

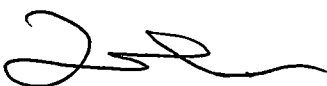
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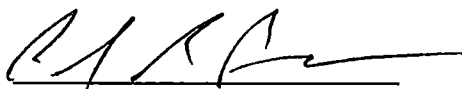
I am submitting herewith a thesis written by Weili Zhao entitled "Fundamental Studies on Poly(Trimethylene Terephthalate) Spunbonded Nonwovens." I have examined the final paper copy of this thesis for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science, with a major in Textiles, Retailing & Consumer Sciences.



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**FUNDAMENTAL STUDIES ON POLY(TRIMETHYLENE
TEREPHTHALATE) SPUNBONDED NONWOVENS**

A THESIS PRESENTED FOR THE

MASTER OF SCIENCE

DEGREE

THE UNIVERSITY OF TENNESSEE, KNOXVILLE

WEILI ZHAO
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DEDICATION

This thesis is dedicated to my beloved husband Zhongren Wang. I would never have achieved this moment without his love, support and encouragement.

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ABSTRACT

Spunbonding is the most productive of all the non-conventional methods of textile fabric formation. Isotactic polypropylene is the most widely used polymer in spunbonding production because it is the least expensive fiber-forming polymer that provides the highest yield. However, the main disadvantage of polypropylene is that it degrades in UV light or gamma radiation. With the increasing demand of the sterilized nonwovens, poly(trimethylene terephthalate) (PTT) has sparked an interest in this application due to its radiation resistance.

In this research, we investigated the fundamental properties of PTT spunbonded nonwovens. The comparisons among PTT, PET and PP spunbonded nonwovens were conducted where appropriate. SEM was applied to examine the web structure. Web uniformity and fiber orientation analysis were conducted to study the web properties.

The results showed that PTT spunbonded nonwovens have better elastic recovery and flexibility compared to PET and PP spunbonded nonwovens. PTT spunbonded nonwovens are relatively thermal stable compared to PET and PP spunbonded nonwovens at high temperature (150°C).

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LIST OF ABBREVIATIONS

1. PTT = Poly (Trimethylene Terephthalate)
2. PET = Poly (ethylene terephthalate)
3. PP = Polypropylene
4. MD = Machine Direction
5. CD = Cross-Machine Direction
6. SEM = Scanning Electron Microscope
7. DSC= Differential Scanning Calorimeter

CHAPTER I

INTRODUCTION

Spunbonding technology is based on melt spinning process that has many similarities. A spunbonded nonwoven fabric has been defined as a fabric formed by filaments that have been extruded, drawn, laid on a belt, and bonded thermally, chemically or mechanically. Spunbonded nonwovens can also be defined generally as continuous-filament fibrous structures that can be made in the form of fabrics, sheets and tapes, and are prepared from synthetic polymers in a process integrated with fiber manufacture [1].

Spunbonding is a unique process that links the innovative polymer, technological and textile research, which involves web formation (extrusion, attenuation and collection of filament bundle), and bonding of web to impart strength, cohesiveness and integrity to the structure. The attenuation of the web may be achieved either by the use of draw-off rolls and subsequently forcing the filament bundle through an aspirator jet with a spreading operation that involves charging the filament bundle with an electrostatic field to deposit the filaments in the form of a web on a conveyor belt, or by drawing filaments into a number of separated sheets with the help of air ducts and then laying the sheets sequentially. Use of supersonic velocity air stream and the movement of air jet from side to side with some mechanical devices have been developed to prepare webs.

Spunbonding system is the most productive of all the non-conventional methods of textile fabric formation [2]. The majority of spunbonded fabrics are made from isotactic

polypropylene and polyester. Some minority of spunbonded fabrics is based on nylon-6,6 and high density polyethylene [3]. Polypropylene is the widely used polymer in spunbonding nonwovens because it is relatively low in cost and easy to extrude. For many applications, it is an ideal candidate. However, the main drawback of polypropylene is that it degrades under UV light or gamma radiation. Moreover, it lacks softness. With the demand of application of sterilized nonwovens, poly(trimethylene terephthalate) has sparked an interest in this application due to its resistance to gamma radiation.

Unique properties of PTT fiber have been reported [4,5,6,7]. It is interesting to know whether PTT spunbonded fabric has some unique properties compared to other traditional spunbonded fabric. In this research, we investigated the fundamental properties of PTT spunbonded web compared to PP and PET webs. Fiber orientation and web uniformity analysis were also conducted to study the web properties.

CHAPTER II

LITERATURE REVIEW

2.1 History of Spunbonding Process

The spunbonding process can be viewed as either an evolution of the existing staple fiber nonwoven production concept or the logical extension of the synthetic filament spinning processes commercialized during the 1940's and 1950's. The origins of the spunbonding process appear to date to the 1940's, when a process of the spunbonding type was patented by Slather and Thomas of Corning Company to produce glass wool [8]. Similar processes for the fabrication of mineral wool from molten siliceous were patented by Calendar in 1945 [9]. Commercially manufactured spunbond nonwovens of synthetic polymers are generally based on the technology patented by DuPont of the U.S. in the 1960's and by Freudenberg of West Germany in the 1950's. DuPont commercialized a product called Reemay[®] in 1950. This was followed by polypropylene Typar[®] and the flash spun polyethylene Tyvek[®] [10].

Freudenberg introduced a spunbonding process called Lutrivil[®] for the manufacture of mixed polyamides in 1965 [11]. DuPont developed the solution flash-spun system in the later 1960s [15]. In 1970, the Lurgi Kohle & Mineral-oltechnik GmbH of Germany introduced their spunbonding process called Docan[®]. In 1984, Reifenhäuser GmbH of Germany introduced the spunbonding process called Reicofil[®] [10].

There is a growing body of patent literature related to spunbonding [12,13]. The summaries can be found in Textile Progress [14] and in International Nonwoven Bulletin [15]. Most of the literature relates to the production processes and end uses of spunbonded nonwoven structures.

2.2 Process Description

Spunbonded fabrics are produced by depositing extruded, spun filaments onto a collecting belt in a uniform random manner, followed by bonding the fibers. The fibers are separated during the web-laying process by air jets or electrostatic charges. The collecting surface is usually perforated to prevent the air stream from deflecting and carrying the fibers in an uncontrolled manner. Bonding imparts strength and integrity to the web by applying heated rolls, embossing rolls, or hot needles to melt the polymer partially and fuse the fibers. Since orientation increases the melting point, fibers that are not highly drawn can be used as thermal bonding sites. Polyethylene or random ethylene-propylene copolymers are used as low melting bonding sites. Since the fabric production is combined with fiber production, the process is generally more economical than when using staple fiber to make nonwoven fabrics [16].

The spunbonding process is controlled by four simultaneous integrated operations: filament extrusion, drawing, lay down, and bonding. The first three operations are directly adapted from common fiber spinning and constitute the spun and web formation phase of the process, while the bonding process is the web consolidation or bond phase of the process, hence the generic term spunbond [17]. The spunbonding process begins with

a polymer resin and ends with a finished fabric. The spunbonded webs are made on an integrated and continuous production line. Generally, a spunbond line consists of the following elements: extruder for forming filaments, metering pump, die assembly, filament spinning, drawing and deposition system, collecting belt, bonding zone, and winding unit. A simple generic flow diagram of the spunbonding process is shown in Fig 2.1 [15].

The polymers (pellets or granules) are fed into the extruder hopper. As the polymer moves along the barrel of the extruder, it melts by heating, friction of viscous flow and mechanical action. The pressurized molten polymer is then conveyed to the metering pump, which is a positive displacement constant volume device for uniform melt delivery to the die assembly. The molten polymer from the gear pump goes to the feed distribution system to provide uniform flow to the die block assembly. The die assembly is one of the most important elements of the spunbonding process. The die assembly consists of polymer feed distribution and spinnerette. The feed distribution in a spunbonding die is more critical than in a film or sheeting die, which is usually designed in such a way that the polymer distribution is less dependent on the shear sensitivity of the polymer. The feed distribution balances both the flow and the residence time across the width of the die. From the feed distribution channel, the polymer melt goes directly to the spinneret. A spinneret is a single block of metal having several thousand orifices, which is usually circular or rectangular in shape. The grouping of spinnerettes is often called a block or bank. In commercial production lines, two or more blocks are used at random to increase the coverage of the fibers [15].

