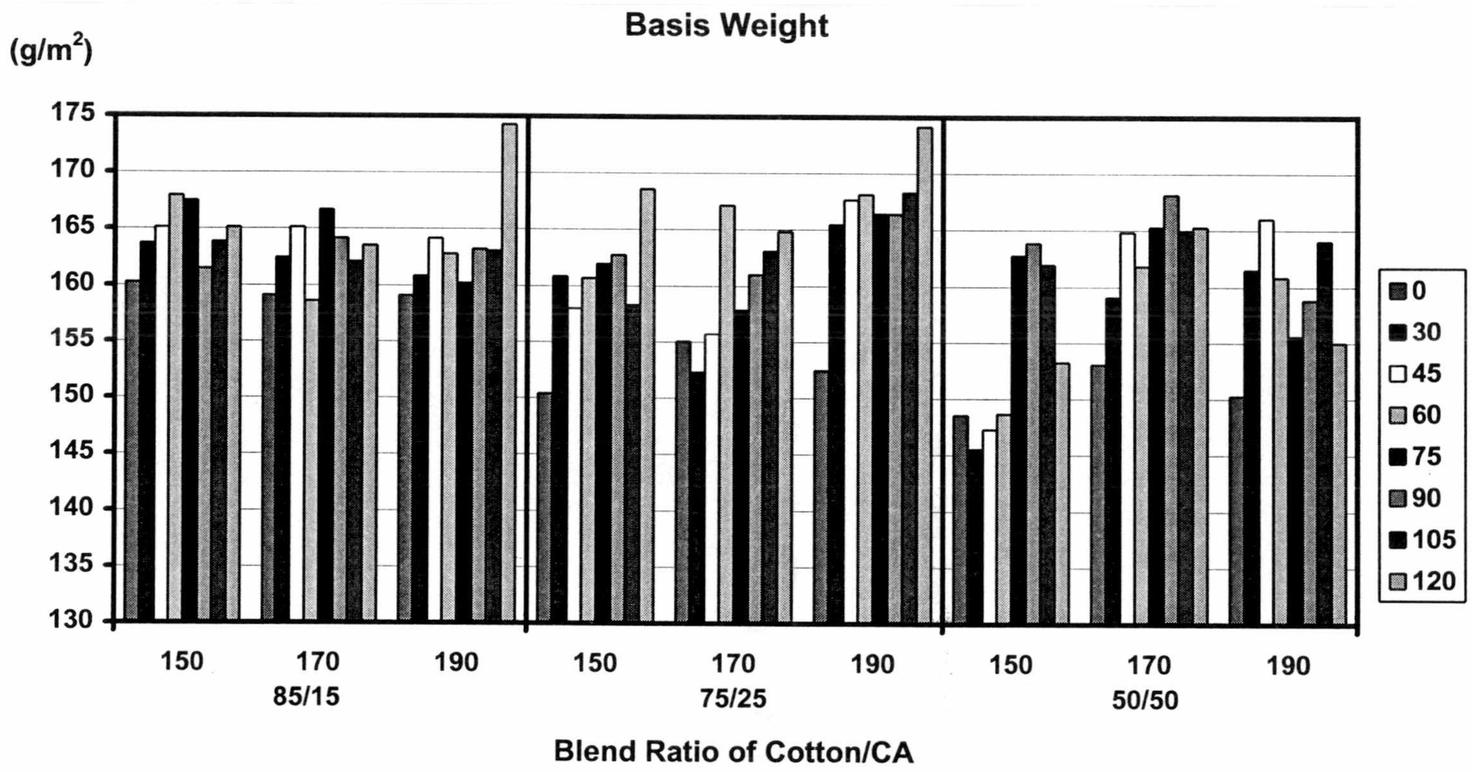
TIME		TEMP		
Frequency **Percent** **Row Percent** **Column Percent**	**150**	**170**	**190**	**Total**
0	1 4.17 33.33 12.50	1 4.17 33.33 12.50	1 4.17 33.33 12.50	3 12.50
30	1 4.17 33.33 12.50	1 4.17 33.33 12.50	1 4.17 33.33 12.50	3 12.50
45	1 4.17 33.33 12.50	1 4.17 33.33 12.50	1 4.17 33.33 12.50	3 12.50
60	1 4.17 33.33 12.50	1 4.17 33.33 12.50	1 4.17 33.33 12.50	3 12.50
75	1 4.17 33.33 12.50	1 4.17 33.33 12.50	1 4.17 33.33 12.50	3 12.50
90	1 4.17 33.33 12.50	1 4.17 33.33 12.50	1 4.17 33.33 12.50	3 12.50
105	1 4.17 33.33 12.50	1 4.17 33.33 12.50	1 4.17 33.33 12.50	3 12.50
120	1 4.17 33.33 12.50	1 4.17 33.33 12.50	1 4.17 33.33 12.50	3 12.50
Total	8	8	8	**24**

A-1. (continued)

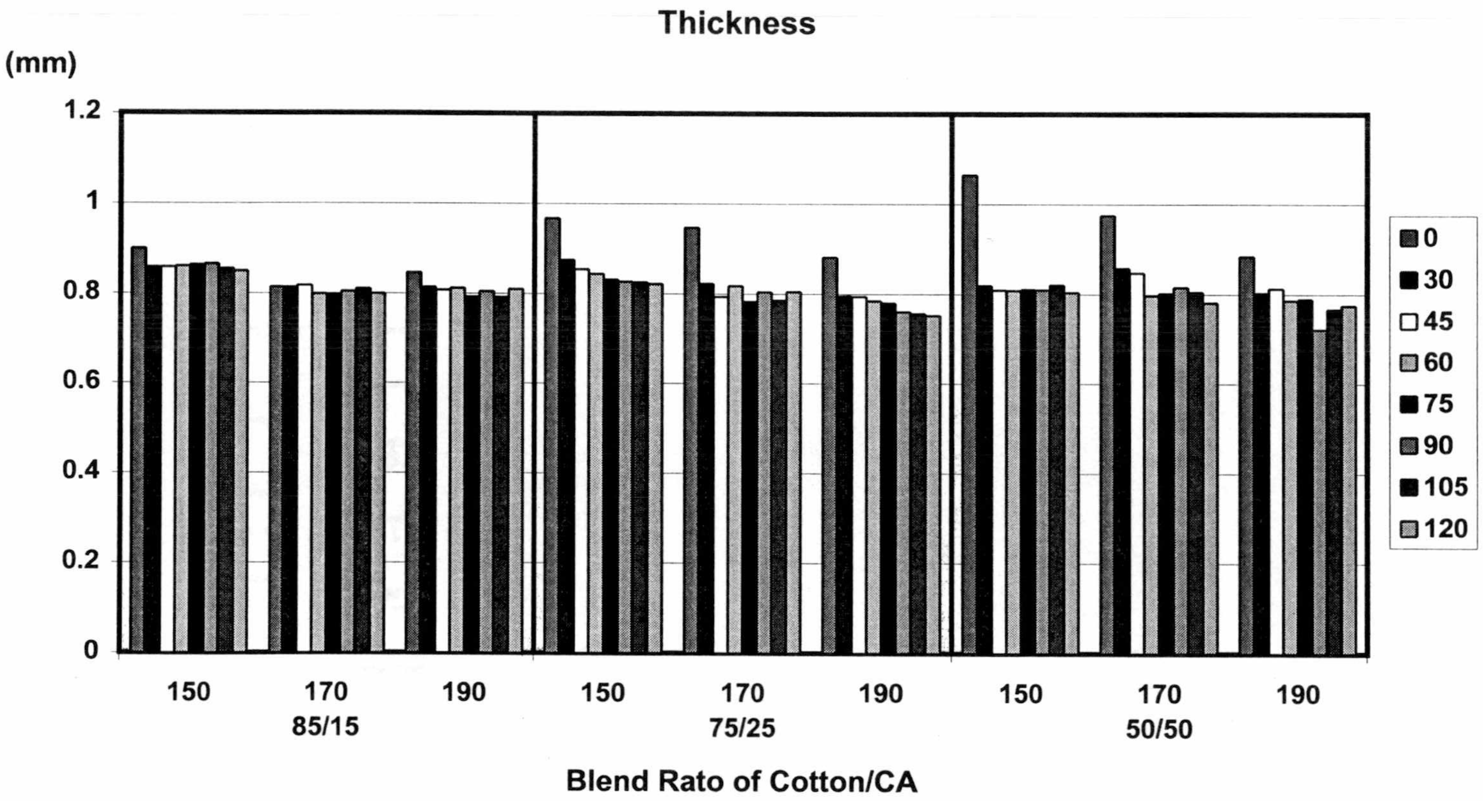
TIME		**BLEND**		
Frequency **Percent** **Row Percent** **Column Percent**	**50/50**	**75/25**	**85/15**	**Total**
0	2 8.33 66.67 25.00	1 4.17 33.33 12.50	0 0.00 0.00 0.00	3 12.50
30	0 0.00 0.00 0.00	2 8.33 66.67 25.00	1 4.17 33.33 12.50	3 12.50
45	1 4.17 33.33 12.50	1 4.17 33.33 12.50	1 4.17 33.33 12.50	3 12.50
60	0 0.00 0.00 0.00	1 4.17 33.33 12.50	2 8.33 66.67 25.00	3 12.50
75	1 4.17 33.33 12.50	0 0.00 0.00 0.00	2 8.33 66.67 25.00	3 12.50
90	1 4.17 33.33 12.50	2 8.33 66.67 25.00	0 0.00 0.00 0.00	3 12.50
105	2 8.33 66.67 25.00	0 0.00 0.00 0.00	1 4.17 33.33 12.50	3 12.50
120	1 4.17 33.33 12.50	1 4.17 33.33 12.50	1 4.17 33.33 12.50	3 12.50
Total	8 33.33	8 33.33	8 33.33	**24** **100**

A-1. (continued)

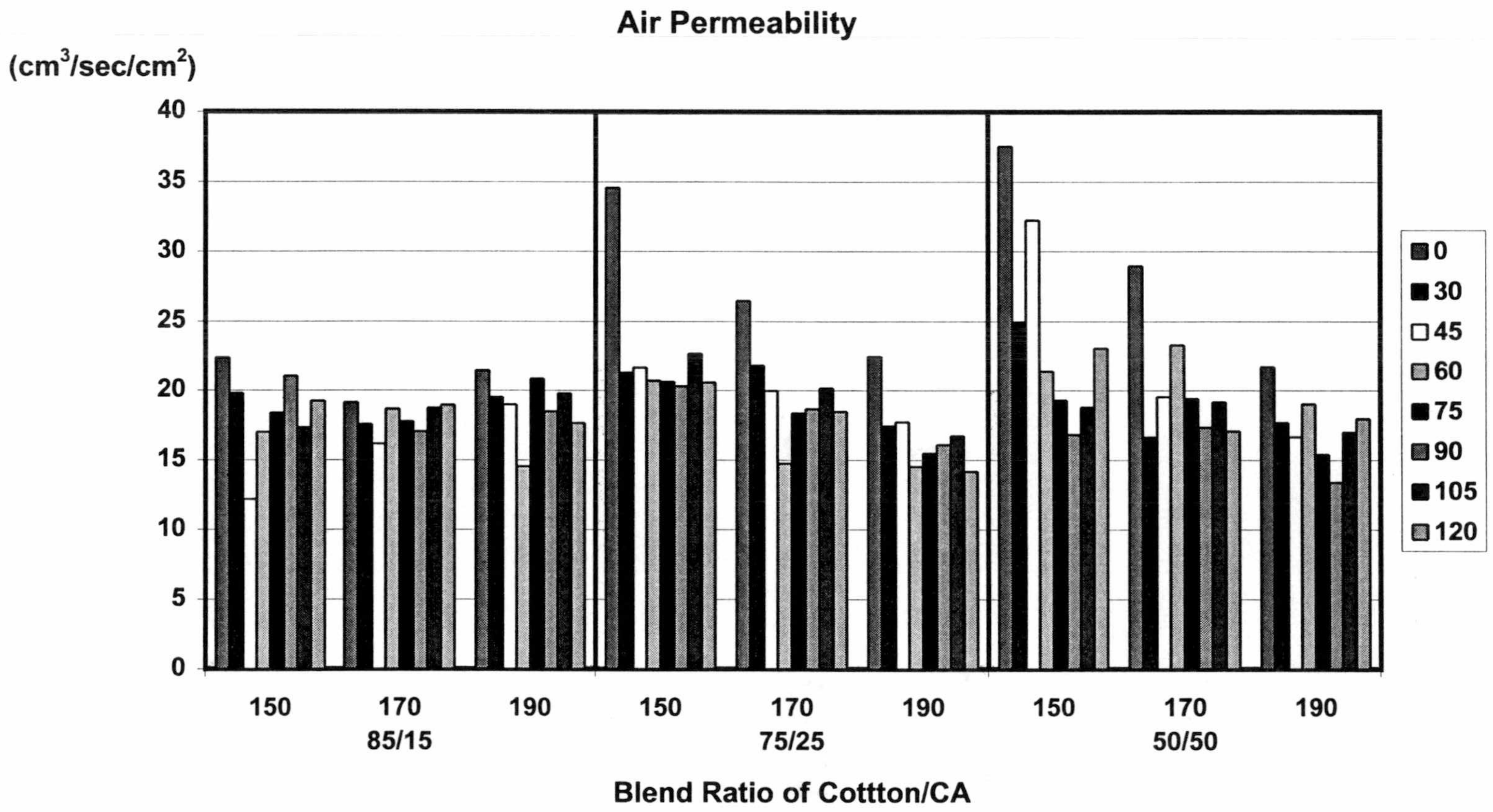
TEMP		**BLEND**		
Frequency Percent Row Percent Column Percent	**50/50**	**75/25**	**85/15**	**Total**
150	3	3	2	8
	12.50	12.50	8.33	33.33
	37.50	37.50	66.67	
	37.50	37.50	25.00	
170	3	2	3	8
	12.50	8.33	12.50	33.33
	37.50	66.67	37.50	
	37.50	25.00	37.50	
190	2	3	3	8
	8.33	12.50	12.50	33.33
	66.67	37.50	37.50	
	25.00	37.50	37.50	
Total	8	8	8	**24**
	33.33	33.33	33.33	**100**



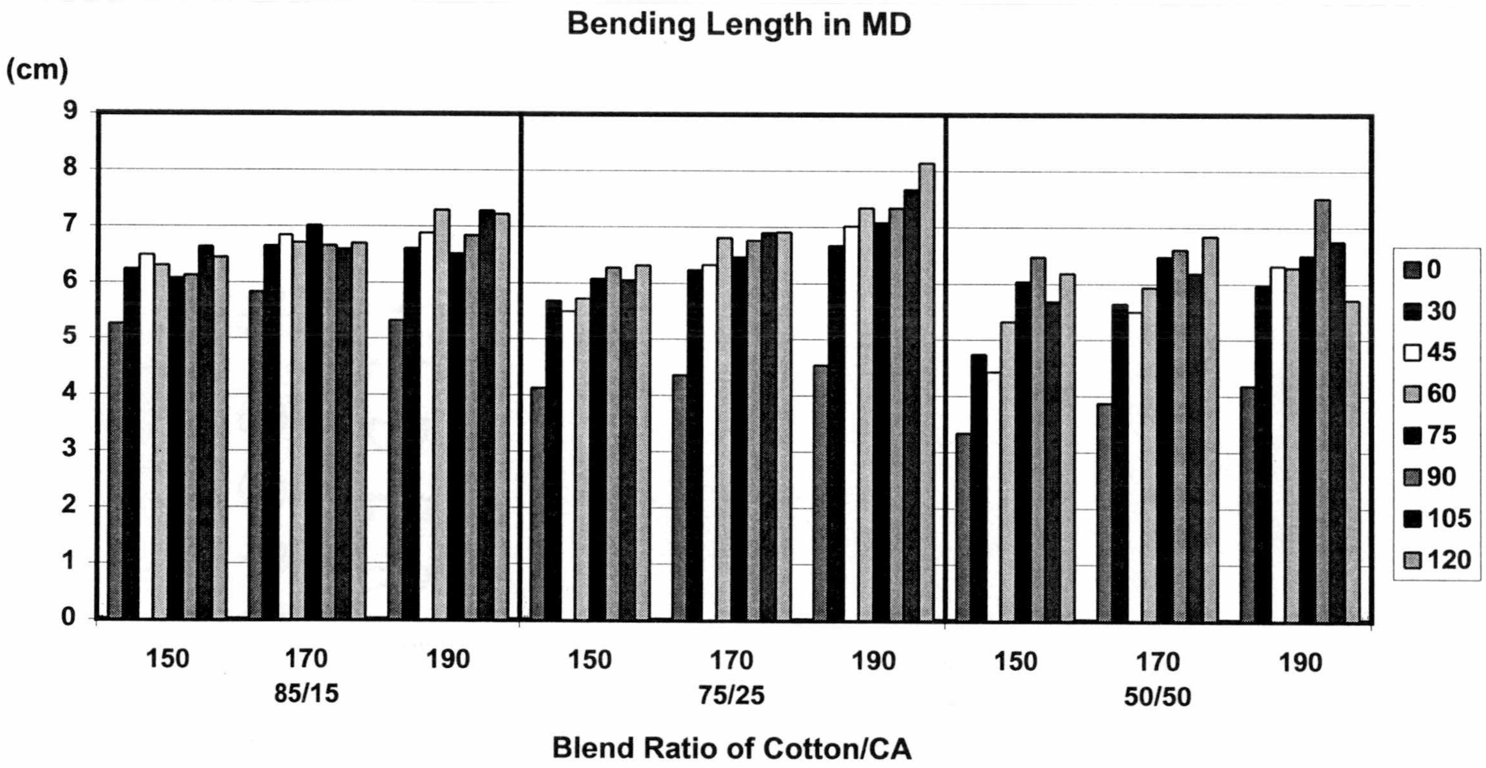
A-2. Results of 18 Physical Properties of Nonwovens from Cotton/CA.