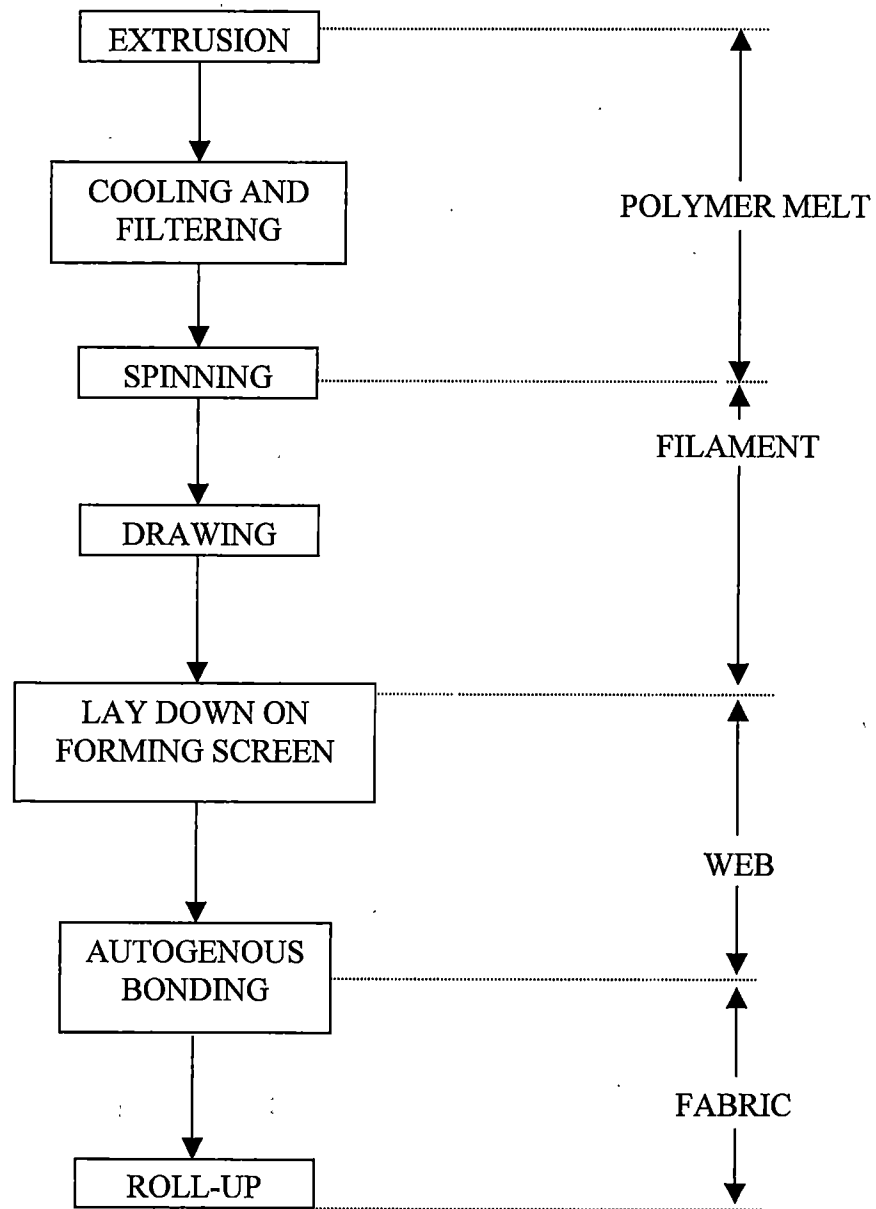


Fig 2.1 A Generic Flow Diagram of the Spunbond Process [15]

The filament spinning, drawing, and deposition are the most critical steps in the spunbonding process. There are three spinning techniques used in the spunbonding process. They are melt spinning, dry spinning, and wet spinning.

Melt spinning is a process in which the fiber-forming polymer is melted and extruded into air or other gas, where it is cooled and solidified. Melt spinning is used in the manufacture of olefins, polyester and nylon.

Dry spinning is a process in which a solution of the fiber-forming polymer is extruded in a continuous stream into a heated chamber to remove the solvent, leaving the solid filament. Dry spinning is used in the manufacture of acetate.

Wet spinning is a process in which a solution of the fiber-forming polymer is extruded into a liquid coagulating medium where the polymer is regenerated. Wet spinning is used in the manufacture of viscose or cupra-amonium rayon.

Any of the above spinning techniques can be used in the spunbond process. However, the melt spinning is widely used mainly due to its simplicity and economics. The drawing of the filaments follows spinning, which is achieved using a specially designed aerodynamic device. The filament deposition follows drawing and is achieved by an aerodynamic device, which is referred to as a fanning or entangler unit.

A number of spunbond processes can be fitted into one of these four routes with appropriate modification. The following are four successful spinning, drawing, and deposition systems which merit a brief discussion.

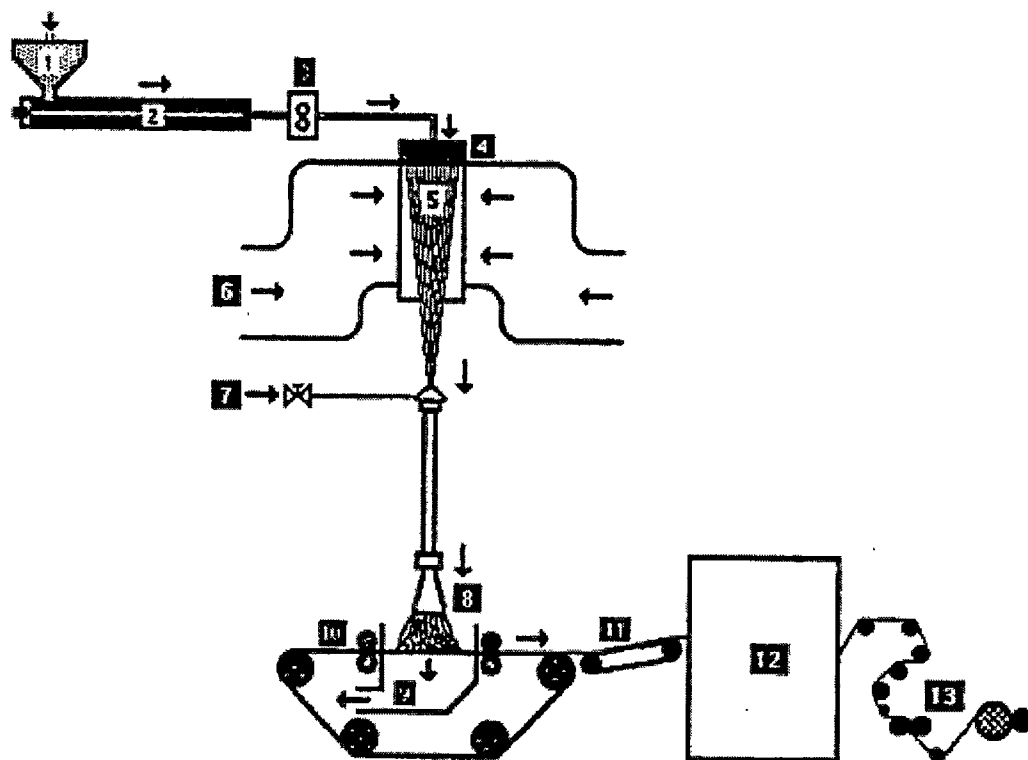
Docan System

Docan system was first developed by the Lurgi Kohle & Mineral-Oftechnik GmbH of

Germany in 1970. Many nonwoven companies have licensed this route from the Lurgi Corporation for commercial production. This route (Fig 2.2) is based on the melt spinning technique. The melt is forced by spin pumps through special spinnerets having a large number of holes. By suitable choice of extrusion and spinning conditions, the desired filament denier is attained. The blow ducts located below individual spinnerets continuously cool the filaments with conditioned air. The force required for filament drawing and orientation is produced by a special aerodynamic system. Each continuous filament bundle is picked up by a draw-off jet operated on high-pressure air and passed through a guide tube to a separator which effects separation and fanning of the filaments [18]. Finally, the filament fan leaving the separators is deposited as a random web on a moving sieve belt. The suction below the sieve belt enhances the random lay down of the filaments [15].