A-2. (continued)



A-2. (continued)



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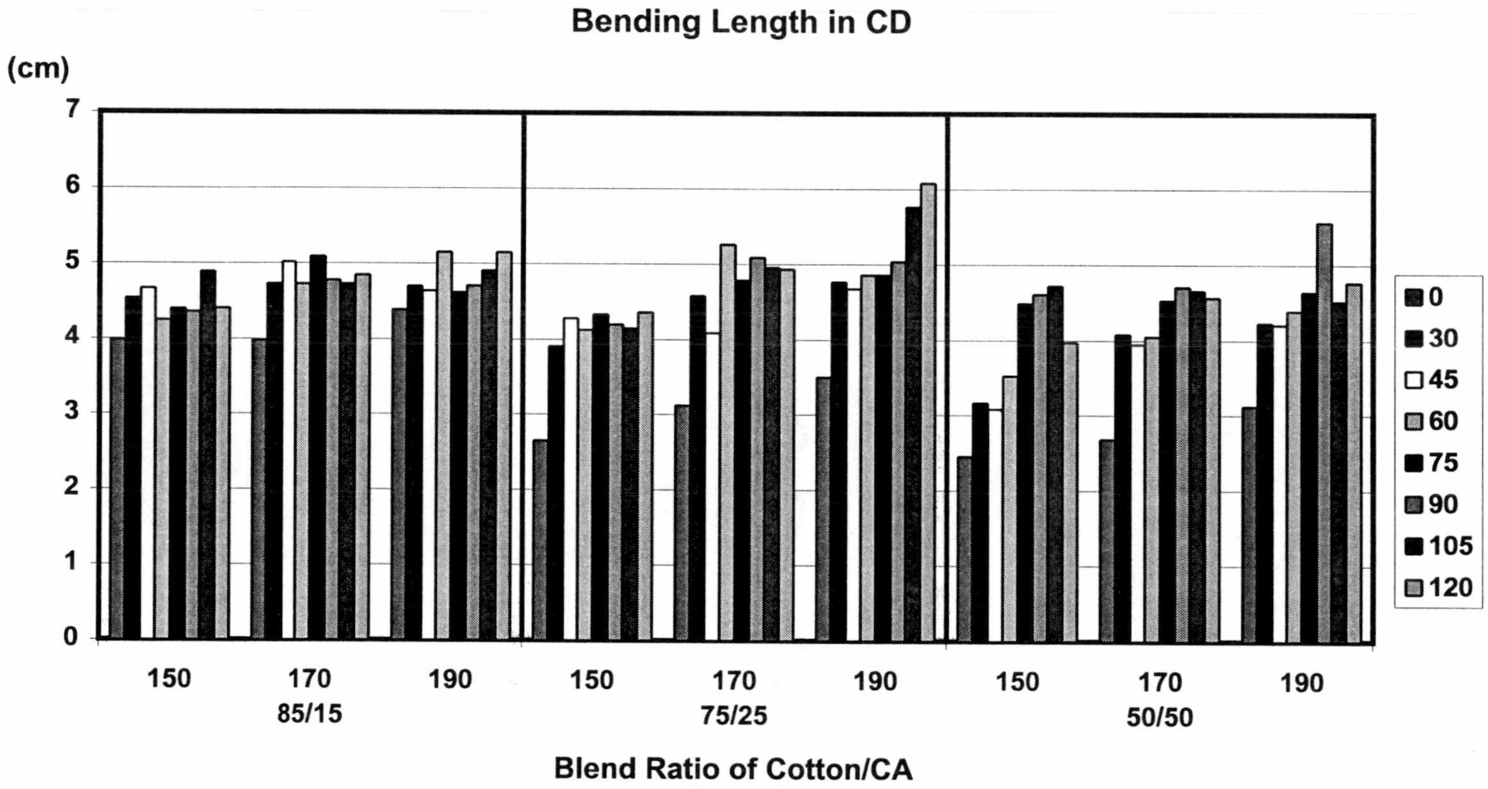
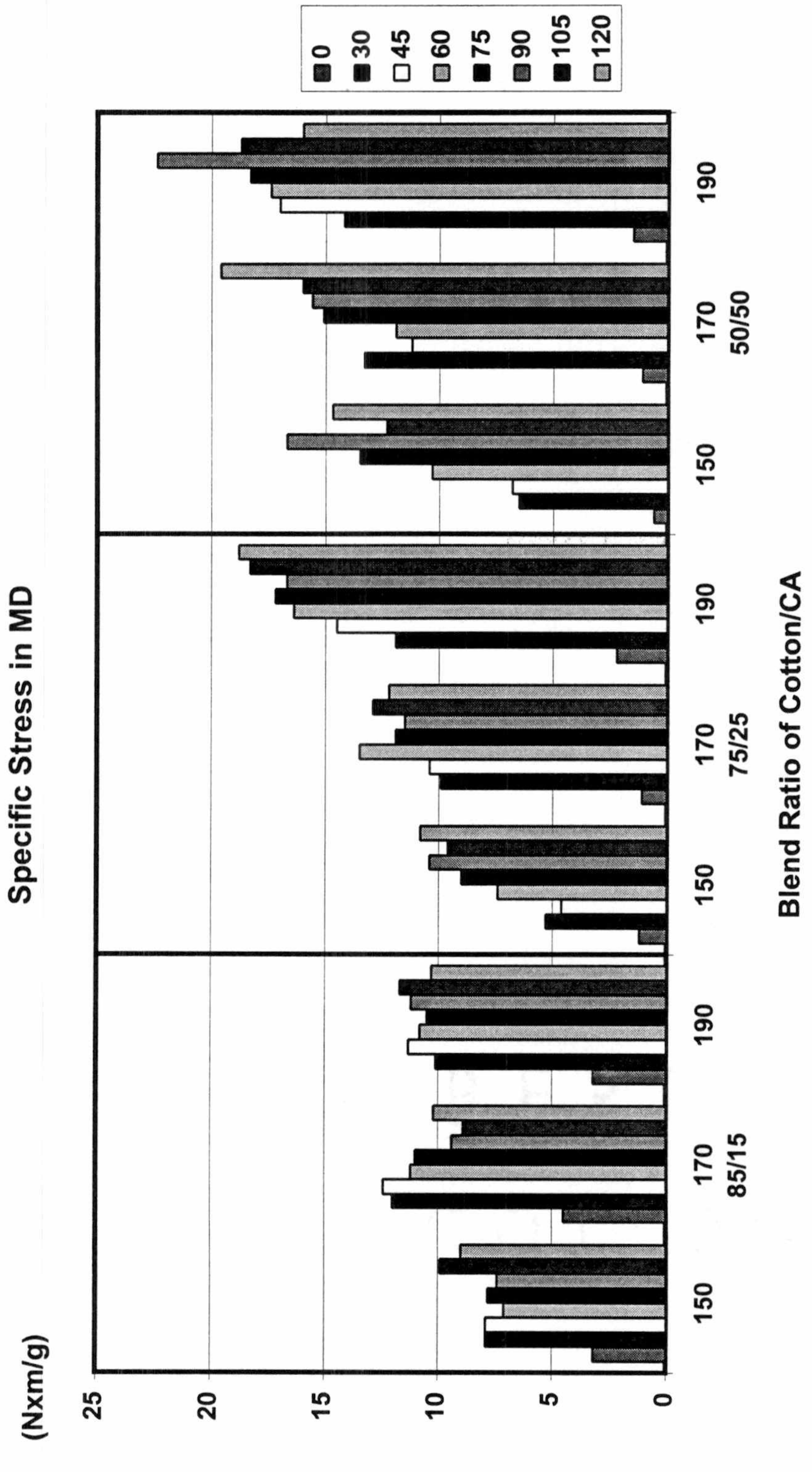


A-2. (continued)



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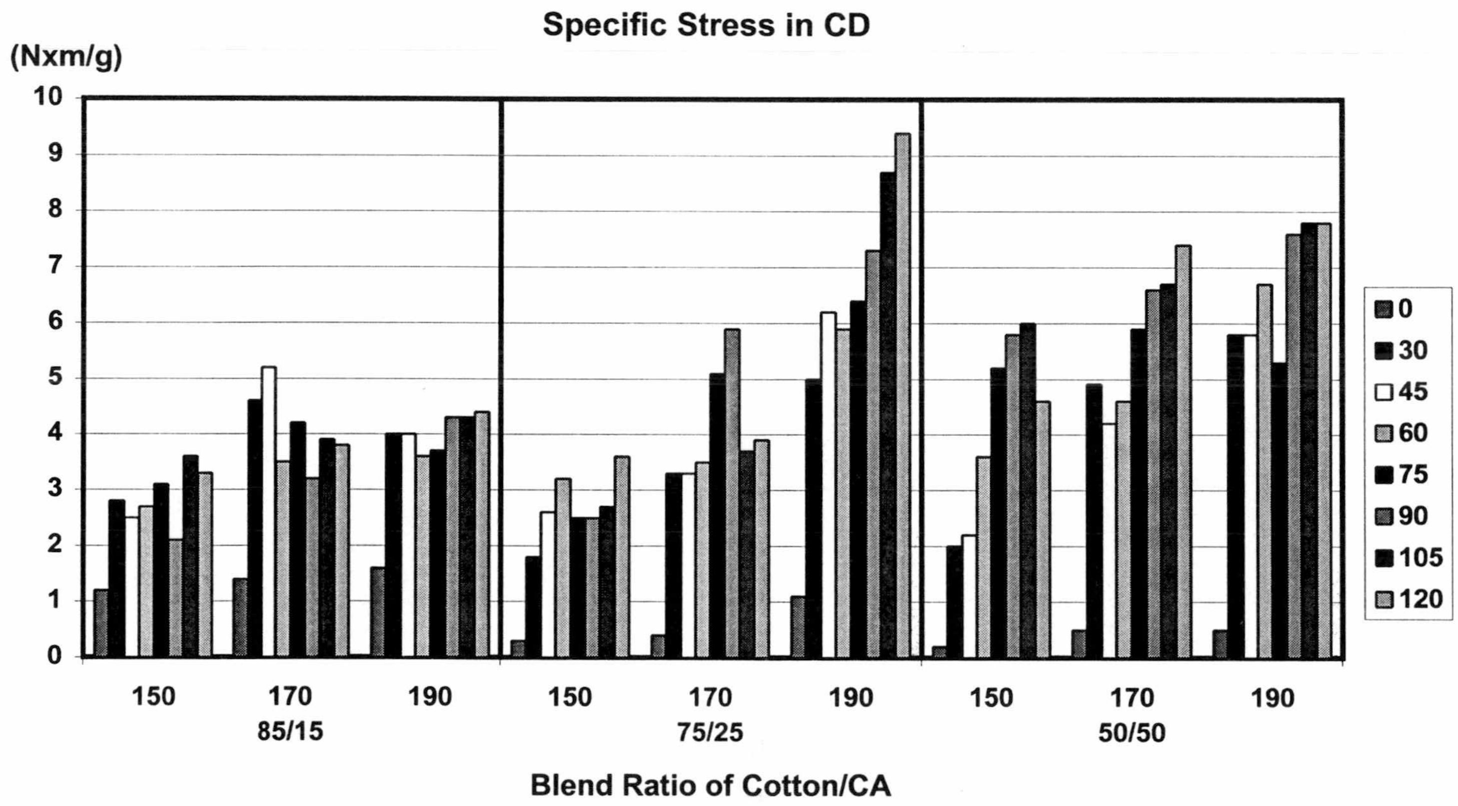
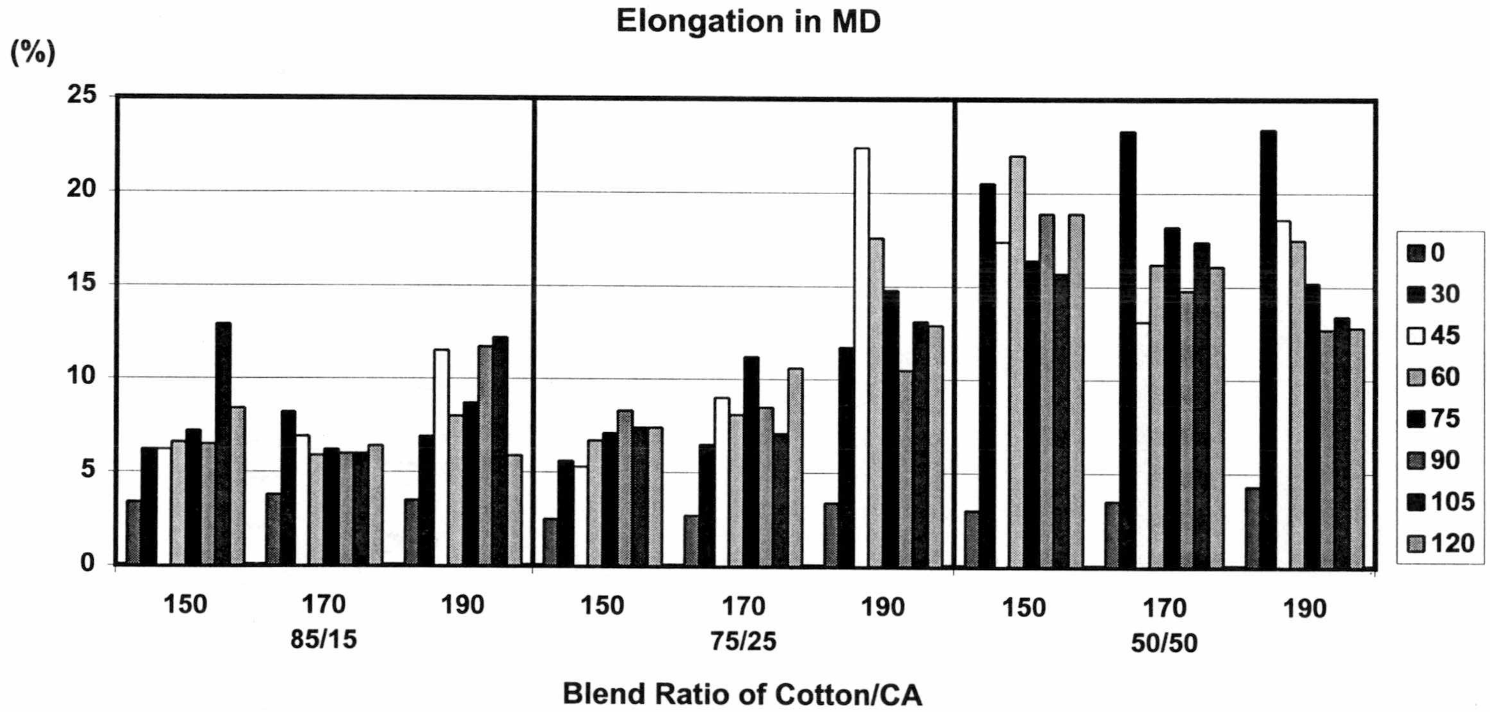
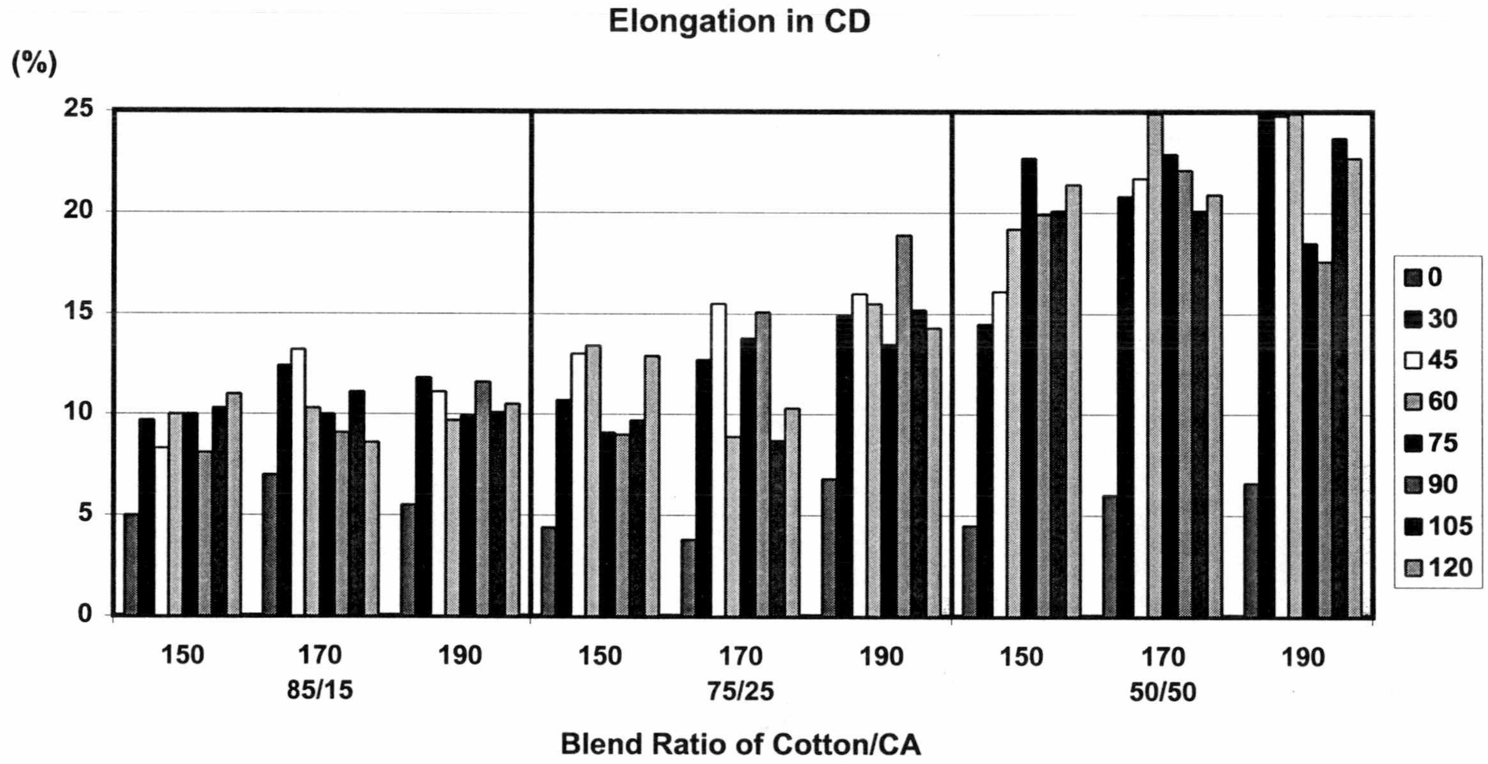


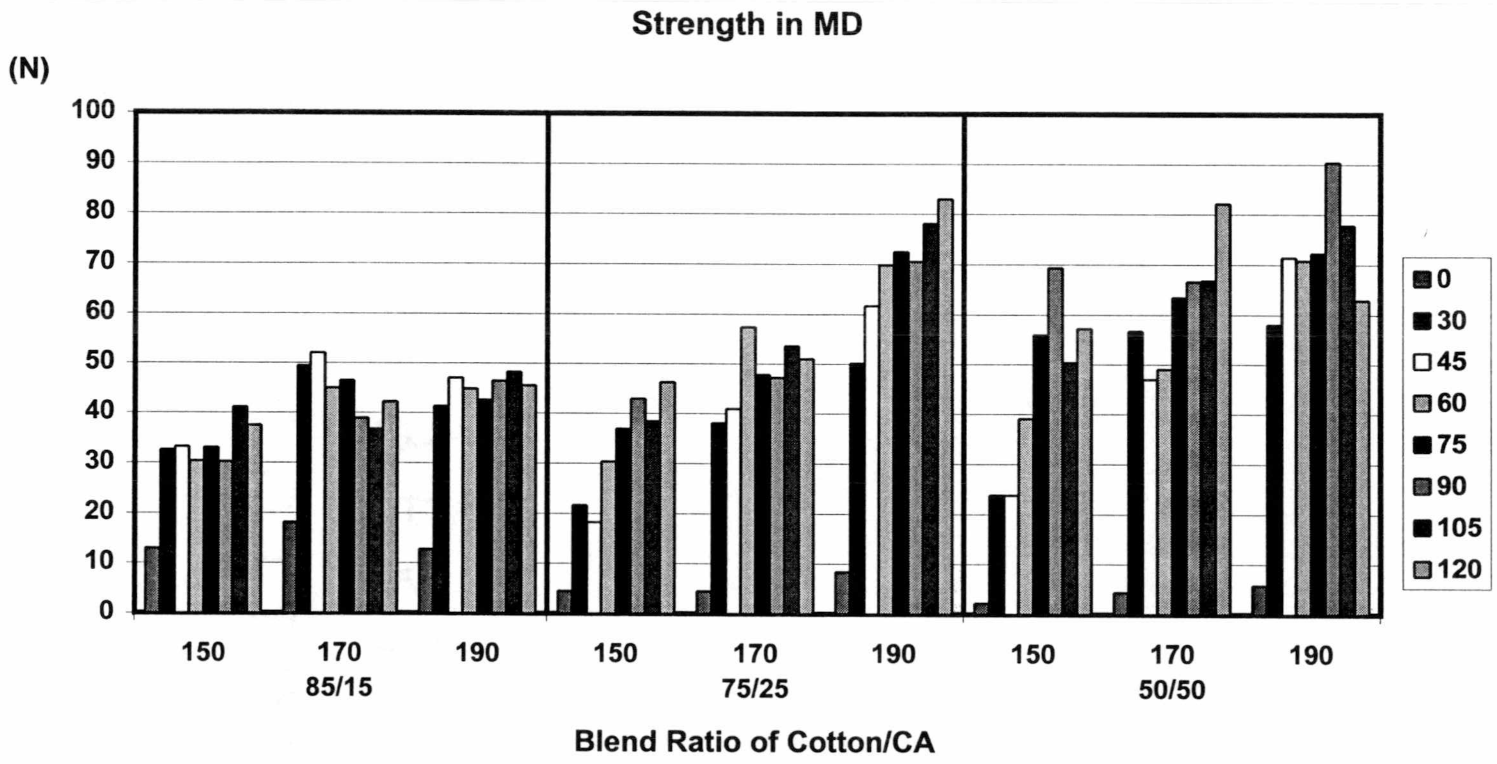
A-2. (continued)



A-2. (continued)



A-2. (continued)



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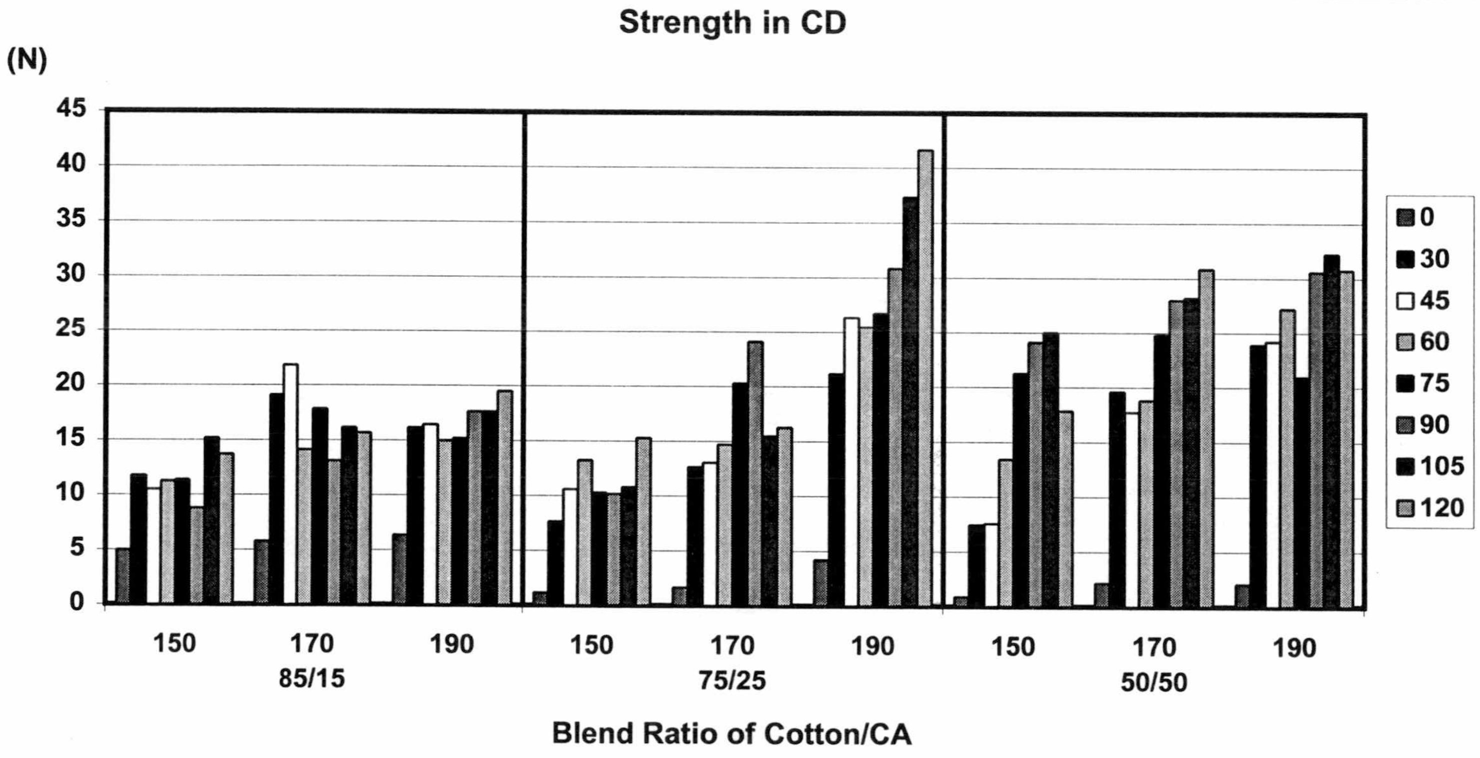

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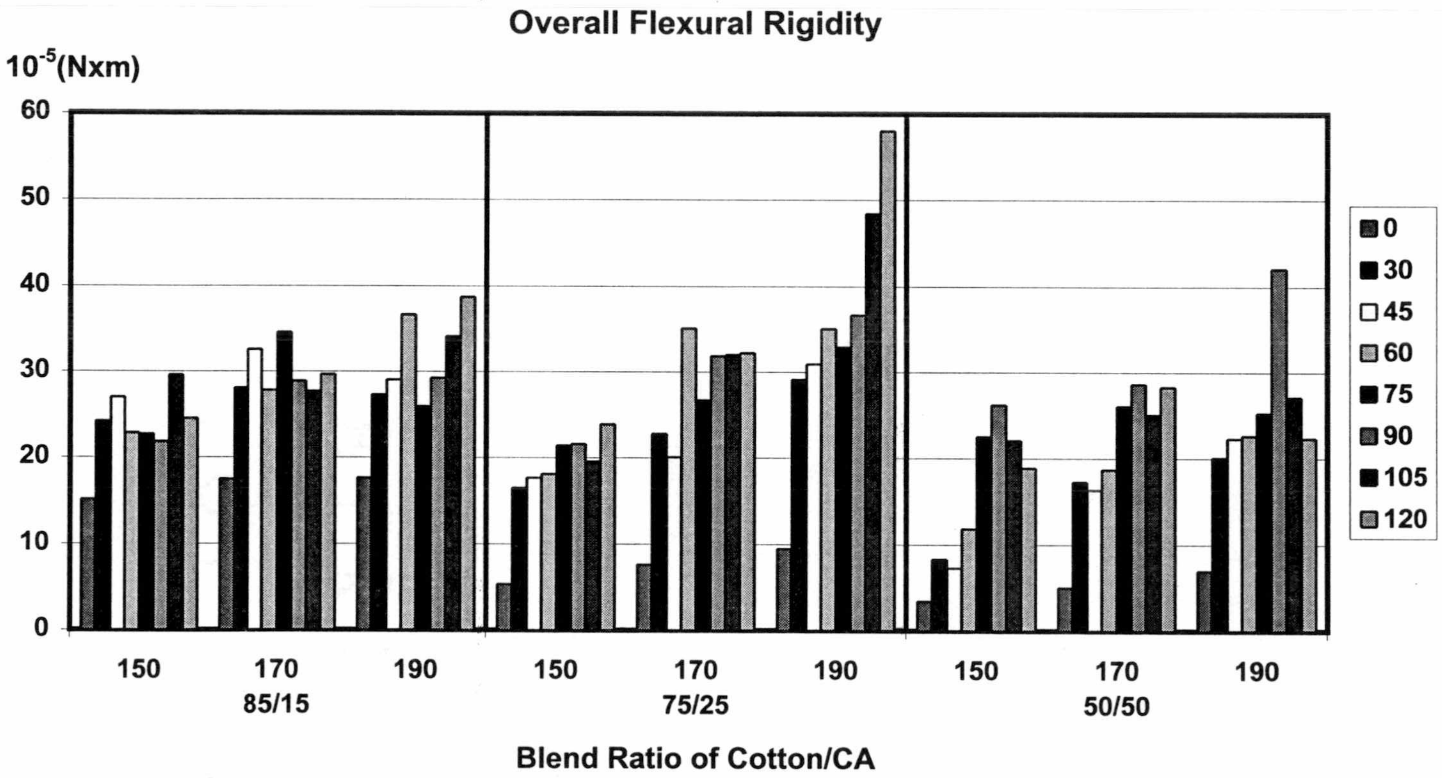

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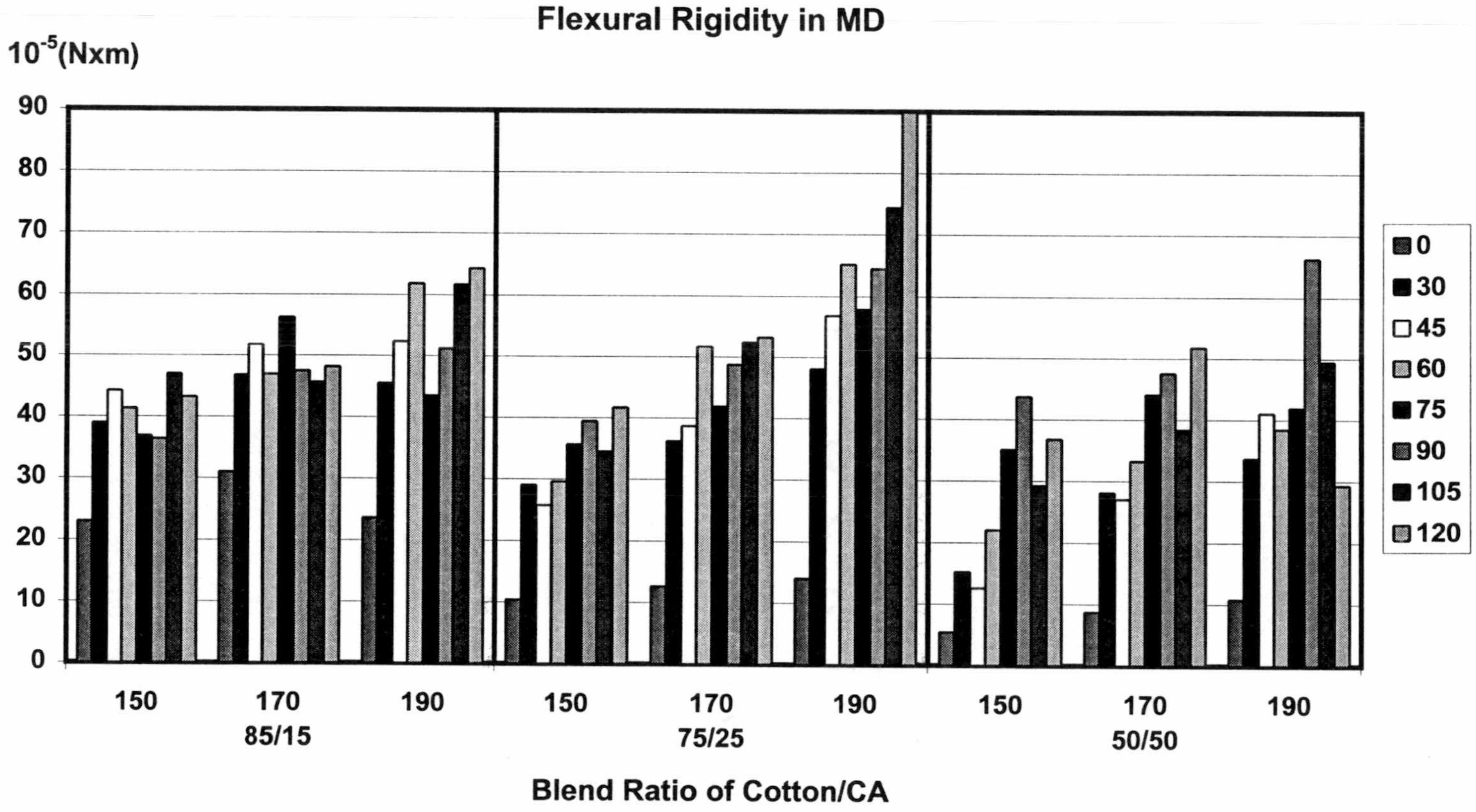