Reicofil System

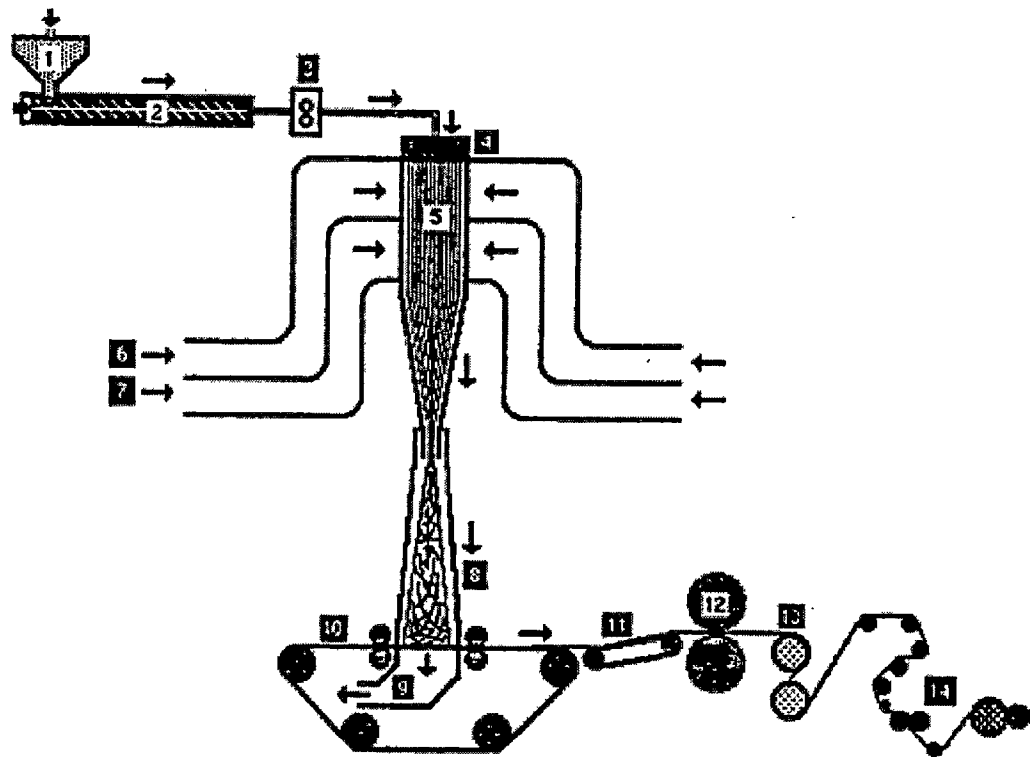
The Reicofil system was developed by Reifenhäuser GmbH of Germany in 1984. Many nonwovens companies have licensed this route from the Reiferihäuser GmbH for commercial production. This route (Fig 2.3) is based on the melt spinning technique. The melt is forced by spin pumps through special spinnerets having a large number of holes. The primary blow ducts, located below the spinneret block, continuously cool the filaments with conditioned air. The secondary blow ducts, located below the primary blow ducts, continuously supply the auxiliary room temperature air. Over the line's entire working width, ventilator generated under-pressure sucks filaments and mixed air down from the spinnerets and cooling chambers. The continuous filaments are sucked through a



Schematic of "Docan" spunbonding system

- | | |
|----------------------------------|--------------------------------|
| 1 hopper | 8 draw-off and lay-down system |
| 2 extruder | 9 suction blower |
| 3 spin pump | 10 spin belt |
| 4 die block | 11 guide belt |
| 5 cooling and stretching chamber | 12 chosen bonding system |
| 6 cool air blower | 13 winding unit |
| 7 operating air | |

Fig 2.2 Schematic of Docan Spunbonding System



Schematic of "Reicofil" spunbonding system

- | | |
|----------------------------------|---------------------|
| 1 hopper | 9 suction blower |
| 2 extruder | 10 spin belt |
| 3 spin pump | 11 guide belt |
| 4 die block | 12 thermal calender |
| 5 cooling and stretching chamber | 13 chill roll |
| 6 cool air blower | 14 winding unit |
| 7 auxiliary air blower | |
| 8 filament entangler | |

Fig 2.3 Schematic of Reicofil Spunbonding System

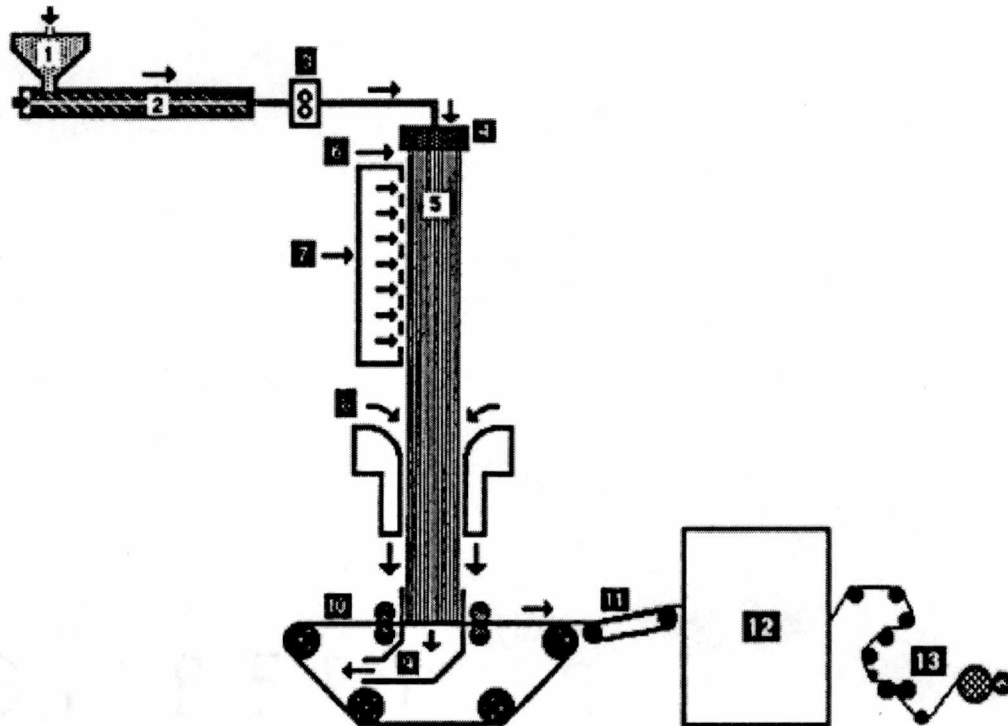
venturi (high velocity, low pressure zone) to a distributing chamber, which affects fanning and entanglement of the filaments. Finally, the entangled filaments are deposited as a random web on a moving sieve belt. The randomness is imparted by the turbulence in the air stream, but there is a small bias in the machine direction due to some directionality imparted by the moving belt. The suction below the sieve belt enhances the random lay down of the filaments [15].

Lutrail System

The Lutrail System was first developed by Carl Freudenberg Company of Germany in 1965. This process is proprietary and is not available for commercial licensing. This route (Fig 2.4) is based on the melt spinning technique. The melt is forced by spin pumps through special spinnerets having a large number of holes. The primary blow ducts, located below the spinneret block, continuously cool the filaments with conditioned air. The secondary blow ducts, located below the primary blow ducts, continuously supply controlled room-temperature air. The filaments are passed through a special device, where high pressure tertiary air draws and orients the filaments. Finally, the filaments are deposited as a random web on a moving sieve belt [15].

Solution Flash-spun System

Solution flash-spun system was developed by the DuPont Co. USA in the late 1960s. This process is proprietary and is not available for commercial licensing. Flash spinning is an alternative technique for the conversion of fiber-forming polymers into spunbond webs using dry spinning technique. In this process (Fig 2.5), a polymer (typically



Schematic of "Lutrasil*" spunbonding system

- | | |
|----------------------------------|-----------------------------|
| 1 hopper | 8 tertiary drawing air jets |
| 2 extruder | 9 suction blower |
| 3 spin pump | 10 spin belt |
| 4 die block | 11 guide belt |
| 5 cooling and stretching chamber | 12 bonding unit |
| 6 primary air | 13 winding unit |
| 7 secondary cooling air | |

Fig 2.4 Schematic of Lutrasil Spunbonding System

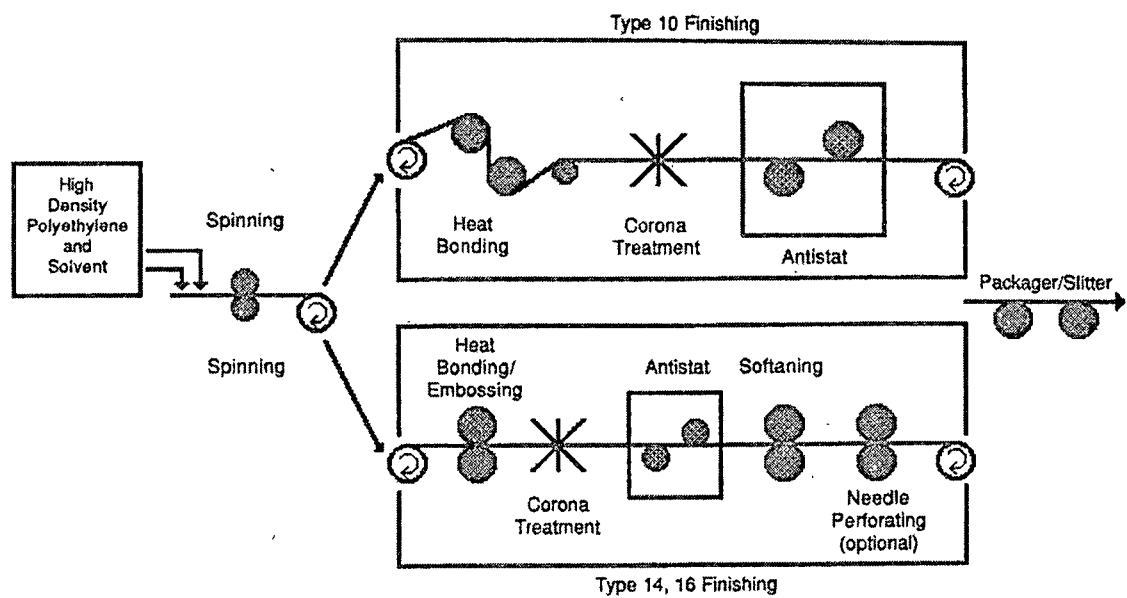


Fig 2.5 Schematic of Flash Spun (Tyvek) Spunbonding System

polyethylene) is blended with a solvent (typically methylene chloride) under high temperature (about 25°C or more above the boiling point of solvent) and high pressure.