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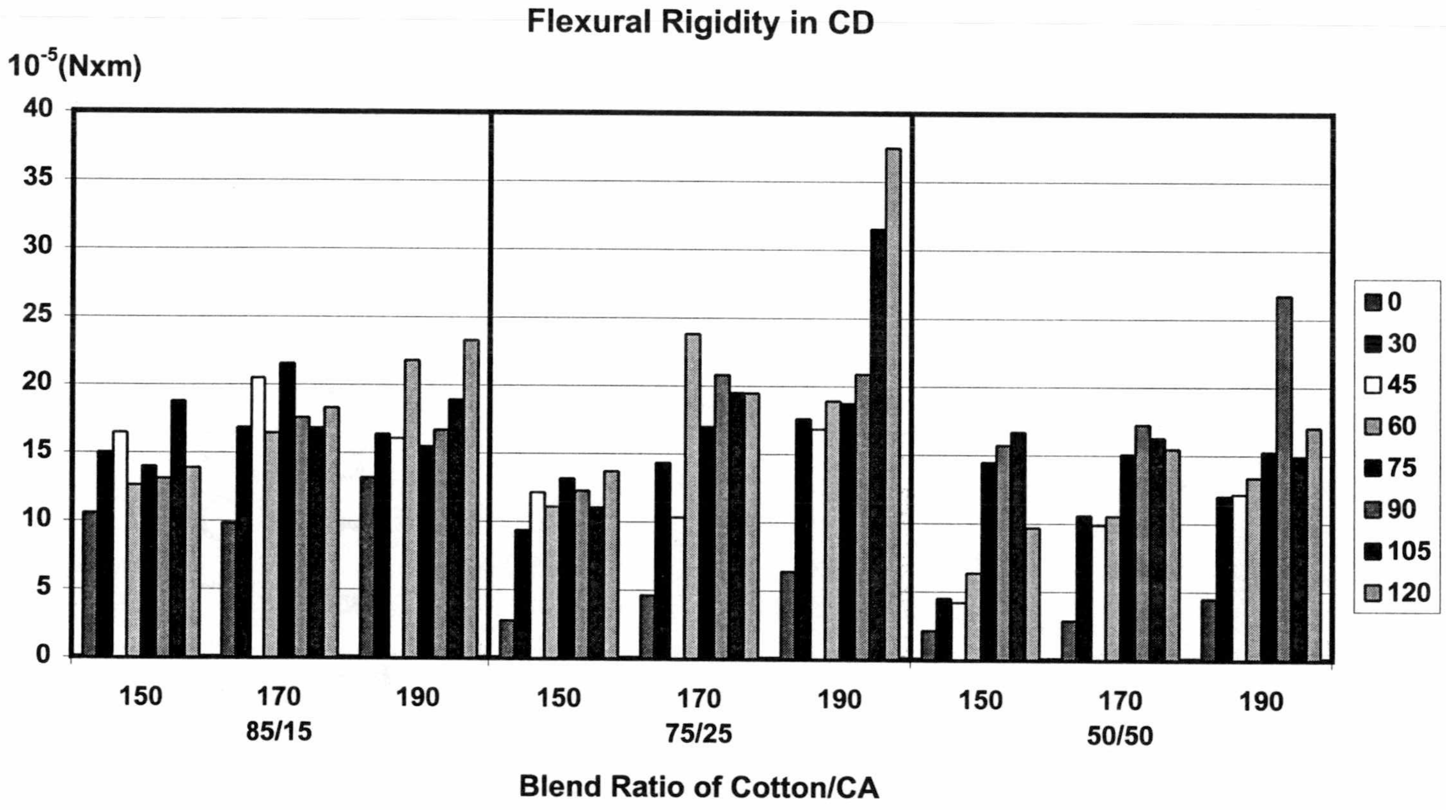

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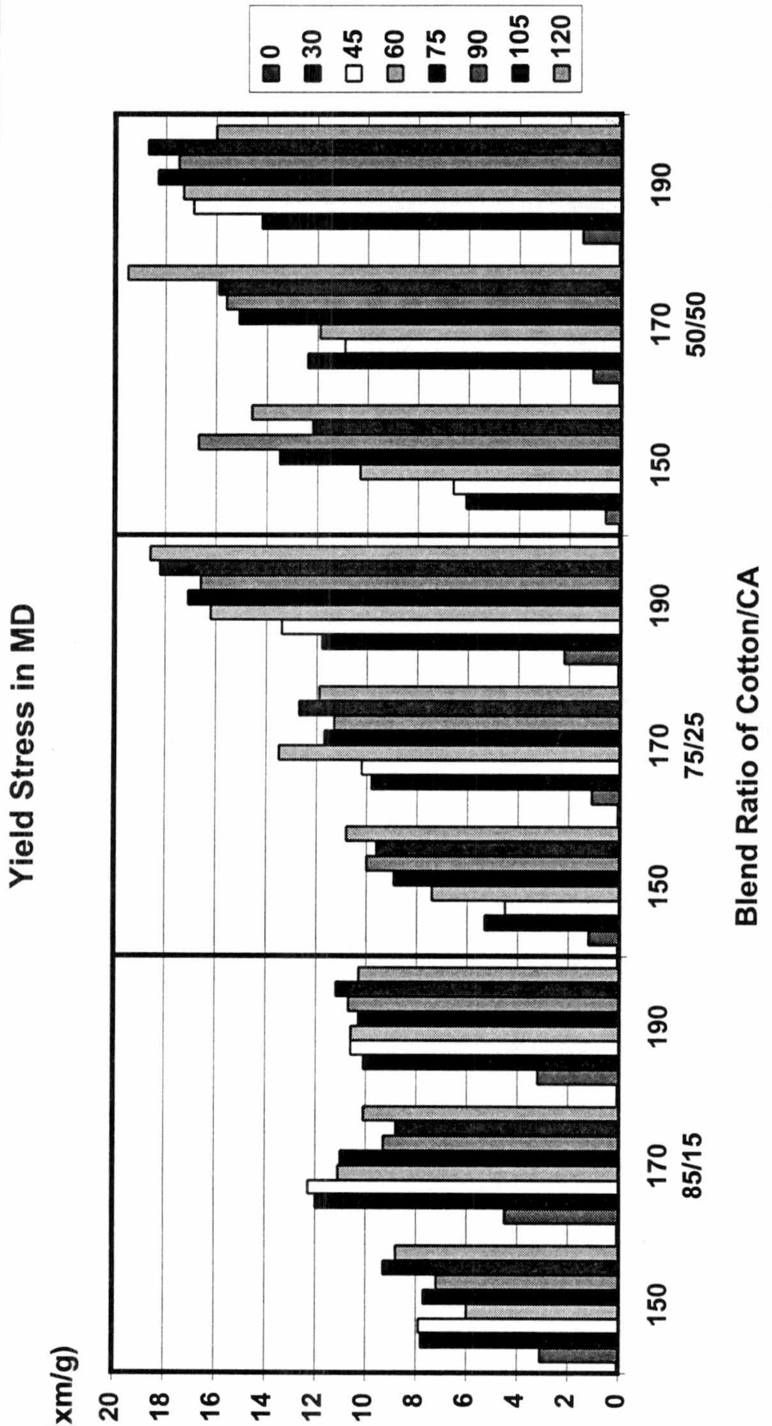

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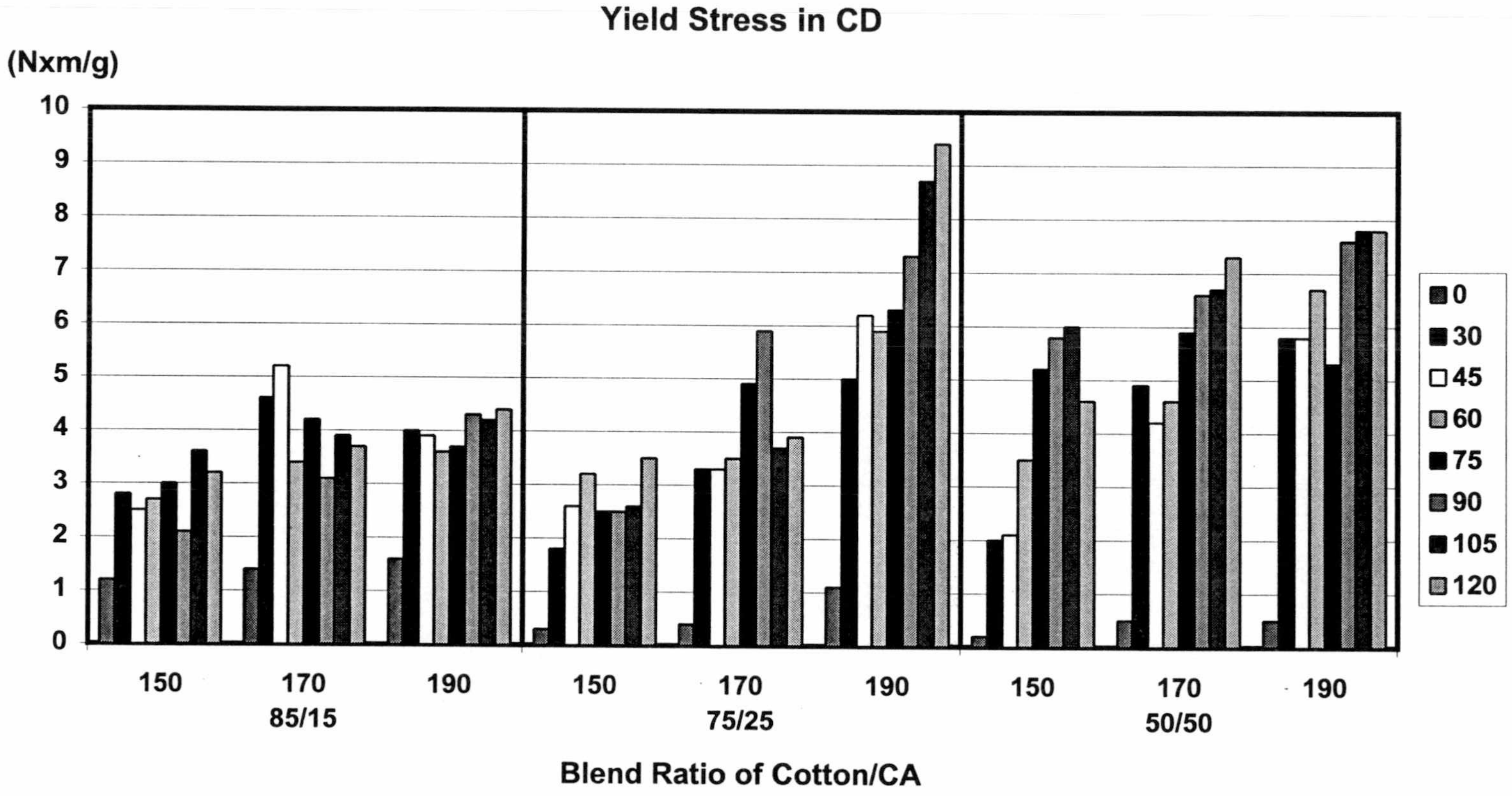

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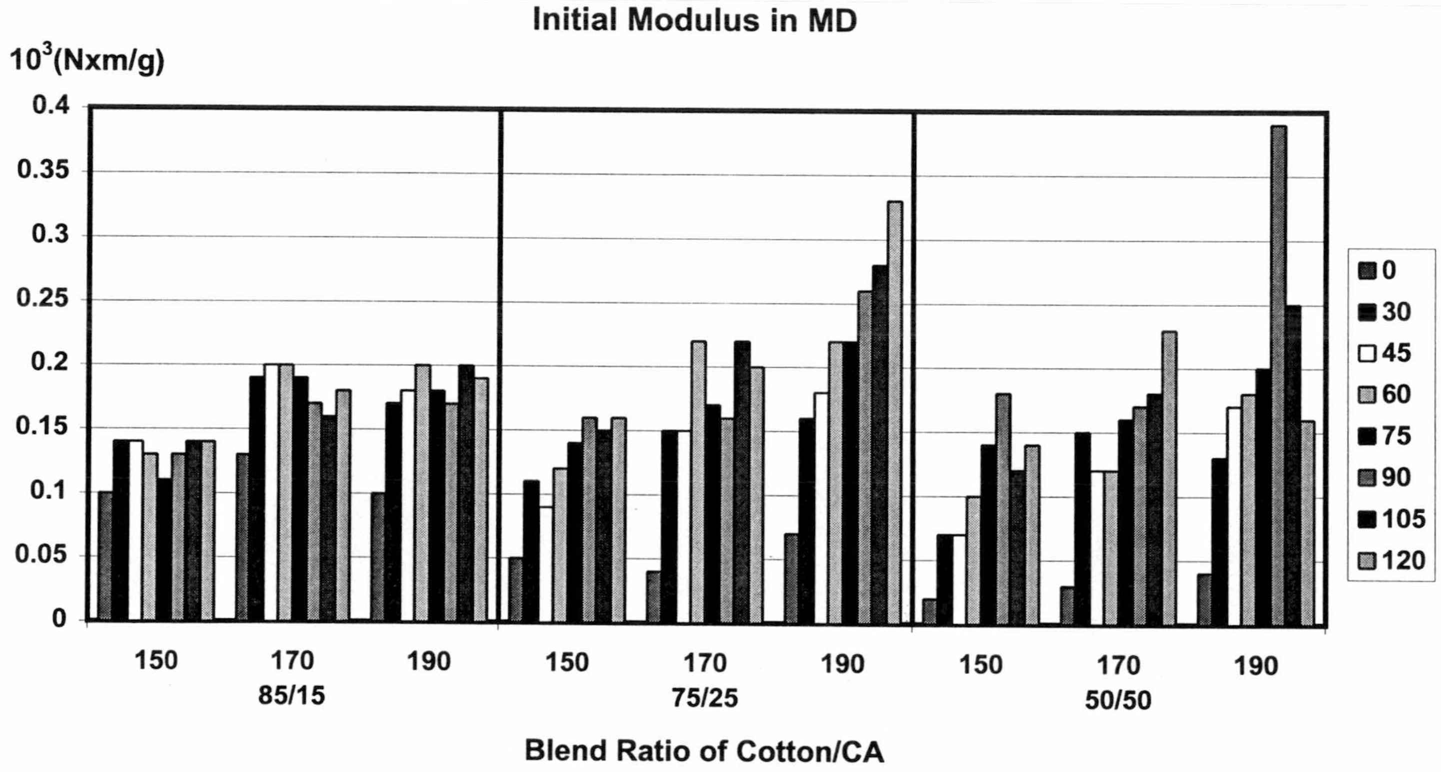