Then the blended solution is released under controlled conditions to produce a three-dimensional network of thin, continuous interconnected ribbons that are less than 4 μm thick. The fibrous networks are usually termed as film-fibrils or plexifilaments [19]. Sometimes, dissolved inert gas, e.g., CO_2 , is used to increase the degree of fibrillation [20]. The fibrous network is collected on a moving belt and consolidated. The individual fibers have high molecular orientation, leading to high fiber strength. A product example of this process is Tyvek® (by DuPont).

Flash spinning is the most complex and difficult method of manufacturing spunbonded fabrics because of the need to spin a heated pressurized solution under precise conditions [21].

The bonding process imparts strength, cohesiveness and integrity to the structure. There are three basic bonding methods in the spunbonding processes: thermal (calendaring), chemical (adhesive), and mechanical (needle punching or hydroentangling). The choice of a particular bonding technique is determined mainly by the ultimate fabric application. Sometimes, a combination of two or more techniques is employed to achieve bonding.

Thermal bonding is the most widely used technique in spunbonding processes. This bonding is achieved by fusing of the filaments in the web at the crossover sections. The fusion is obtained by direct action of heat and pressure via a calendar or an oven. The degree of fusion determines most qualities of the web, such as hand and softness. Normally, the thermal bonding can be achieved in combining a filament of the same

material but of different softening point, or distributing different material in powder form through the web, or using bicomponent filaments in which the bonding agent forms a thin sheath around each web filament or is present as an integral sector of the filament cross-section [15].

Chemical bonding (adhesive bonding) is achieved using polymer latex or a polymer solution deposited in and around the fibrous structure and then cured thermally to get bonding. In spray bonding, the bonding agent usually stays close to the surface of the material, resulting in a web with little strength, high bulk, and a fair degree of openness. In case of saturated bonding, all the fibers are bonded to each other in a continuous matrix so that the rigidity, stiffness, and thinness of the web increase. In case of partial solvation bonding, the overall bonding is obtained using gaseous hydrogen chloride. The gaseous HCl solvates the outer surface of the carrier fiber and bonds the fibers together at all fiber cross-over points by welding together the contacting solvated surface. The removal or neutralization of the solvent is the final step in bond formation [22].

Mechanical bonding (needle punching) is achieved by entangling the fibers using barded needles, which are set into a board and penetrate into the web and then recede, leaving the fibers entangled. Needle configuration, needle length, barb shape, and web advance rate can influence the degree of fibrous entanglement. The needle punched webs are quite extensive, bulky, and comfortable. Needle punching is suitable for heavier spunbonded webs [15].

There have been numerous empirical studies dealing with the mechanical and physical properties of commercial products and the suitability and performance of spunbonded

fabrics. Spunbonded fabrics exhibit extremely high tensile and tear strengths compared with conventional woven, or adhesive-bonded nonwoven, fabrics. The mechanical properties of the spunbonded fabrics depend on filament properties, bonding parameters, and filament arrangement. Filament properties include linear density, tenacity, modulus, elongation, crimp, cross-section, and fiber morphology. Bonding parameters include binder type, binder concentration, binder distribution, and self-bonding. Filament arrangement includes fabric weight uniformity, filament separation, and orientation. The understanding and prediction of the behavior of filaments during melt spinning and the pattern of laying down during fabric formation require the consideration of aerodynamics and the electrostatic and mechanical forces acting on spinline [23].

2.3 Polymers Used in Spunbonding Process

Since the spunbonding process begins with the filament spinning, the most convenient fiber materials that have been commercially used are those of thermoplastic polymers.

In general, high molecular weight and broad molecular weight distribution polymers can be used by the spunbonding process. These include PP, PET and Polyamide. Some medium melt-viscosity polymers used for production of fibers by melt spinning are also used in spunbonding process.

Polypropylene

Isotactic polypropylene is the most widely used polymer for spunbond nonwovens production because it is the least expensive fiber-forming polymer that provides the

highest yield and covering power because of its low density. The main disadvantage of polypropylene is that it degrades in UV light or gamma radiation [3].

Polyethylene

Polyethylene is more expensive than polypropylene and has slightly lower fabric yield and filament strength. The properties of polyethylene fibers that are melt spun by traditional methods are inferior to those of polypropylene fibers. Advances in polyethylene technology may lead to the commercialization of spunbond structures with characteristics not yet attainable with polypropylene. Polyethylene has received only limited acceptance because of higher costs and continuing improvements in polypropylene resin technology.

Polyester

Polyester is used in a number of commercial spunbond products and offers certain advantages over polypropylene although it is less economical. Polyester fabrics are easily dyed and printed with conventional equipment. Tensile strength, modulus, and heat stability of polyester fabrics are superior to those of polypropylene fabrics.

Nylon

Spunbond fabrics are made from both nylon-6, and nylon-6,6. Because nylon is highly energy intensive, it is more expensive than polyester or polypropylene. Nylon-6,6 spunbond fabrics are produced with weights as low as 10 g/m² and with excellent cover and strength. Unlike olefins and polyester fabrics, those made from nylon readily absorb water through hydrogen bonding between the amide group and water molecules.

Polyurethane

The first commercial production of polyurethane fabric was announced in Japan. Polyurethane fabrics are well suited for use in apparel and other applications requiring stretch and recovery.

Rayon

Rayon is the oldest commercial manmade fiber. Many type of rayons have been successfully processed into usable spunbond webs. The main advantage of rayon is that it provides good drape properties and softness to the web [3].

Polymer Combination

Some fabrics are composed of several polymers. A lower melting polymer can function as the binder element which may be a separate fiber interspersed with higher melting fibers, or two polymers may be combined into a single fiber type. In the latter case the fibers are called bi-component or multi-component fibers. The configuration of the bi-component or multi-component fiber can be sheath/core, side-by-side, pie, daisy and islands-in-the sea. Fibers with over 1000 islands have been recently produced [24].

2.4 Applications of Spunbonded Nonwovens

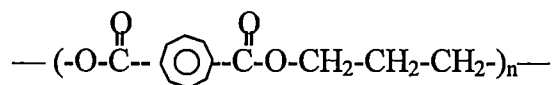
The applications of spunbonded nonwovens range from baby diapers to automotive interiors and from surgical gowns to geotextiles. Today spunbonded nonwovens are used throughout the automobile as a backing for tufted automobile floor carpets, trim parts, trunkliners, interior door panel, and seat covers. Spunbonded civil engineering webs are expanded into a variety of related uses, e.g. erosion control, revetment protection,

railroad bed stabilization, canal and reservoir lining protection, highway and airfield black top cracking prevention, roofing, etc. Spunbonded nonwovens as coverstock are also widely used in sanitary napkins and incontinence devices. The medical applications include disposable operation room gowns, shoe covers, and sterilizable packaging. The examples of packaging materials include: metal-core wrap, medical sterile packaging, floppy disk liner, high performance envelopes and stationery products [23].

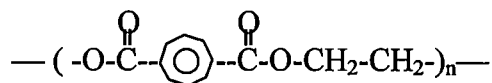
2.5 PTT Spunbonded Nonwovens

PTT was first synthesized by Whinfield and Dickson of the Caligo Printing Ink in 1941. [25]. PTT is an aromatic polymer made by polycondensation reaction of PTA (purified terephthalic acid) and PDO (1,3 propanediol). The properties of PTT polymers are unique when compared to the other aromatic polyester PET (polyethylene terephthalate) and PP (polypropylene). The molecular structure of PTT, PP and PET is shown in Fig 2.6. The key properties of PTT vs. PET and PP are shown in Table 2.1.

PTT:



PET:



PP:

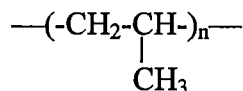


Fig 2.6 Molecular Structures of PTT, PET and PP

Table 2.1 Key Properties of PTT vs. PET and PP

	PTT	PET	PP
Melting Point °C	228	265	168
Glass Transition, °C	45-65	80	-17
Density (g/cm ³)	1.33	1.40	0.91

PTT has many advantages compared to PP and PET. PTT fiber offers resilience and elastic recovery equal to or better than nylon 6,6. While combining the best properties of nylon and polyester, PTT polymer can be extruded on all commonly used machinery with minor modification. Moreover, it is easy to dye and has a remarkable wool-like feel.

Although the desirable properties of PTT have been known for many years, PTT polymer remained an obscure polymer for a long time for the economic reasons. One of the starting raw material, 1,3-propanediol (PDO) is very expensive, which makes commercialization impossible. Shell Chemical Company produced PDO in the 1960's and had sampled various polyester producers to pursue PTT. This led to a brief period of research activities. Finally in the early nineties, after extensive research, Shell Chemical Company developed a cost-effective manufacturing process for PDO. In 1996, Shell Chemical Company began production of PTT polymers to supply fibers including nonwoven fabric [26].

PTT has been spunbonded on a Hills Inc./ASON Technologies pilot line, and on two Reifenhäuser pilot lines. PP spunbond nonwovens lost most of their strength (and are in fact turning to dust) after they were treated by gamma irradiation. The PTT and PET/PTT

spunbond nonwovens do not appear to be affected by radiation [27]. PTT readily forms fine denier fibers at high speeds. The resistance PTT shows to gamma radiation makes PTT fabric an ideal candidate for application needing sterility or radiation resistance [28]. The unique properties of PTT fiber have been explored [4,5,6,7]. It is interesting to know whether PTT spunbond fabric has some unique properties compared to other traditional spunbonded fabrics. In this research, we investigated the fundamental properties of PTT spunbonded nonwoven compared to PET and PP spunbonded nonwovens. In order to understand the similarity and differences among these three spunbonded nonwovens, Web uniformity analysis and fiber orientation analysis were also conducted to study the web properties.

CHAPTER III

EXPERIMENTAL DETAILS

3.1 Sample Description

PTT spunbonded samples were produced on Reicofil[®] 2 spunbonding line at TANDEC and Reicofil[®] 3 spunbonding line at Reifenhäuser, Germany. On the first Reifenhäuser line, the filament spinning speed is about 1500m/min. On the second Reifenhäuser line, the filament spinning speed is about 3000m/min. PP and PET spunbonded samples were collected from Kobelco, Nordson and Reifenhäuser. The description of samples is shown in the Table 3.1.

PTT-538 and PTT-539 were produced on Reicofil[®] 3 spunbonding line but based on the different basis weight. The basis weight of PTT-538 and PTT-539 is about 40g/m² and 20 g/m² respectively. These two samples were relatively uniform and had soft handle.

PTT-2 and PTT-3 were produced on Reicofil[®] 2 spunbonding line based on the similar basis weight. The basis weight of these two samples is about 70 g/m². These two samples somewhat like films due to the relatively high bonding temperature.

PTT-1.3 and PTT-2.1 were produced on Reicofil[®] 2 spunbonding line based on the similar basis weight. The basis weight of these two samples is about 100 g/m². These two samples are more flexible than PTT-2 or PTT-3 due to the relatively low bonding temperature. However, these two samples were less uniform compared to the other samples produced on Reicofil[®] 2 spunbonding line.

Table3.1 Sample Description

Sample ID	Fabric Weight (g/m²)	Polymer Type	Technology
PET-N	14.64	PET	Nordson
PET-K3	20.08	PET	Kobelco
PET-K1	22.68	PET	Kobelco
PP-KA	10.30	PP	Kobelco
PP-N	14.59	PP	Nordson
PP-KB	15.43	PP	Kobelco
PP-R143	17.18	PP	Reicofil [®] 3
PP-R149	18.29	PP	Reicofil [®] 3
PP-KC	20.63	PP	Kobelco
PTT-539	20.26	PTT	Reicofil [®] 3
PTT-538	42.58	PTT	Reicofil [®] 3
PTT-2	69.56	PTT	Reicofil [®] 2
PTT-3	73.36	PTT	Reicofil [®] 2
PTT-1.3	100.82	PTT	Reicofil [®] 2
PTT-2.1	102.85	PTT	Reicofil [®] 2
PTT-14	105.62	PTT	Reicofil [®] 2
PTT-16	103.37	PTT	Reicofil [®] 2

For the spunbonded nonwoven based on the same type polymer, the polymers may be supplied by different vendors for the individual manufacturer. This may cause the difference in the property of the final product. The reason we selected samples from different manufactures is to compare the properties based on various polymers of the same type (eg. PP).

3.2 Experiments Conducted

The experiments conducted include basic weight, fiber diameter, thickness, air permeability, hydrostatic pressure, liquid strike-through-time, stiffness, drape, elastic recovery, tensile strength and break elongation, and heat shrinkage.

Fiber orientation and web uniformity analysis were also conducted using Webpro software developed at TANDEC, University of Tennessee, Knoxville.

3.2.1 Basis Weight and Thickness

The basis weight and thickness of the samples were measured according to ASTM D3773-96 (IST 130.2) and ASTM D5729-97 (IST 120.1) respectively.

The bulk density of the fabric was calculated using the equation:

$$\text{Bulk Density (g/cm}^3\text{)} = \frac{\text{Basis Weight (g / m}^2\text{)}}{1000 \times \text{Thickness (mm)}}$$

3.2.2 Fiber Diameter

The fiber diameter was measured by using the optical microscope with 50X magnification lens. 20 measurements were taken for each spunbonded web because the

fiber diameters of spunbonded web are relatively uniform. The microscope is connected to the software available at TANDEC so that the results can be processed simultaneously while making the measurements.

3.2.3 Air Permeability

Air permeability is the rate of air flow passing perpendicularly through a known area under a prescribed air pressure differential between the two surfaces of a material. Air permeability of the nonwovens was measured according to ASTM D737-96.

3.2.4 Hydrostatic Pressure

The hydrostatic pressure test measures the resistance of nonwoven fabrics to the penetration of water under low hydrostatic pressure. A nonwoven fabric specimen is mounted to form the cover on the test reservoir. This specimen is subjected to a standardized water pressure, increased at a constant rate until leakage appears on the outer surface of the fabric. Water pressure is measured at the hydrostatic head height reached at the first sign of leakage in three separate areas on the specimen. The head height results are recorded in millibars of water pressure on the specimen. A higher value indicates greater resistance to water penetration. TESTEX FX-300 tester was used to measure the hydrostatic pressure according to INDA standard test (IST 80.6 - 98).

3.2.5 Liquid Strike-Through-Time

Liquid strike-through-time is the time taken for a known volume of liquid to pass through the nonwoven test specimen, where the liquid is applied to the nonwoven's surface and the nonwoven is in contact with an underlying standard absorbent pad. The time method for determination of liquid strike-through-time may be used to determine a nonwoven fabric's ability to pass simulated urine through the nonwoven. This test method is designed to compare strike-through-time of nonwoven coverstocks and is not intended to simulate in-use conditions of finished product. Liquid strike-through-time was measured according to INDA standard test: IST 70.3 (01).

3.2.6 Stiffness

The stiffness test was conducted using a cantilever tester according to ASTM D5732-95 (IST90.1). Bending length is a measure of the interaction between fabric weight and fabric stiffness by the way in which a fabric bends under its own weight. Flexural rigidity is a measure of stiffness when the couple on either end of a strip or unit width bent into unit curvature, in the absence of any tension. The lower the flexural rigidity, the softer the fabric is likely to be. The flexural rigidity was calculated using the equation:

$$\text{Flexural rigidity} = \frac{W}{10} \times C^3 \text{ (mg-cm)}$$

Where W: Basis weight, g/m²

C: Bending length, cm

3.2.7 Drape

Drape of a fabric is defined as the degree to which a fabric will deform under its own weight when it is hanged. Fabric drape is a multi-dimensional and visually observed fabric property. It is somewhat analogous to the assessment of bending length, but in three dimensions. The drape coefficient is defined as the percentage of the area of the annular ring of fabric obtained by vertically projecting the shadow of the draped fabric specimen. A single number is obtained whose theoretical range is zero to 100%. A drape meter was used to perform the drape test. The development of the drapemeter and the calculation of the drape coefficient can be found in reference [29] and [30].

In this test, the drape coefficient was measured using the equation:

$$F = \frac{W_3 - W_2}{W_1 - W_2} \times 100$$

Where W_1 : weight of 10 inch diameter tracing paper, mg.

W_2 : weight of 4 inch diameter tracing paper, mg.

W_3 : weight of projected image, cut from tracing paper W_1 , mg.

The smaller the drape coefficient, the more flexible the fabric is.

3.2.8 Elastic Recovery

The elastic recovery of single PTT fiber has been studied [31,32,33]. PTT fiber is generally considered as having good elastic recovery. It is interesting to know whether this translated to PTT spunbond nonwoven webs. This experiment was designed to quantity the elasticity of the spunbonded nonwoven.

A constant-rate-of-extension (CRE) united tensile tester was used to conduct this test. A 1-inch wide specimen was cut using a guillotine and mounted in the pneumatic grips of a united tensile tester. The distance between the two grips was 5 inches. A force is applied to draw the specimen to 10% elongation relative to the length. The rate of extension is 1 inch per minute. On reaching 10% elongation, the specimen was immediately released to its original position and removed from the grips. The distance between the two grips on the specimen was marked before extension and was measured 2 minutes after the specimen returned to its original position. A total of 5 specimens were tested in the machine direction and 8 specimens in the cross-machine direction. The permanent deformation is defined as:

$$\text{Permanent deformation (\%)} = (L - L_0) / L_0 \times 100\%$$

Where L: the distance between the marked lines after the specimen returned to its original position.