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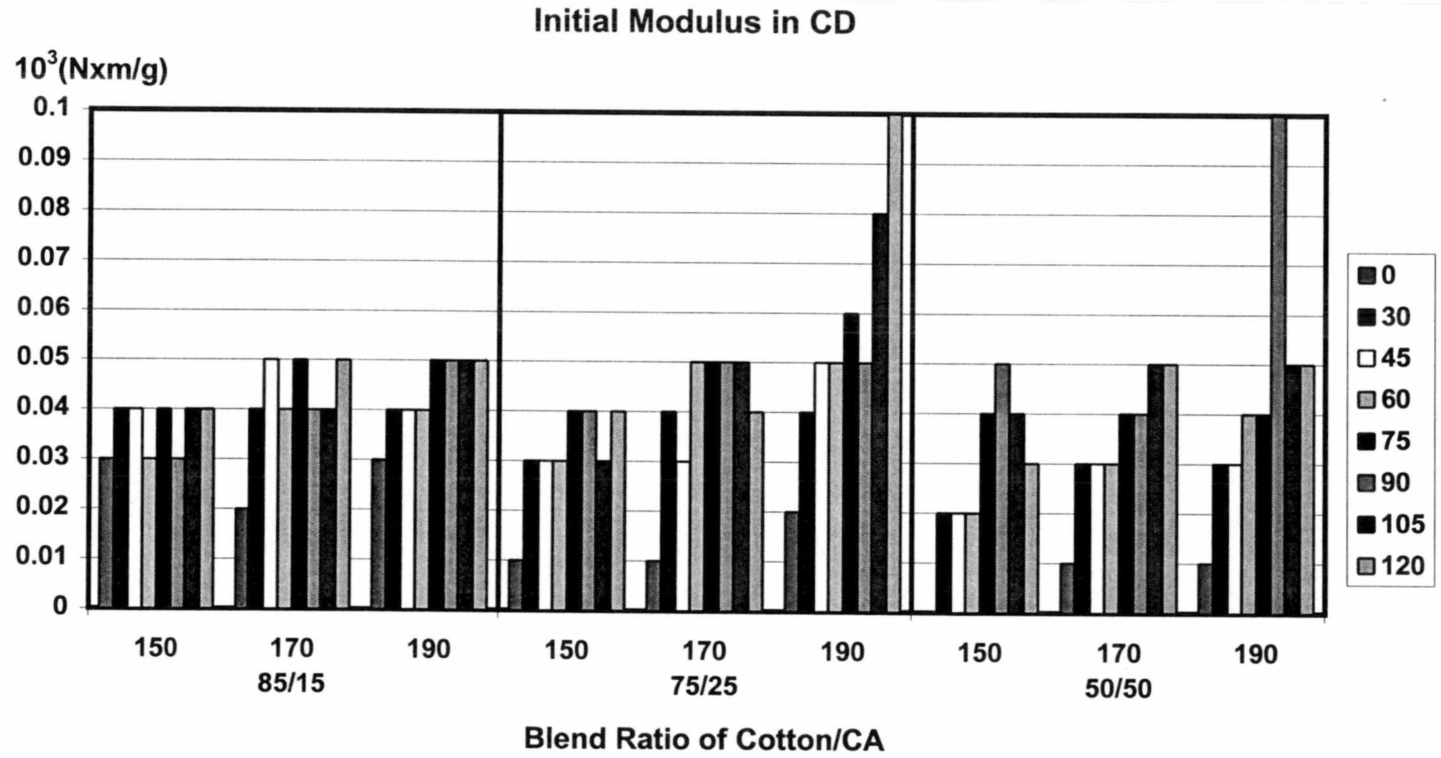


A-2. (continued)

A-3. Results of 18 Physical Properties of Nonwovens from Cotton and Cellulose Acetate Blends

Sample No.*	Basis Weight (g/m^2)	Thickness (mm)	Air Permeability $(cm^3/sec/cm^2)$	Bending Length (cm)		Specific Stress (N·m/g)		Elongation (%)	
				MD	CD	MD	CD	MD	CD
150-50-00	148.52	1.0646	37.53	3.34	2.45	0.6	0.2	3.0	4.5
150-50-30	145.51	0.8193	24.94	4.74	3.16	6.5	2.0	20.5	14.5
150-50-45	147.29	0.8093	32.24	4.43	3.08	6.8	2.2	17.4	16.1
150-50-60	148.67	0.8086	21.38	5.33	3.52	10.3	3.6	22.0	19.2
150-50-75	162.75	0.8110	19.30	6.04	4.49	13.5	5.2	16.4	22.7
150-50-90	163.86	0.8102	16.82	6.48	4.61	16.7	5.8	18.9	19.9
150-50-105	161.96	0.8207	18.78	5.69	4.72	12.3	6.0	15.7	20.1
150-50-120	153.26	0.8044	23.05	6.19	3.97	14.7	4.6	18.9	21.4
170-50-00	153.07	0.9750	28.95	3.87	2.68	1.1	0.5	3.5	6.0
170-50-30	159.08	0.8581	16.62	5.65	4.08	13.3	4.9	23.3	20.8
170-50-45	164.81	0.8481	19.54	5.51	3.94	11.2	4.2	13.1	21.7
170-50-60	161.84	0.7973	23.30	5.94	4.05	11.9	4.6	16.2	24.9
170-50-75	165.28	0.8024	19.40	6.48	4.53	15.1	5.9	18.2	22.9
170-50-90	168.13	0.8157	17.35	6.61	4.71	15.6	6.6	14.8	22.1
170-50-105	164.97	0.8053	19.16	6.19	4.66	16.0	6.7	17.4	20.1
170-50-120	165.28	0.7821	17.07	6.84	4.57	19.6	7.4	16.1	20.9

* Sample Designation; **Bonding Temperature**(150, 170, and 190 °C) - **Blend Ratio**(50/50, 75/25, and 85/15) - **Solvent Treatment Time**(00, 30, 45, 60, 75, 90, 105, and 120 min.)

A-3. (continued)

Sample No.	Basis Weight (g/m^2)	Thickness (mm)	Air Permeability (cm^3/sec/cm^2)	Bending Length (cm)		Specific Stress (N·m/g)		Elongation (%)	
				MD	CD	MD	CD	MD	CD
190-50-00	150.26	0.8837	21.68	4.18	3.12	1.5	0.5	4.3	6.6
190-50-30	161.49	0.8031	17.68	5.97	4.23	14.2	5.8	23.4	24.9
190-50-45	166.00	0.8131	16.66	6.32	4.21	17.0	5.8	18.6	24.8
190-50-60	160.85	0.7868	19.02	6.29	4.39	17.4	6.7	17.5	24.9
190-50-75	155.63	0.7895	15.38	6.50	4.64	18.3	5.3	15.2	18.5
190-50-90	158.83	0.7218	13.40	7.52	5.56	22.4	7.6	12.7	17.6
190-50-105	164.02	0.7671	16.99	6.75	4.52	18.7	7.8	13.4	23.7
190-50-120	155.00	0.7751	17.95	5.72	4.77	16.0	7.8	12.8	22.7
150-75-00	150.41	0.9674	34.56	4.13	2.65	1.2	0.3	2.5	4.4
150-75-30	160.85	0.8748	21.29	5.69	3.90	5.3	1.8	5.6	10.7
150-75-45	158.00	0.8541	21.65	5.50	4.28	4.6	2.6	5.3	13.0
150-75-60	160.69	0.8442	20.72	5.73	4.13	7.4	3.2	6.7	13.4
150-75-75	161.96	0.8315	20.61	6.08	4.33	9.0	2.5	7.1	9.1
150-75-90	162.75	0.8275	20.31	6.28	4.20	10.4	2.5	8.3	9.0
150-75-105	158.32	0.8258	22.69	6.06	4.15	9.6	2.7	7.4	9.7
150-75-120	168.60	0.8225	20.58	6.32	4.36	10.8	3.6	7.4	12.9

A-3. (continued)

Sample No.	Basis Weight (g/m^2)	Thickness (mm)	Air Permeability (cm^3/sec/cm^2)	Bending Length (cm)		Specific Stress (N·m/g)		Elongation (%)	
				MD	CD	MD	CD	MD	CD
170-75-00	155.06	0.9466	26.47	4.36	3.12	1.1	0.4	2.7	3.8
170-75-30	152.31	0.8227	21.80	6.24	4.58	9.9	3.3	6.5	12.7
170-75-45	155.73	0.7940	19.98	6.33	4.09	10.4	3.3	9.0	15.5
170-75-60	167.14	0.8178	14.74	6.81	5.26	13.5	3.5	8.1	8.9
170-75-75	157.85	0.7829	18.37	6.47	4.79	11.9	5.1	11.2	13.8
170-75-90	161.01	0.8044	18.67	6.76	5.09	11.5	5.9	8.5	15.1
170-75-105	163.07	0.7859	20.15	6.90	4.96	12.9	3.7	7.1	8.7
170-75-120	164.81	0.8050	18.47	6.91	4.94	12.2	3.9	10.6	10.3
190-75-00	152.47	0.8809	22.47	4.54	3.50	2.2	1.1	3.4	6.8
190-75-30	165.44	0.7955	17.43	6.67	4.77	11.9	5.0	11.7	14.9
190-75-45	167.65	0.7944	17.72	7.02	4.68	14.5	6.2	22.4	16.0
190-75-60	168.13	0.7852	14.52	7.34	4.86	16.4	5.9	17.6	15.5
190-75-75	166.39	0.7806	15.48	7.08	4.86	17.2	6.4	14.8	13.5
190-75-90	166.39	0.7615	16.09	7.34	5.04	16.7	7.3	10.5	18.9
190-75-105	168.29	0.7570	16.71	7.67	5.76	18.3	8.7	13.1	15.2
190-75-120	174.14	0.7527	14.16	8.14	6.08	18.8	9.4	12.9	14.3

A-3. (continued)

Sample No.	Basis Weight (g/m^2)	Thickness (mm)	Air Permeability (cm^3/sec/cm^2)	Bending Length (cm)		Specific Stress (N·m/g)		Elongation (%)	
				MD	CD	MD	CD	MD	CD
150-85-00	160.28	0.8996	22.35	5.27	3.99	3.2	1.2	3.4	5.0
150-85-30	163.70	0.8588	19.81	6.24	4.54	7.9	2.8	6.2	9.7
150-85-45	165.12	0.8583	12.17	6.49	4.67	7.9	2.5	6.2	8.3
150-85-60	167.97	0.8603	17.01	6.31	4.25	7.1	2.7	6.6	10.0
150-85-75	167.50	0.8635	18.38	6.08	4.40	7.8	3.1	7.2	10.0
150-85-90	161.49	0.8654	21.05	6.13	4.36	7.4	2.1	6.5	8.1
150-85-105	163.86	0.8550	17.34	6.64	4.89	9.9	3.6	12.9	10.3
150-85-120	165.12	0.8500	19.25	6.45	4.41	9.0	3.3	8.4	11.0
170-85-00	159.11	0.8142	19.13	5.84	3.98	4.5	1.4	3.8	7.0
170-85-30	162.43	0.8139	17.56	6.65	4.73	12.0	4.6	8.2	12.4
170-85-45	165.12	0.8182	16.17	6.84	5.02	12.4	5.2	6.9	13.2
170-85-60	158.64	0.7993	18.66	6.71	4.73	11.2	3.5	5.9	10.3
170-85-75	166.70	0.7989	17.75	7.01	5.09	11.0	4.2	6.2	10.0
170-85-90	164.17	0.8050	17.05	6.66	4.78	9.4	3.2	6.0	9.1
170-85-105	162.12	0.8106	18.74	6.60	4.73	8.9	3.9	6.0	11.1
170-85-120	163.54	0.8003	18.96	6.70	4.85	10.2	3.8	6.4	8.6

A-3. (continued)

Sample No.	Basis Weight (g/m^2)	Thickness (mm)	Air Permeability (cm^3/sec/cm^2)	Bending Length (cm)		Specific Stress (N·m/g)		Elongation (%)	
				MD	CD	MD	CD	MD	CD
190-85-00	159.11	0.8466	21.44	5.33	4.39	3.2	1.6	3.5	5.5
190-85-30	160.85	0.8147	19.51	6.61	4.70	10.1	4.0	6.9	11.8
190-85-45	164.17	0.8080	18.99	6.88	4.64	11.3	4.0	11.5	11.1
190-85-60	162.81	0.8122	14.55	7.29	5.15	10.8	3.6	8.0	9.7
190-85-75	160.22	0.7931	20.85	6.52	4.62	10.5	3.7	8.7	9.9
190-85-90	163.22	0.8051	18.50	6.84	4.71	11.2	4.3	11.7	11.6
190-85-105	163.07	0.7922	19.78	7.28	4.91	11.7	4.3	12.2	10.1
190-85-120	174.30	0.8101	17.64	7.22	5.15	10.3	4.4	5.9	10.5
****E150-75-00**	152.94	0.8662	31.22	4.73	3.18	2.2	0.9	3.5	5.4
E150-75-30	160.69	0.8212	22.08	6.06	4.03	7.6	2.4	6.7	9.9
E150-50-45	166.38	0.7822	17.27	5.58	3.97	13.3	3.9	13.2	18.5
E150-85-60	158.80	0.8137	22.32	6.00	4.28	7.3	2.8	5.7	10.2
E150-50-75	168.92	0.7888	18.36	6.16	4.57	17.0	4.9	14.2	18.8
E150-75-90	168.60	0.7986	19.84	6.55	4.91	11.0	3.9	7.0	12.2
E150-85-105	174.77	0.8128	17.50	7.39	4.97	9.7	3.6	5.8	10.1
E150-50-120	179.52	0.7724	12.13	7.07	5.34	19.8	9.2	16.8	22.4

** E : Extra nonwovens fabricated based on fractional factorial design.

A-3. (continued)

Sample No.	Basis Weight (g/m^2)	Thickness (mm)	Air Permeability (cm^3/sec/cm^2)	Bending Length (cm)		Specific Stress (N·m/g)		Elongation (%)	
				MD	CD	MD	CD	MD	CD
E170-50-00	150.04	0.8819	37.29	3.84	2.81	1.2	0.5	3.9	5.9
E170-75-30	165.12	0.8136	21.05	6.66	4.59	11.1	3.5	7.4	13.5
E170-75-45	165.12	0.7996	18.85	6.68	4.56	11.9	4.0	7.9	15.0
E170-85-60	168.44	0.8082	19.06	6.67	4.80	8.9	2.9	6.4	9.3
E170-85-75	168.76	0.8005	17.60	6.91	5.12	8.9	5.0	6.0	12.0
E170-50-90	166.70	0.7573	15.41	6.52	4.38	19.6	6.7	14.4	23.0
E170-50-105	171.45	0.7622	11.10	6.86	4.80	20.4	7.7	15.5	20.9
E170-85-120	170.66	0.8025	16.84	7.08	5.09	10.1	3.2	6.3	7.8
E190-50-00	153.10	0.8381	25.58	4.59	3.53	2.3	0.9	5.5	7.9
E190-85-30	167.97	0.7944	16.64	6.79	4.88	9.8	2.9	6.2	7.6
E190-85-45	164.17	0.7925	17.29	6.36	4.52	7.0	2.5	5.5	10.0
E190-75-60	163.86	0.7756	17.70	7.10	4.77	12.9	4.9	13.5	15.8
E190-85-75	171.13	0.7855	15.55	7.23	4.88	9.7	3.1	5.8	7.3
E190-75-90	171.13	0.7705	15.47	7.27	5.02	15.0	5.8	12.8	15.4
E190-50-105	173.35	0.7220	11.35	7.20	5.19	20.7	8.6	14.7	18.8
E190-75-120	170.66	0.7871	13.03	7.29	5.00	14.6	5.9	16.9	17.1

A-3. (continued)

Sample No.*	Strength (N)		Overall Flexural Rigidity (G_o) (10^{-5} N·m)	Flexural Rigidity (10^{-5} N·m)		Yield Stress (N·m/g)		Initial Modulus (10^3 N·m/g)	
	MD	CD		MD	CD	MD	CD	MD	CD
150-50-00	2.25	0.88	3.42	5.41	2.14	0.6	0.2	0.02	0.00
150-50-30	23.81	7.45	8.27	15.19	4.50	6.1	2.0	0.07	0.02
150-50-45	23.81	7.55	7.27	12.55	4.21	6.6	2.1	0.07	0.02
150-50-60	39.10	13.43	11.84	22.06	6.35	10.3	3.5	0.10	0.02
150-50-75	55.86	21.27	22.52	35.14	14.44	13.5	5.2	0.14	0.04
150-50-90	69.38	24.11	26.22	43.70	15.73	16.7	5.8	0.18	0.05
150-50-105	50.37	24.99	22.09	29.24	16.69	12.2	6.0	0.12	0.04
150-50-120	57.23	17.84	18.89	36.79	9.70	14.6	4.6	0.14	0.03
170-50-00	4.41	2.16	5.01	8.69	2.89	1.1	0.5	0.03	0.01
170-50-30	56.74	19.60	17.26	28.12	10.58	12.4	4.9	0.15	0.03
170-50-45	46.94	17.74	16.34	27.02	9.88	10.9	4.2	0.12	0.03
170-50-60	49.00	18.82	18.72	33.24	10.54	11.9	4.6	0.12	0.03
170-50-75	63.41	24.79	26.06	44.07	15.05	15.1	5.9	0.16	0.04
170-50-90	66.64	28.03	28.63	47.59	17.22	15.6	6.6	0.17	0.04
170-50-105	66.84	28.22	25.05	38.35	16.26	15.9	6.7	0.18	0.05
170-50-120	82.12	30.87	28.31	51.83	15.46	19.5	7.3	0.23	0.05

A-3. (continued)

Sample No.	Strength (N)		Overall Flexural Rigidity (G_o) (10^{-5} N·m)	Flexural Rigidity (10^{-5} N·m)		Yield Stress (N·m/g)		Initial Modulus (10^{3} N·m/g)	
	MD	CD		MD	CD	MD	CD	MD	CD
190-50-00	5.78	2.06	6.94	10.75	4.47	1.5	0.5	0.04	0.01
190-50-30	58.02	23.91	20.08	33.67	11.98	14.2	5.8	0.13	0.03
190-50-45	71.44	24.21	22.32	41.06	12.14	16.9	5.8	0.17	0.03
190-50-60	70.85	27.24	22.65	38.48	13.34	17.3	6.7	0.18	0.04
190-50-75	72.23	20.97	25.26	41.89	15.24	18.3	5.3	0.20	0.04
190-50-90	90.36	30.58	42.08	66.19	26.75	17.5	7.6	0.39	0.10
190-50-105	77.81	32.24	27.09	49.43	14.85	18.7	7.8	0.25	0.05
190-50-120	62.92	30.77	22.34	29.34	17.02	16.0	7.8	0.16	0.05
150-75-00	4.51	1.18	5.34	10.39	2.74	1.2	0.3	0.05	0.01
150-75-30	21.66	7.64	16.47	29.04	9.35	5.3	1.8	0.11	0.03
150-75-45	18.27	10.58	17.68	25.76	12.14	4.5	2.6	0.09	0.03
150-75-60	30.38	13.23	18.13	29.63	11.09	7.4	3.2	0.12	0.03
150-75-75	36.95	10.29	21.43	35.67	13.15	8.9	2.5	0.14	0.04
150-75-90	42.92	10.19	21.61	39.50	12.26	10.0	2.5	0.16	0.04
150-75-105	38.51	10.78	19.55	34.53	11.06	9.6	2.6	0.15	0.03
150-75-120	46.26	15.29	23.90	41.71	13.69	10.8	3.5	0.16	0.04

A-3. (continued)

Sample No.	Strength (N)		Overall Flexural Rigidity (G_o) (10^{-5} N·m)	Flexural Rigidity (10^{-5} N·m)		Yield Stress (N·m/g)		Initial Modulus (10^3 N·m/g)	
	MD	CD		MD	CD	MD	CD	MD	CD
170-75-00	4.51	1.67	7.62	12.60	4.62	1.1	0.4	0.04	0.01
170-75-30	38.12	12.64	22.80	36.27	14.34	9.8	3.3	0.15	0.04
170-75-45	40.96	13.03	20.10	38.71	10.34	10.2	3.3	0.15	0.03
170-75-60	57.43	14.70	35.11	51.73	23.83	13.5	3.5	0.22	0.05
170-75-75	47.82	20.29	26.69	41.90	16.98	11.7	4.9	0.17	0.05
170-75-90	47.24	24.11	31.85	48.75	20.81	11.3	5.9	0.16	0.05
170-75-105	53.51	15.48	32.00	52.49	19.50	12.7	3.7	0.22	0.05
170-75-120	51.01	16.27	32.21	53.28	19.47	11.9	3.9	0.20	0.04
190-75-00	8.43	4.21	9.47	13.98	6.41	2.2	1.1	0.07	0.02
190-75-30	50.08	21.17	29.10	48.11	17.60	11.8	5.0	0.16	0.04
190-75-45	61.74	26.36	30.94	56.84	16.84	13.4	6.2	0.18	0.05
190-75-60	69.87	25.48	35.10	65.16	18.91	16.2	5.9	0.22	0.05
190-75-75	72.52	26.75	32.91	57.87	18.72	17.1	6.3	0.22	0.06
190-75-90	70.56	30.87	36.69	64.48	20.87	16.6	7.3	0.26	0.05
190-75-105	78.11	37.34	48.43	74.42	31.52	18.2	8.7	0.28	0.08
190-75-120	83.01	41.65	58.05	89.93	37.48	18.6	9.4	0.33	0.10

A-3. (continued)

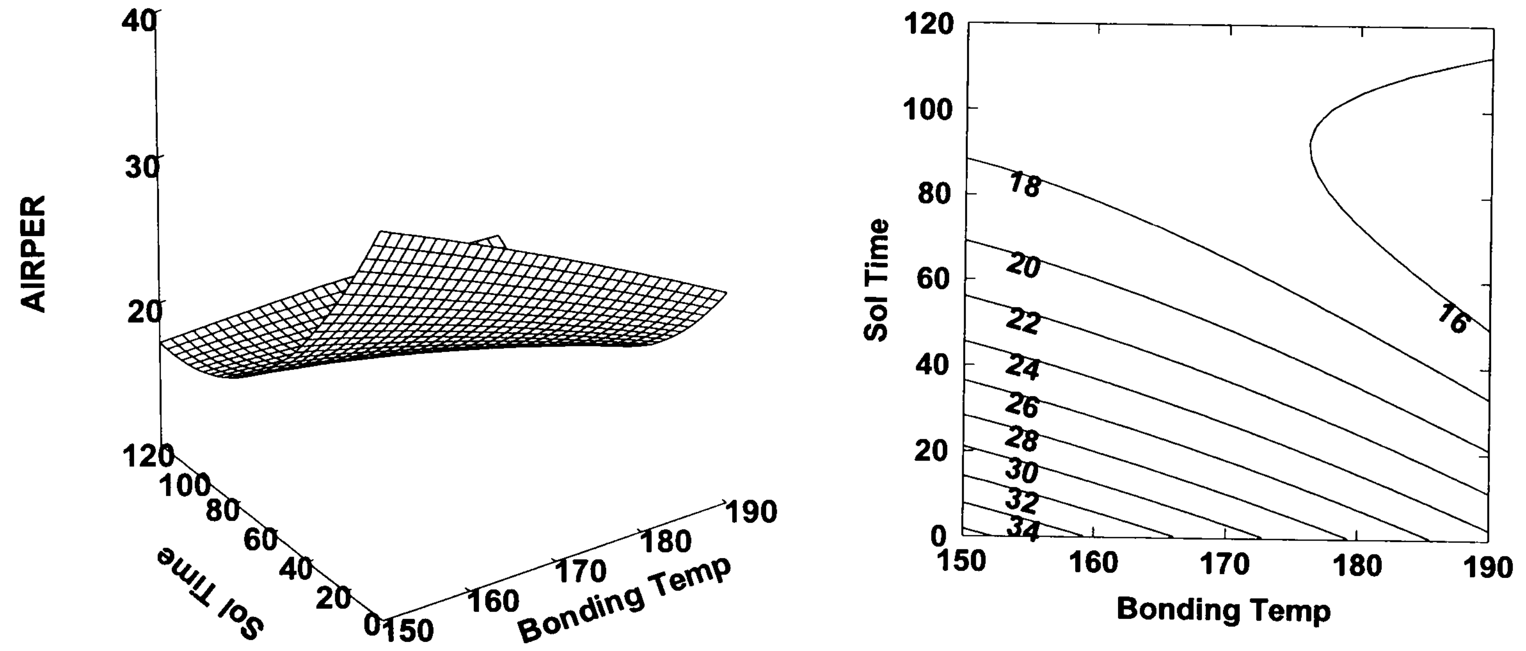
Sample No.	**Strength (N)**		**Overall Flexural Rigidity (G_o) (10^{-5} N·m)**	**Flexural Rigidity (10^{-5} N·m)**		**Yield Stress (N·m/g)**		**Initial Modulus (10^{3} N·m/g)**	
	MD	**CD**		**MD**	**CD**	**MD**	**CD**	**MD**	**CD**
150-85-00	12.94	5.00	15.14	22.99	10.59	3.1	1.2	0.10	0.03
150-85-30	32.63	11.76	24.19	38.97	15.01	7.8	2.8	0.14	0.04
150-85-45	33.22	10.49	27.00	44.24	16.47	7.9	2.5	0.14	0.04
150-85-60	30.38	11.27	22.86	41.36	12.63	6.0	2.7	0.13	0.03
150-85-75	33.03	11.37	22.72	36.90	13.98	7.7	3.0	0.11	0.04
150-85-90	30.28	8.82	21.86	36.46	13.11	7.2	2.1	0.13	0.03
150-85-105	41.16	15.19	29.60	47.01	18.78	9.3	3.6	0.14	0.04
150-85-120	37.53	13.72	24.55	43.25	13.88	8.8	3.2	0.14	0.04
170-85-00	18.13	5.78	17.47	31.06	9.83	4.5	1.4	0.13	0.02
170-85-30	49.39	19.11	28.08	46.81	16.85	12.0	4.6	0.19	0.04
170-85-45	52.04	21.85	32.56	51.78	20.47	12.3	5.2	0.20	0.05
170-85-60	44.98	14.11	27.80	46.97	16.45	11.1	3.4	0.20	0.04
170-85-75	46.45	17.84	34.62	56.27	21.54	11.0	4.2	0.19	0.05
170-85-90	39.00	13.13	28.90	47.53	17.57	9.3	3.1	0.17	0.04
170-85-105	36.85	16.17	27.71	45.68	16.82	8.8	3.9	0.16	0.04
170-85-120	42.24	15.68	29.68	48.21	18.29	10.1	3.7	0.18	0.05

A-3. (continued)

Sample No.	**Strength (N)**		**Overall Flexural Rigidity (G_o) (10^{-5} N·m)**	**Flexural Rigidity (10^{-5} N·m)**		**Yield Stress (N·m/g)**		**Initial Modulus (10^{3} N·m/g)**	
	MD	**CD**		**MD**	**CD**	**MD**	**CD**	**MD**	**CD**
190-85-00	12.74	6.37	17.65	23.61	13.19	3.2	1.6	0.10	0.03
190-85-30	41.36	16.17	27.29	45.52	16.37	10.1	4.0	0.17	0.04
190-85-45	47.04	16.46	29.02	52.39	16.07	10.6	3.9	0.18	0.04
190-85-60	44.88	14.99	36.70	61.82	21.80	10.6	3.6	0.20	0.04
190-85-75	42.63	15.19	25.96	43.52	15.48	10.3	3.7	0.18	0.05
190-85-90	46.45	17.64	29.25	51.19	16.71	10.7	4.3	0.17	0.05
190-85-105	48.22	17.64	34.14	61.66	18.91	11.2	4.2	0.20	0.05
190-85-120	45.57	19.50	38.73	64.29	23.33	10.3	4.4	0.19	0.05
****E150-75-00**	8.53	3.43	8.74	16.47	4.82	2.2	0.9	0.07	0.02
E150-75-30	31.16	9.80	19.00	35.04	10.31	7.6	2.4	0.12	0.03
E150-50-45	55.96	16.37	17.00	28.33	10.20	13.2	3.9	0.15	0.03
E150-85-60	29.50	11.07	20.26	33.61	12.20	7.3	2.7	0.10	0.04
E150-50-75	73.01	21.17	24.73	38.69	15.80	17.0	4.9	0.19	0.04
E150-75-90	47.04	16.86	30.14	46.43	19.56	11.0	3.8	0.17	0.04
E150-85-105	42.92	15.78	38.12	69.12	21.03	9.7	3.5	0.20	0.04
E150-50-120	90.36	42.14	40.81	62.17	26.79	19.8	9.2	0.26	0.08

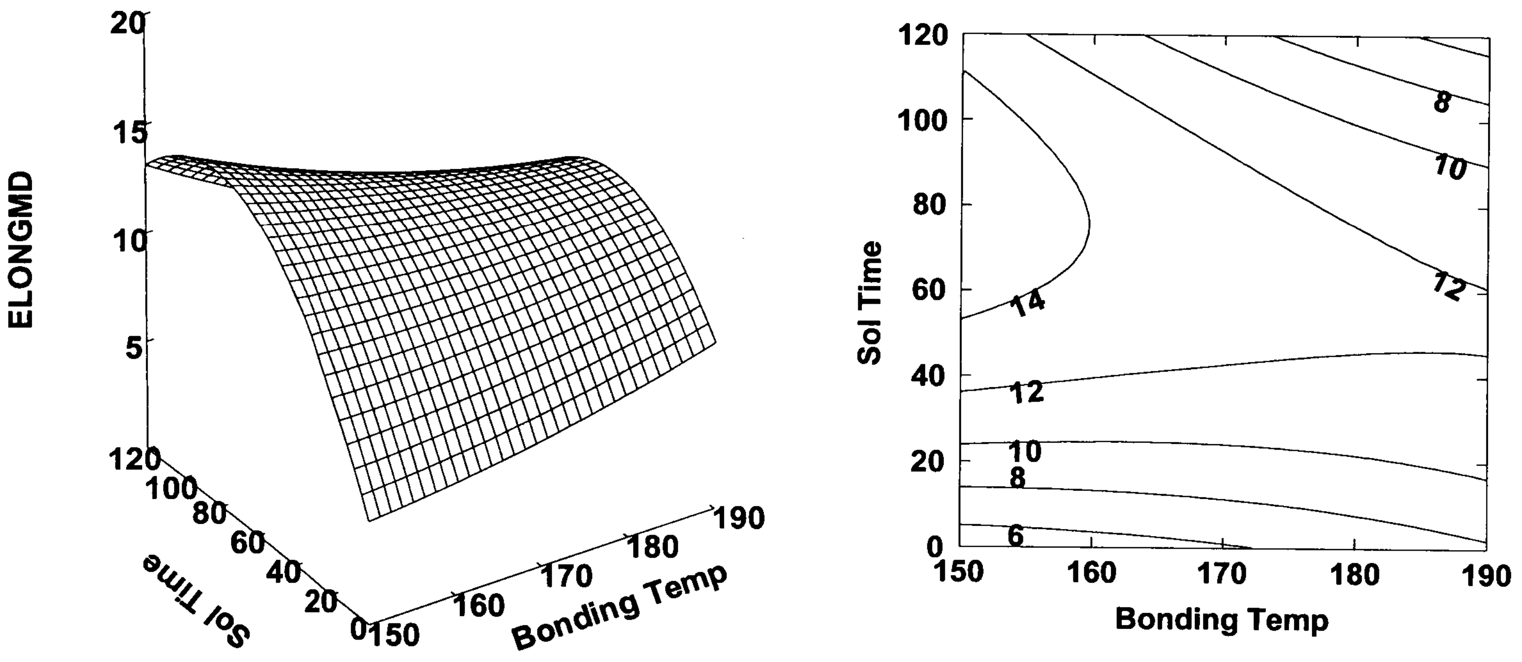
A-3. (continued)

Sample No.	**Strength (N)**		**Overall Flexural Rigidity (G_o) (10^{-5} N·m)**	**Flexural Rigidity (10^{-5} N·m)**		**Yield Stress (N·m/g)**		**Initial Modulus (10^3 N·m/g)**	
	MD	**CD**		**MD**	**CD**	**MD**	**CD**	**MD**	**CD**
E170-50-00	4.41	2.06	5.21	8.33	3.26	1.2	0.5	0.03	0.01
E170-75-30	46.45	14.90	27.35	47.80	15.65	11.0	3.5	0.17	0.04
E170-75-45	49.69	16.86	27.20	48.24	15.35	11.8	4.0	0.17	0.04
E170-85-60	38.02	12.45	29.90	48.98	18.26	8.9	2.9	0.16	0.04
E170-85-75	37.93	21.17	34.80	54.57	22.20	8.8	4.9	0.17	0.05
E170-50-90	83.01	28.52	24.93	45.28	13.73	19.6	6.7	0.27	0.05
E170-50-105	88.98	33.71	31.76	54.29	18.58	20.4	7.7	0.30	0.07
E170-85-120	43.71	13.82	36.18	59.36	22.05	10.1	3.2	0.19	0.05
E190-50-00	9.11	3.53	9.79	14.51	6.60	2.3	0.9	0.05	0.01
E190-85-30	41.65	12.25	31.40	51.53	19.13	9.7	2.9	0.17	0.05
E190-85-45	29.11	10.34	24.79	41.39	14.86	7.0	2.5	0.14	0.03
E190-75-60	53.51	20.48	31.65	57.48	17.42	12.5	4.9	0.19	0.04
E190-85-75	42.14	13.43	35.14	63.39	19.49	9.7	3.1	0.19	0.05
E190-75-90	65.27	25.38	33.52	53.15	21.22	14.8	5.8	0.22	0.05
E190-50-105	90.94	37.93	38.81	63.41	23.75	20.6	8.6	0.34	0.09
E190-75-120	63.21	25.48	36.81	64.80	20.90	14.0	5.9	0.21	0.05