L_0 : the distance between the two grips. It is equal to 5 inches in this test.

The elastic recovery is calculated based on the permanent deformation using the equation:

$$\text{Elastic recovery (\%)} = 1 - \text{permanent deformation}$$

3.2.9 Heat Shrinkage

The heat shrinkage test measures the heat dimensional stability of nonwoven fabrics when they are exposed to high temperature. The heat shrinkage test was conducted at different temperatures. The selected temperature for this test was 90°C, 120°C and 150°C

respectively. The sample underwent the default temperature for 2 minutes in the oven. The heat shrinkage is expressed as the area change between before and after heat treatment.

3.2.10 Web Uniformity Analysis

Web uniformity analysis was conducted using Webpro software developed at TANDEC, University of Tennessee, Knoxville. Weight uniformity has traditionally been used as a quality index for nonwoven webs. Physical, mechanical and aesthetic properties of the webs, as well as variation in web performance, are known to be influenced by the uniformity of web structure. It is obviously useful to characterize web uniformity. This software utilizes various measures of optical uniformity and is able to evaluate several different types of structural uniformity. Web uniformity actually analyzes the optical property. However, both theory and experiments showed that web basis weight was linearly related to optical intensity. Optical and weight uniformities were in excellent agreement and required no corrections. Uniformity spectra provide information about web uniformity at different size resolution, and were demonstrated to be useful for providing detailed uniformity information for webs. The CV% of small cell size was associated with microscopic structural uniformity whereas the CV% of large cell size was associated with macroscopic structural uniformity [34]. A total of 256 images were randomly selected to evaluate the web uniformity in this test.

3.2.11 Fiber Orientation Analysis.

Fiber orientation analysis was conducted using the same Webpro software as in the previous test. The spatial distribution of fibers in nonwoven webs is an important structural feature that significantly influences many physical and mechanical properties of webs.

The orientation of a fiber is described by the angle between the fiber path and some arbitrary web direction (e.g. machine direction). The orientation angle of a fiber segment was defined as the angle between the center line of the fiber segment and the machine direction. The sign of this angle was defined as positive if the fiber segment was aligned in the southeast direction and negative if aligned in the southwest direction.

The diameter-based distribution is the sum of fiber diameter aligned in the certain individual direction divided by the total fiber diameter aligned in all directions.

Since web mechanical properties might be expected to be related more directly to fiber diameter than fiber number, and since diameter information is easier to extract from images when extensive entanglement exists, the orientation distribution of fibers was characterized in terms of diameter-based rather than number-based distribution. When the camera captured the images, highly curved fibers were divided into several segments. Snake-like fibers were fitted visually with straight lines and the number of fibers in entangled bundles was estimated visually.

The diameter-based fiber bundle orientation distribution over the entire angular range from -90 to 90 degrees ($MD=0^\circ$), and four parameters extracted from the distribution are provided. The four parameters are:

- (1) Percentage of fiber diameters (N1) orientated near the machine direction ($\pm 20^\circ$ around MD).
- (2) Percentage of fiber diameters (N2) orientated near the cross-machine direction ($\pm 20^\circ$ around CD).
- (3) The ratio of the fiber percentage near the MD to that near the CD (N1/N2).
- (4) Mean fiber orientation angle (weighed mean of the two absolute average angles calculated separately for negative and positive angles) [34].

A total of 400 images were randomly obtained to evaluate the orientation for each web in this test.

CHAPTER IV

RESULTS AND DISCUSSION

All the test data obtained are shown in appendices (See appendices for details). The basis weight distribution of all samples is shown in Fig 4.1. For samples PET-K1, PP-KC and PTT-539, the basis weights are relatively close and fiber diameters range from 10 μ m to 15 μ m. These three samples were selected to compare the web property based on different polymer types. The basis weight and fiber diameter distribution of these three samples are plotted in Fig 4.2. The basis weight and fiber diameter distribution of PTT webs are shown in Fig 4.3.

4.1 Bulk Density

Polypropylene is the lightest of the widely used thermoplastic polymers. Fig 4.4 shows that PP web has the lowest bulk density compared to PET and PTT webs. We can see from this figure that PTT can be produced having the same bulk density as PET.

The web thickness and bulk density for PTT-539, PP-KC and PET-K1 are shown in the Fig 4.5. As can be seen from this figure, the bulk density of PTT-539 is lower than that of PET but higher than that of PP. The bulk density and thickness of four PTT webs are shown in Fig 4.6.

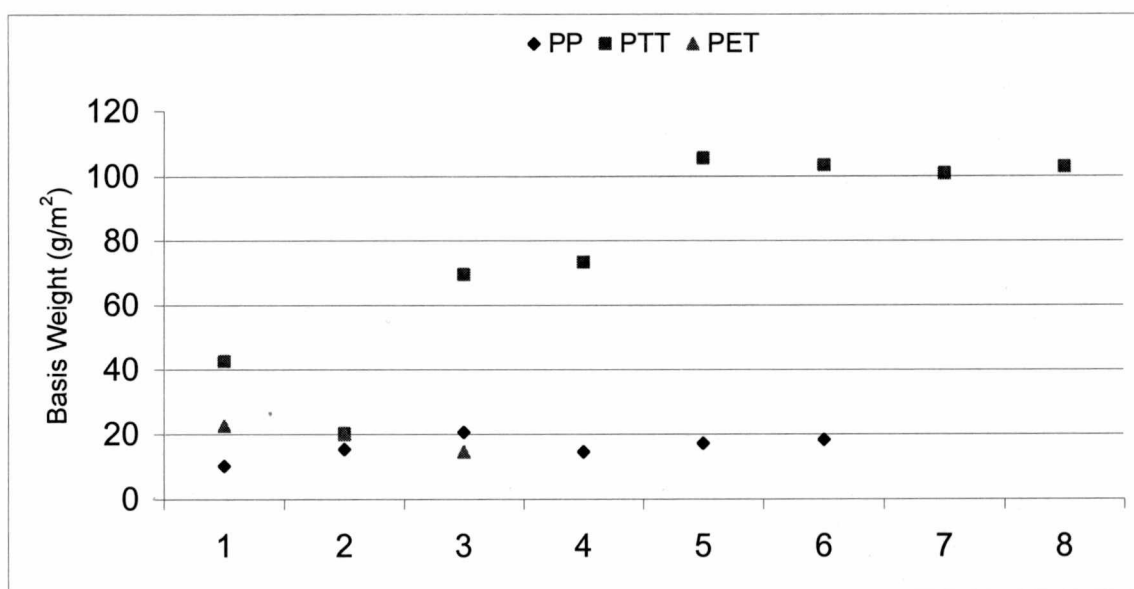


Fig 4.1 Basis Weight Distribution of All Webs

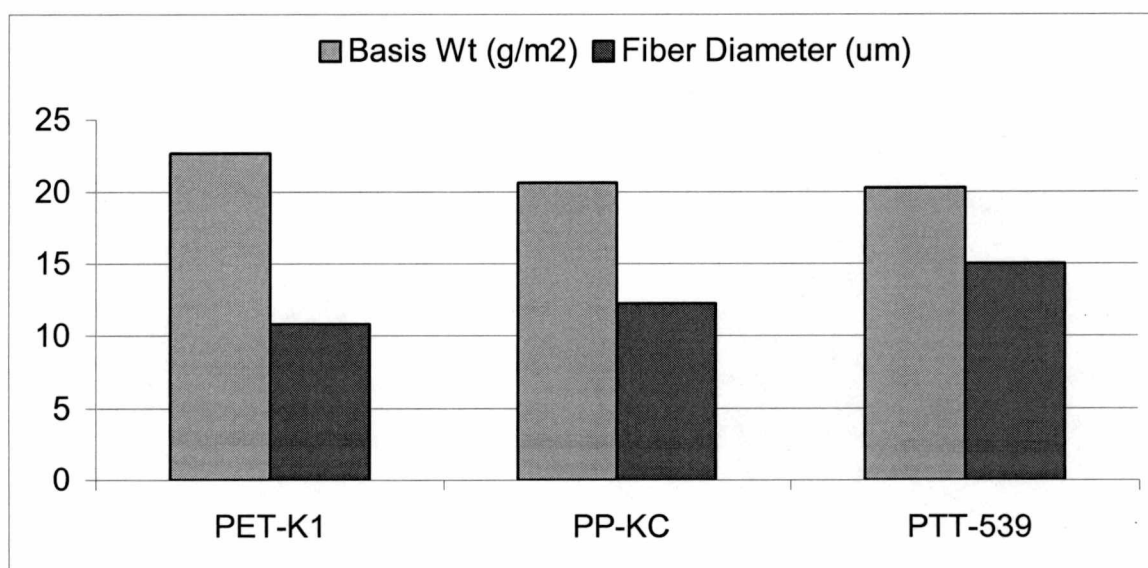


Fig 4.2 Basis Weight and Fiber Diameter of PTT, PET and PP Webs

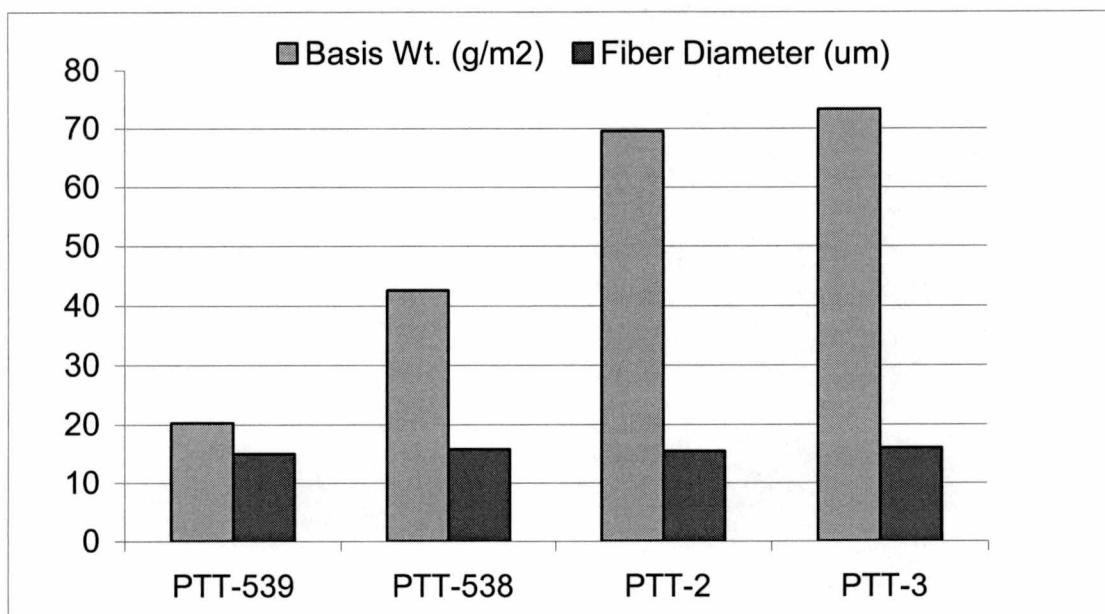


Fig 4.3 Basis Weight and Fiber Diameter of PTT Webs

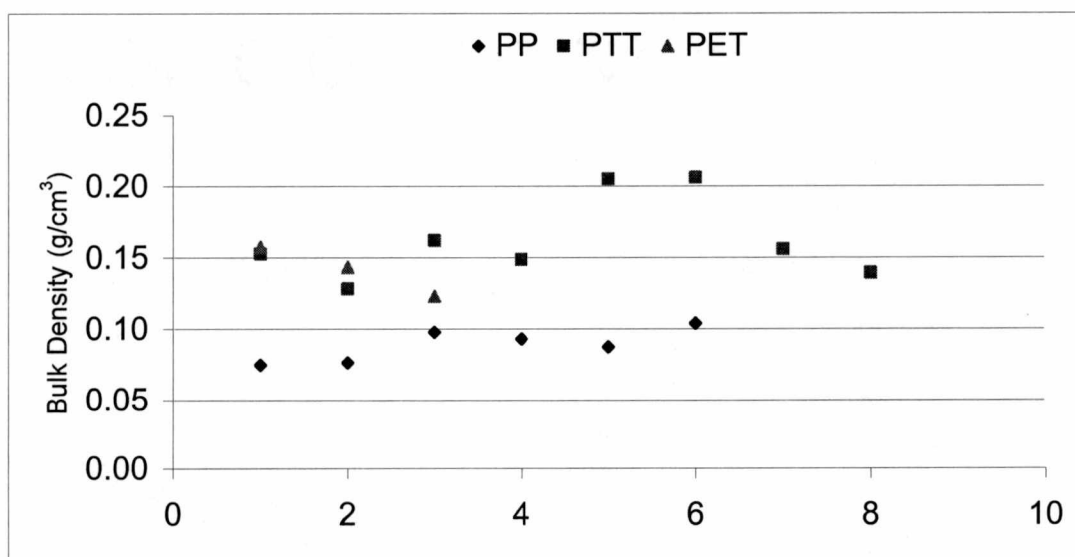


Fig 4.4 Bulk Density of All Webs

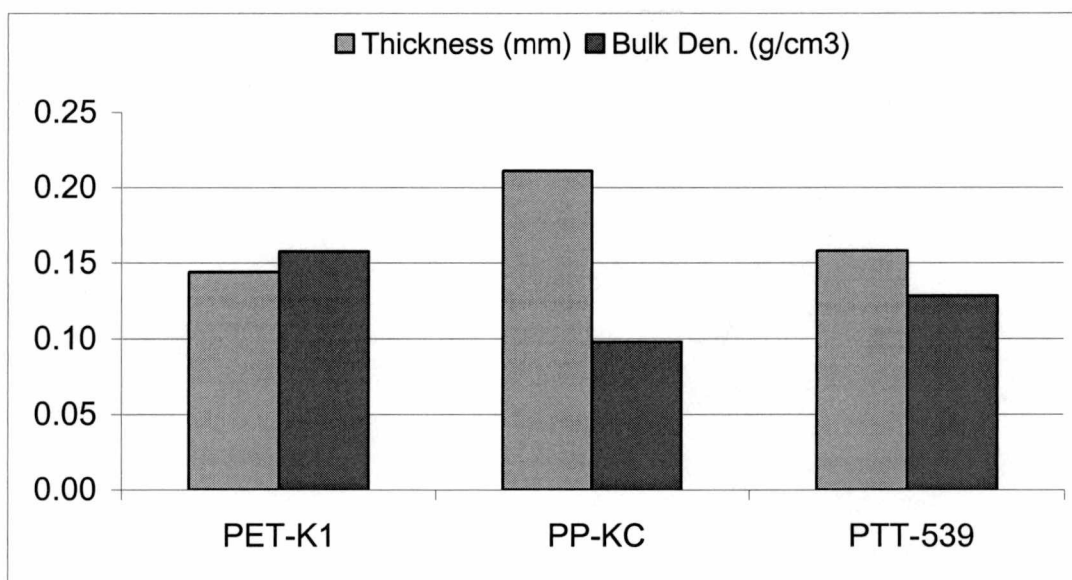


Fig 4.5 Bulk Density and Thickness of PTT, PET and PP Webs

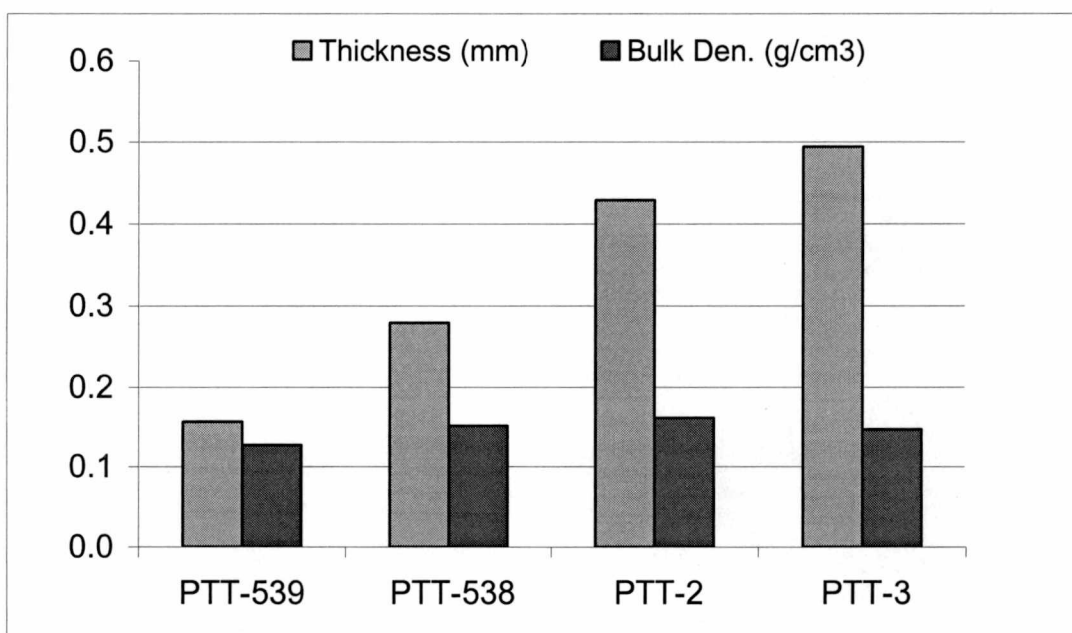


Fig 4.6 Bulk Density and Thickness of PTT Webs

4.2 Web Uniformity Analysis

The web uniformities of the three webs were plotted in Fig 4.7. Spectral values can be seen to generally decrease with increasing cell size (n). A general decrease in CV% is expected as cell size increases since some structural variations among smaller specimens are physically smoothed in larger specimens. Fig 4.7 shows that CV% decreased at a greater rate with increasing cell size for PP web than for PTT web. This may be interpreted in the following way: PP web was less uniform than PTT web when evaluated on a size scale smaller than 1mm x 1mm but PP Web was more uniform than PTT web when evaluated on larger size scales. That is, the CV% of small cell areas may be associated with microscopic structural uniformity whereas the CV% of large cell areas may be associated with macroscopic structural uniformity. Fig 4.7 also shows that PET web was more uniform than PTT web no matter whether it was evaluated on smaller size scales or larger size scales. This means the PET web was more uniform than PTT web both from a microscopic and a macroscopic structural point of view.