Response Surface of Air Permeability for 50/50 Cotton/CA

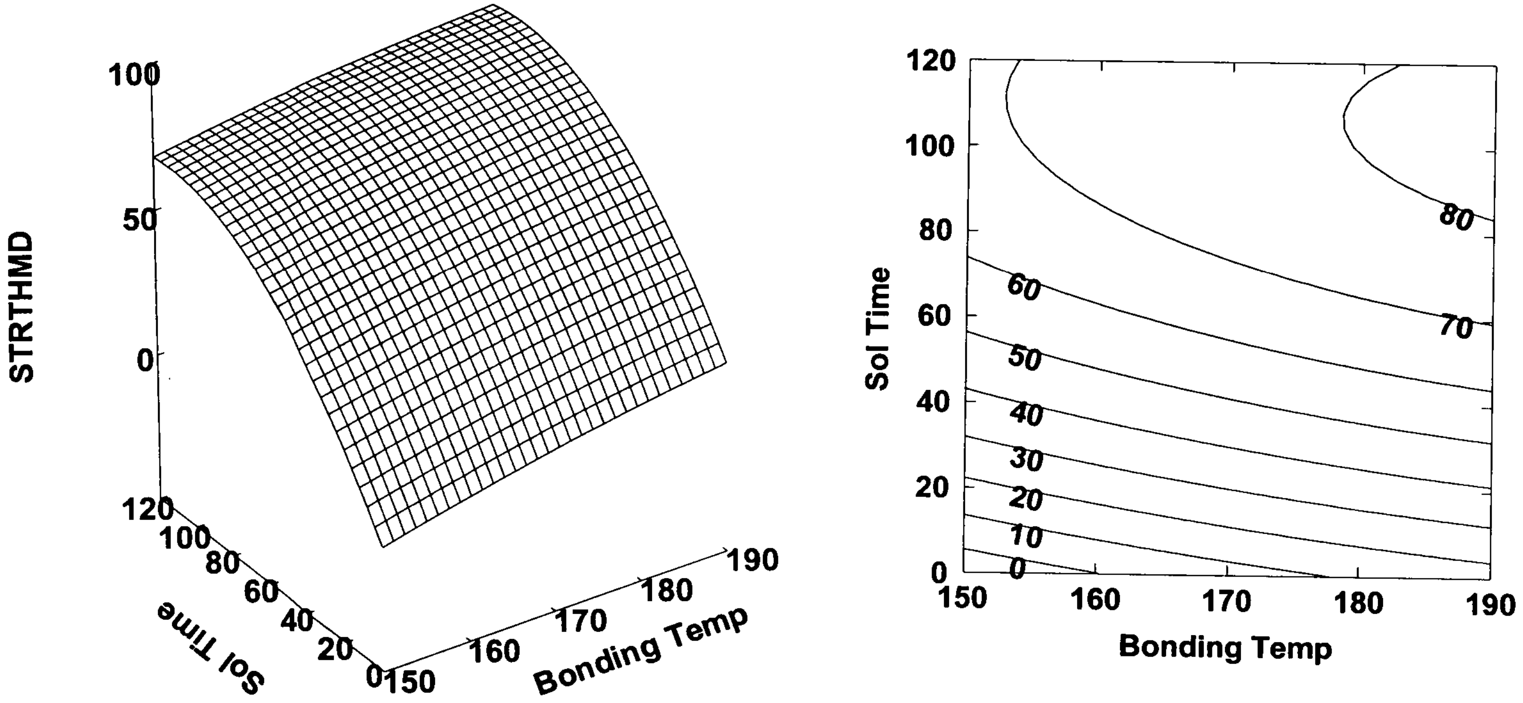
A-4. 3-D and Contour Plots of Four Physical Properties by Blend Ratio.

Response Surface of MD Elongation for 50/50 Cotton/CA



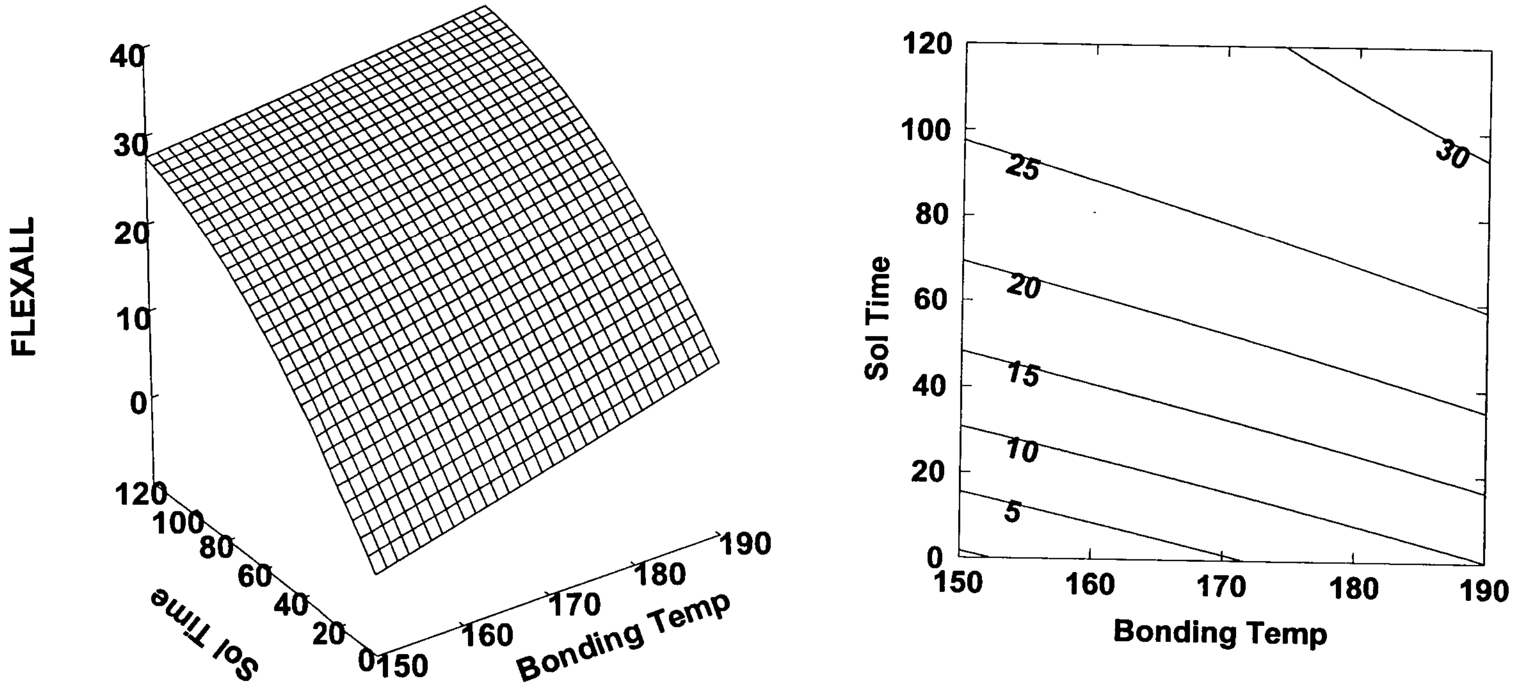
A-4. (continued)

Response Surface of MD Strength for 50/50 Cotton/CA



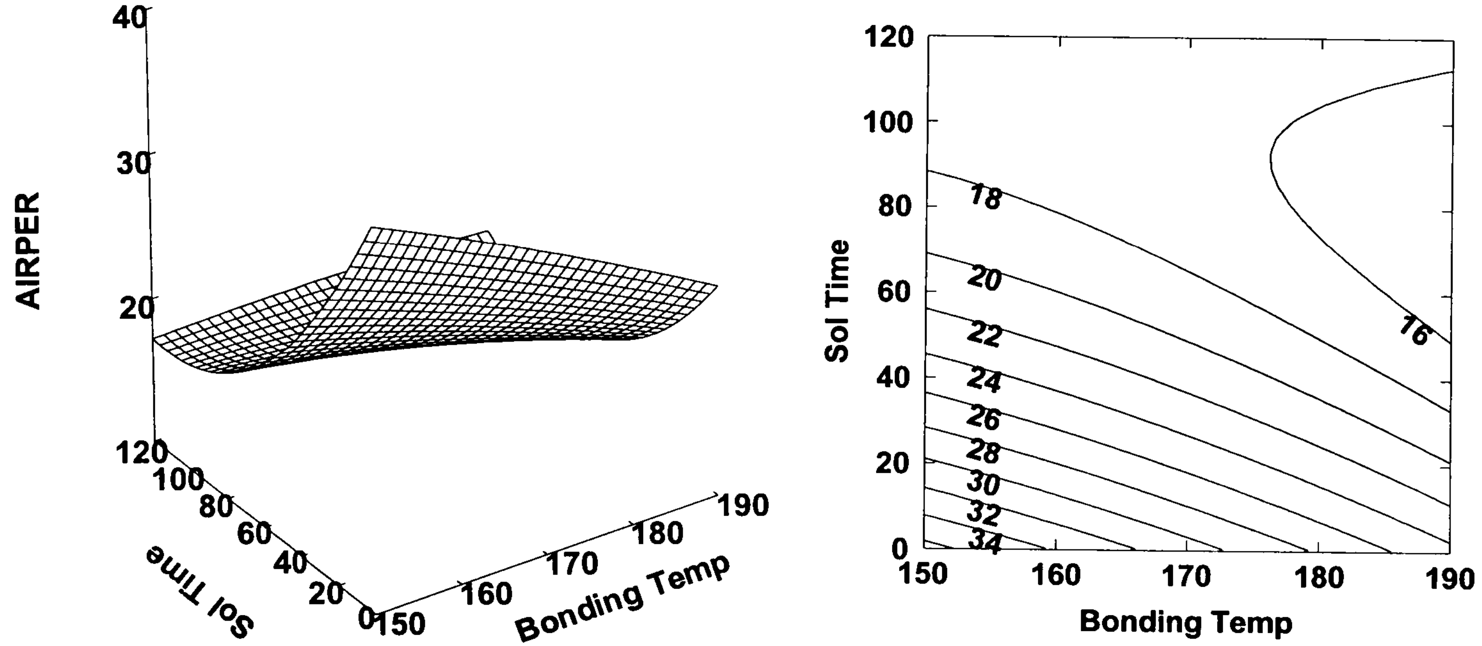
A-4. (continued)

Response Surface of Overall Flexural Rigidity for 50/50 Cotton/CA



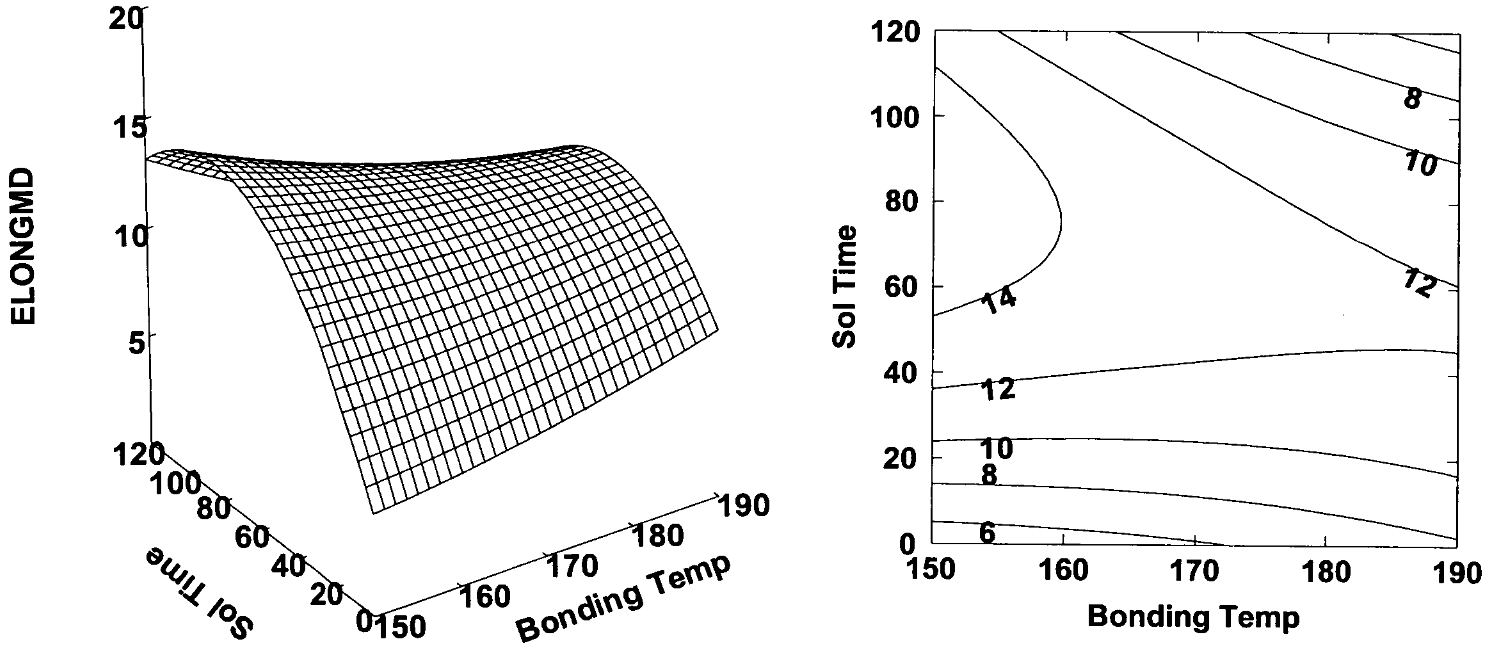
A-4. (continued)

Response Surface of Air Permeability for 75/25 Cotton/CA



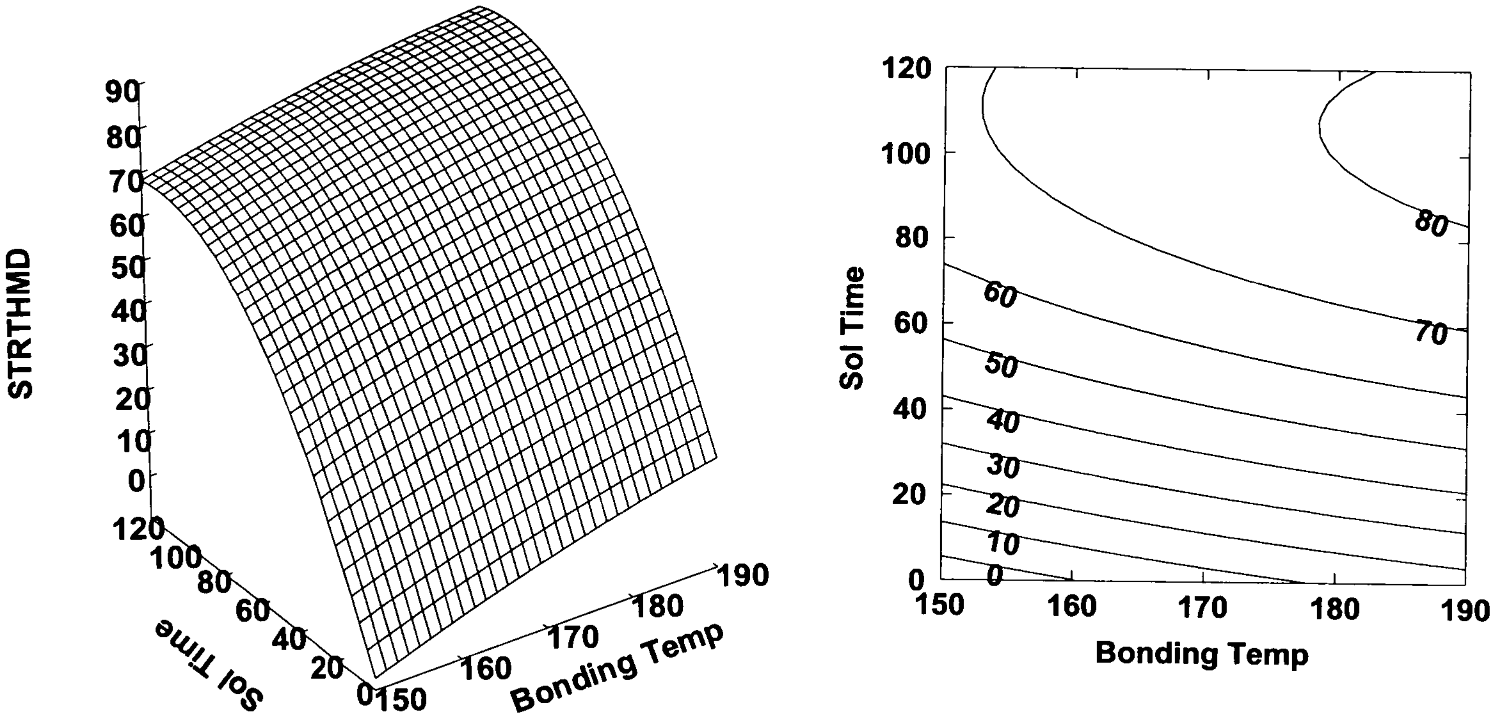
A-4. (continued)

Response Surface of MD Elongation for 75/25 Cotton/CA



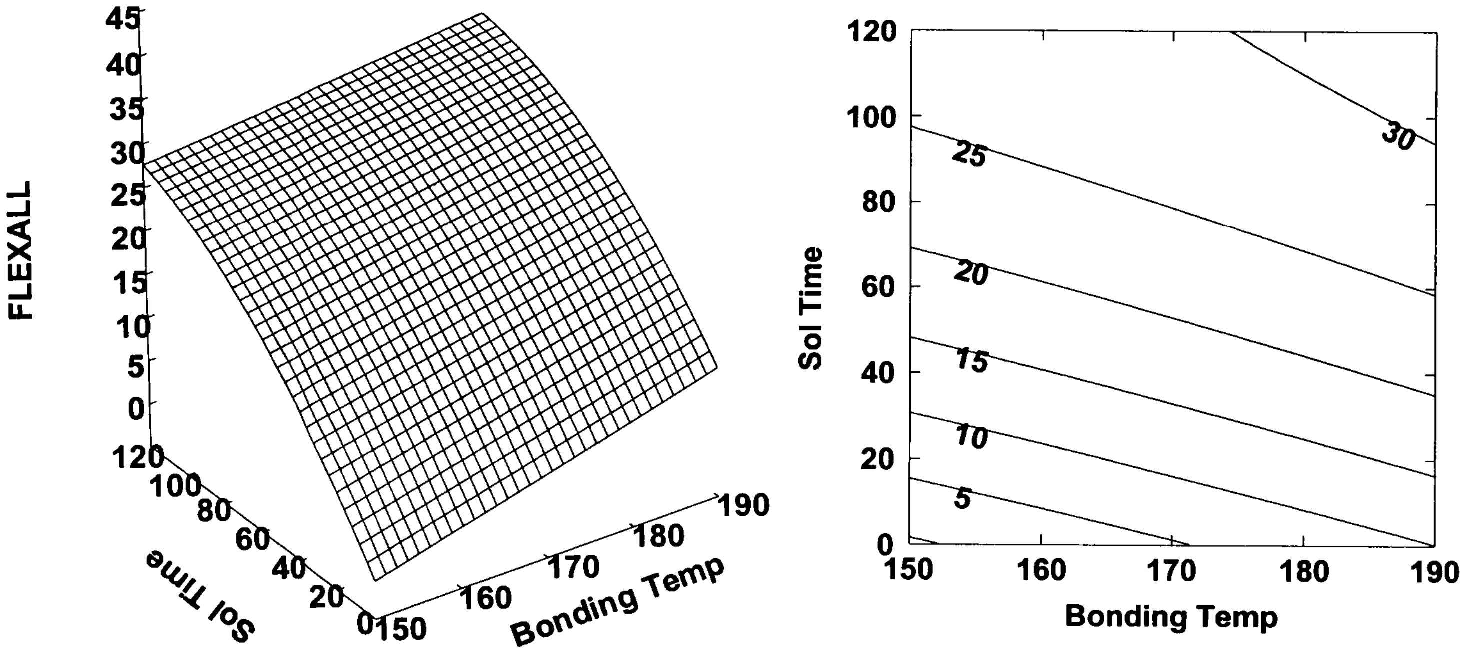
A-4. (continued)

Response Surface of MD Strength for 75/25 Cotton/CA



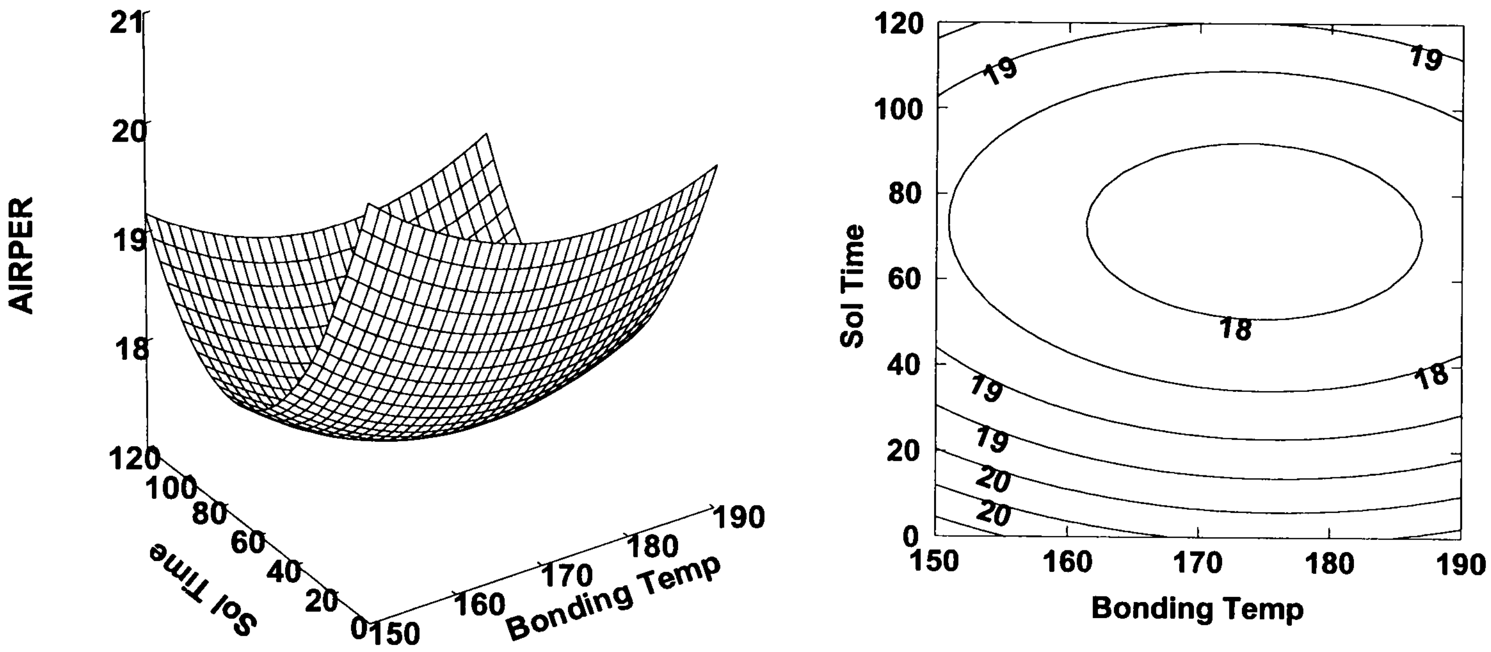
A-4. (continued)

Response Surface of Overall Flexural Rigidity for 75/25 Cotton/CA



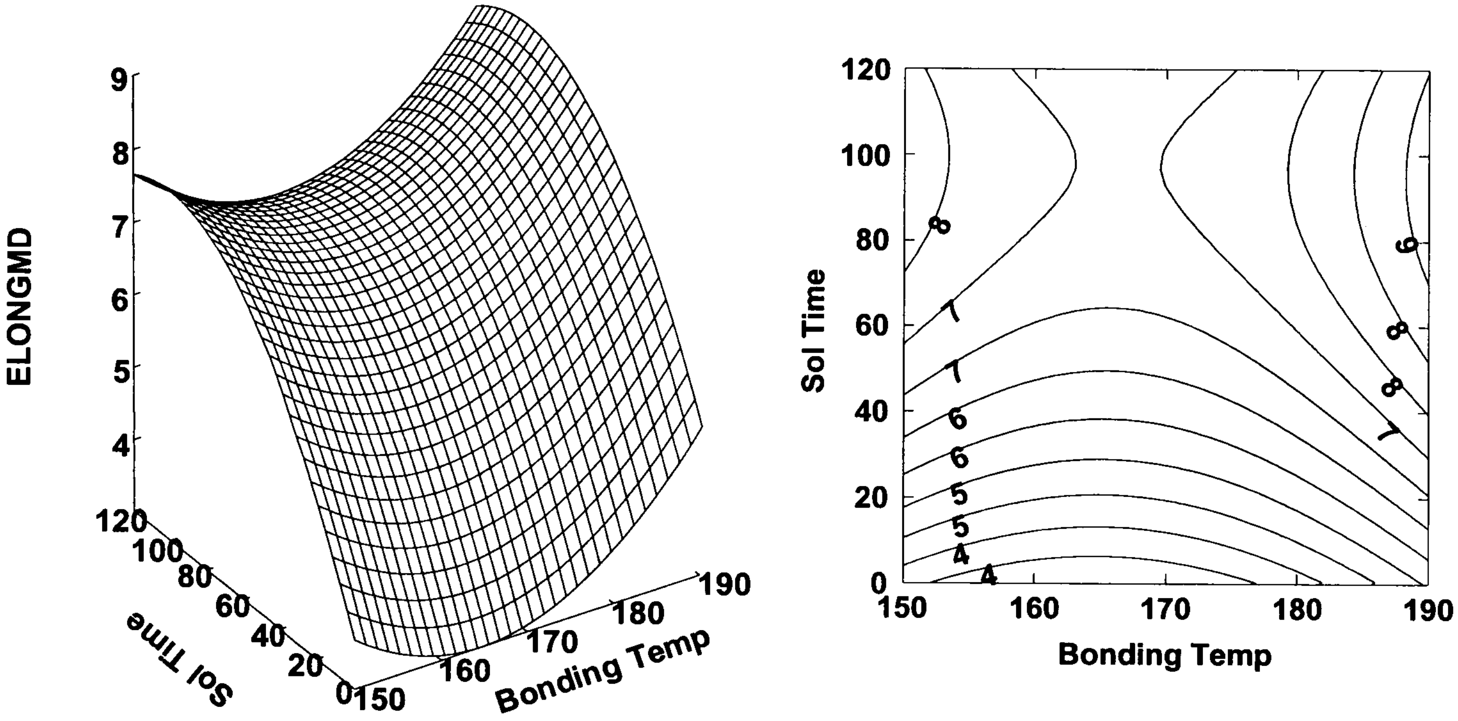
A-4. (continued)

Response Surface of Air Permeability for 85/15 Cotton/CA



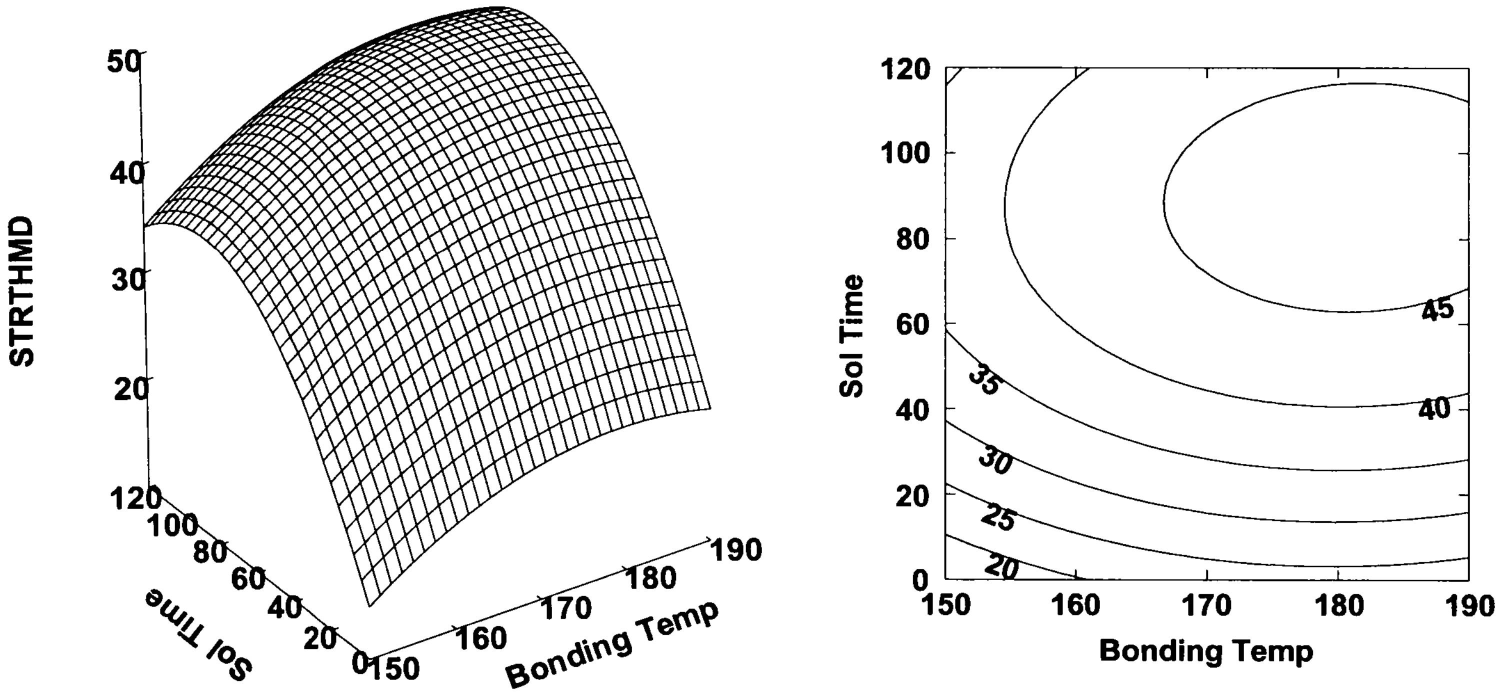
A-4. (continued)

Response Surface of MD Elongation for 85/15 Cotton/CA



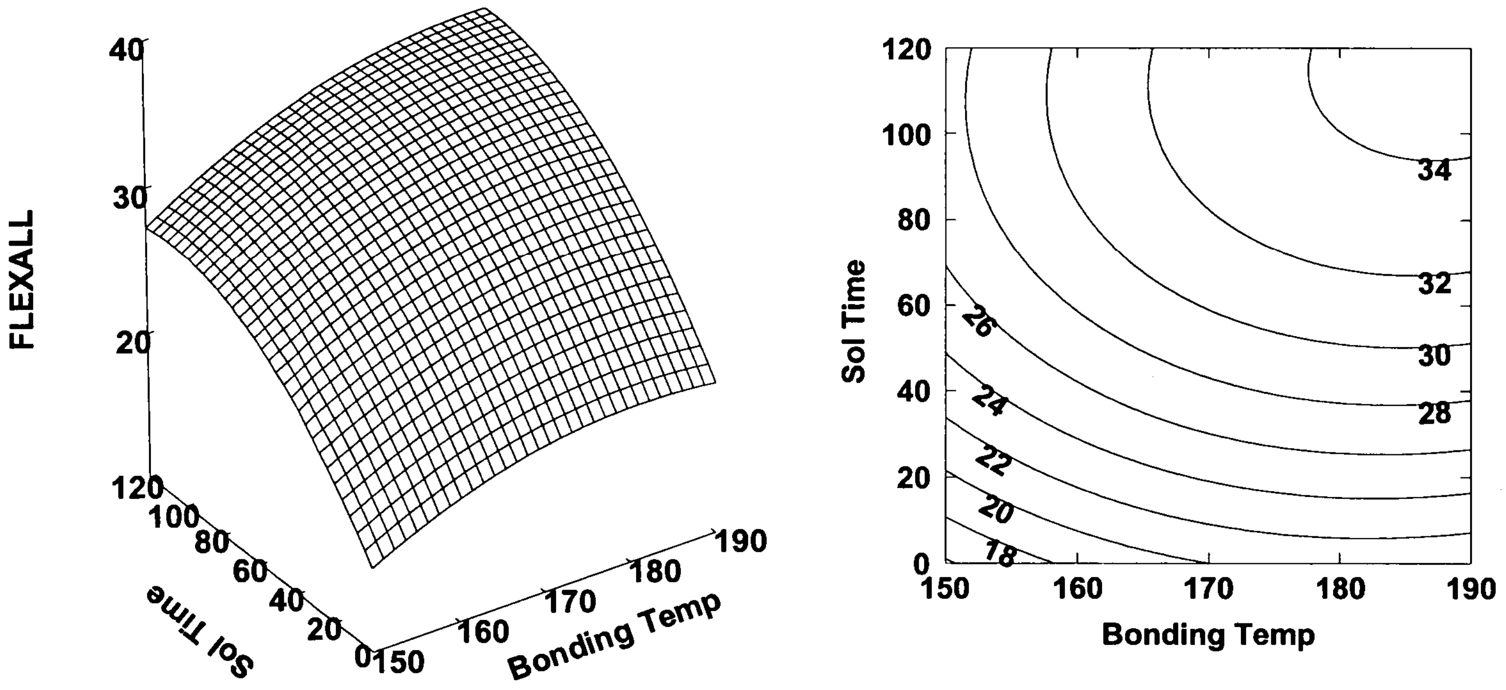
A-4. (continued)

Response Surface of MD Strength for 85/15 Cotton/CA



A-4. (continued)

Response Surface of Overall Flexural Rigidity for 85/15 Cotton/CA



A-4. (continued)

VITA

Hageun Suh was born and raised in South Korea. In 1987 she received her Bachelor of Science in Clothing and Textiles with an emphasis on Textile Science from Yonsei University in Seoul, Korea. Her Bachelor's project was entitled "A Study on Water-Proof Polyamide Fabrics." She came to the USA, and worked on her Master's degree in textile dyeing and finishing area. In 1991 she received her Master of Science in Textile Science from Kansas State University. She entered the Ph.D. program in Textile Science at the University of Tennessee in August, 1992, and has been working on several projects related to biodegradation, nonwoven processing and nonwoven characterization. In 1996 she received a Chancellor's Extraordinary Professional Promise award from the University of Tennessee.

She is presently working on nonwoven processing and characterization as a Research Associate in Textiles and Nonwovens Development Center (TANDEC) at the University of Tennessee, Knoxville.