Compared to PTT-538 and PTT-539, PTT-1.3 and PTT-2.1 are two poorly bonded webs. We can even visualize that the webs are really non-uniform. The web uniformity spectra of these two webs were plotted in Fig 4.8. The web uniformity of PTT-538 and PTT-539 were also plotted in the same figure because we considered these two webs to be relatively uniform. By comparing these four uniformity spectra, we can clearly see that the CV% value of PTT-1.3 and PTT-2.1 is much higher than that of PTT-538 and PTT-539 even though the basis weight of PTT-1.3 or PTT-2.1 is 5 times as much as that of PTT-539.

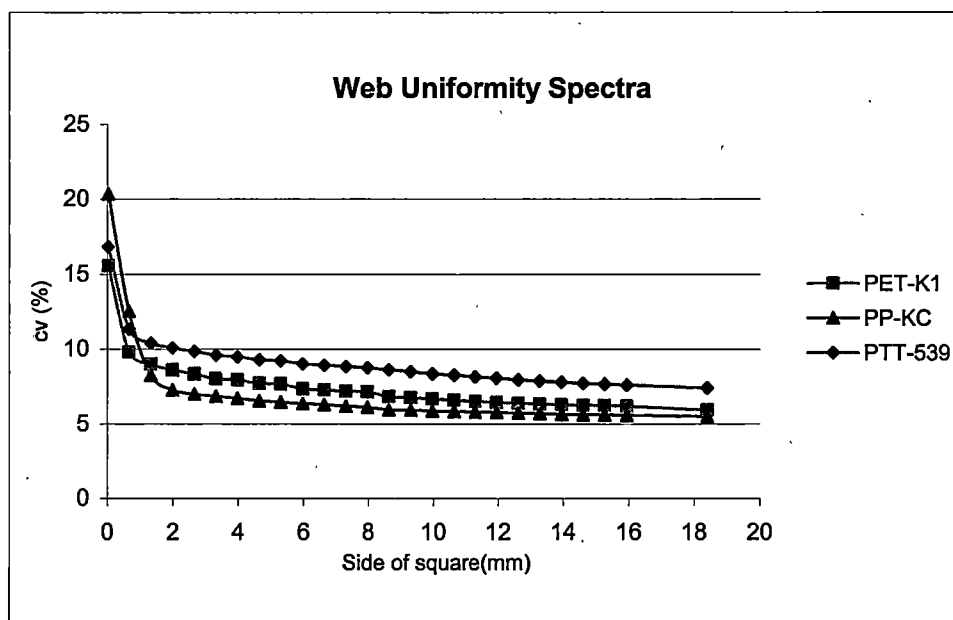


Fig 4.7 Web Uniformity Spectra of PTT, PET and PP

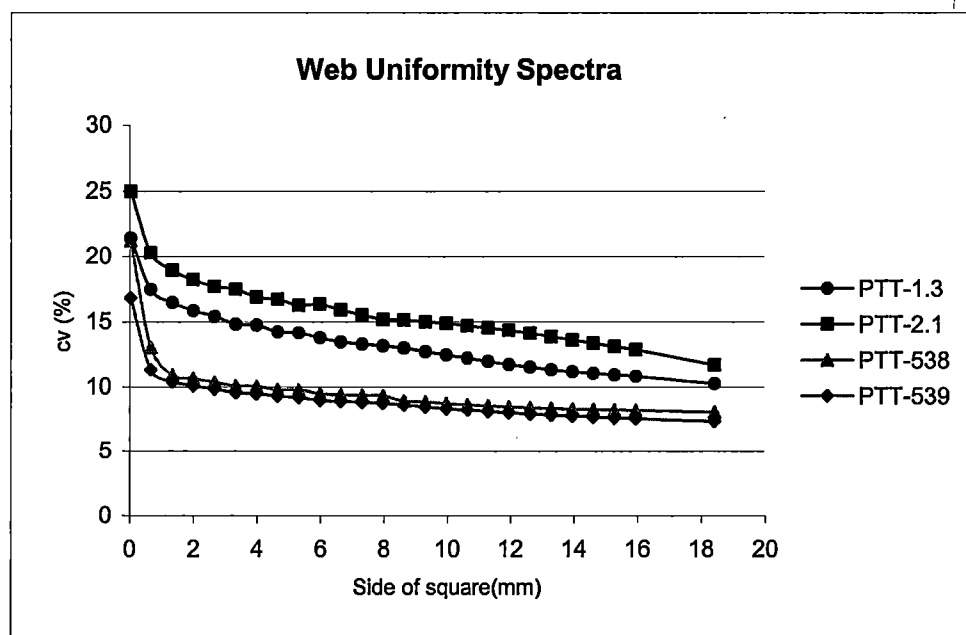


Fig 4.8 Web Uniformity Spectra of PTT-538, PTT-539, PTT-1.3 and PTT-2.1

PTT-2 and PTT-3 were the thinnest webs produced on Reicofil[®] 3 spunbonding line. The uniformity spectra of these two webs were plotted in Fig 4.9. The uniformity spectra of PTT-538 and PTT-539 are also plotted in the same figure to compare the uniformity. The fiber diameters of all these four samples are approximately 15 μ m. As can be seen from this figure, PTT-3 was more uniform than the other three webs when evaluated on larger size cell scales. However, this web was based on the larger basis weight compared to the samples produced on Reicofil[®] 3 spunbonding line. The basis weight of PTT-2 or PTT-3 is over 3 times as much as that of PTT-539. Samples produced on Reicofil[®] 3 were more uniform than the samples produced on Reicofil[®] 2 when evaluated on size scales smaller than 2mm x 2mm even though the basis weight of samples produced on Reicofil[®] 3 are lighter.

4.3 Fiber Orientation Analysis

Fig 4.10 shows the comparison of fiber orientation of the PTT, PET and PP webs. Fig 4.11 shows the fiber orientation distribution of four PTT webs. The orientation ratio of MD to CD is shown in Table 4.1.

Fig 4.10 indicates that three webs differ in orientation with PP web being the most orientated and PTT being the least orientated in the machine direction. Each of the three webs appears to be approximately, but not accurately, orthotropic with regard to fiber orientation. But all the webs tend to be orientated along the machine direction.

Fig 4.11 indicates that these four webs show the different fiber orientation distribution. Basis weight difference may be one of the possible factor affect the fiber orientation. We

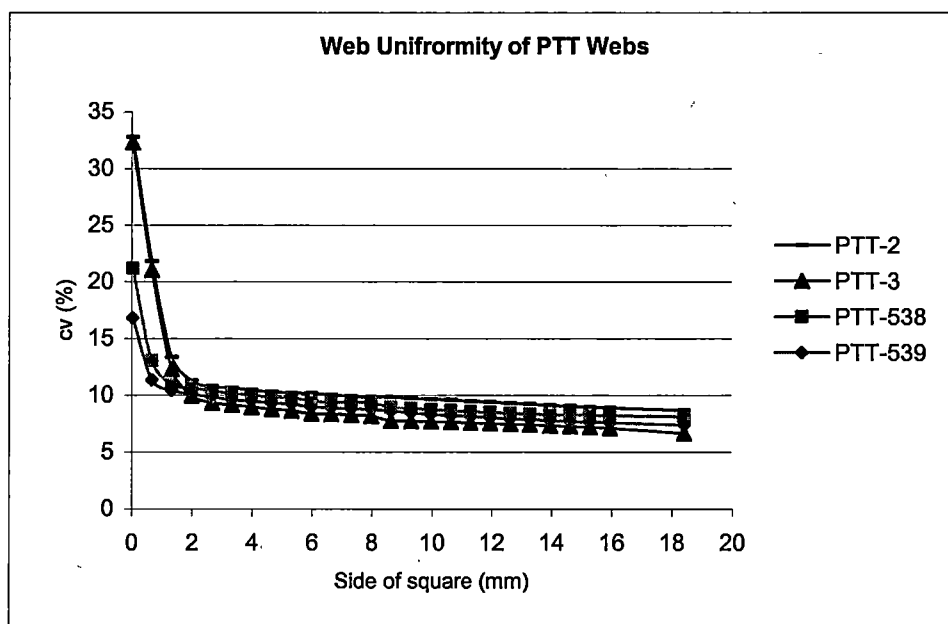


Fig 4.9 Web Uniformity spectra of PTT-538, PTT-539, PTT-2 and PTT-3

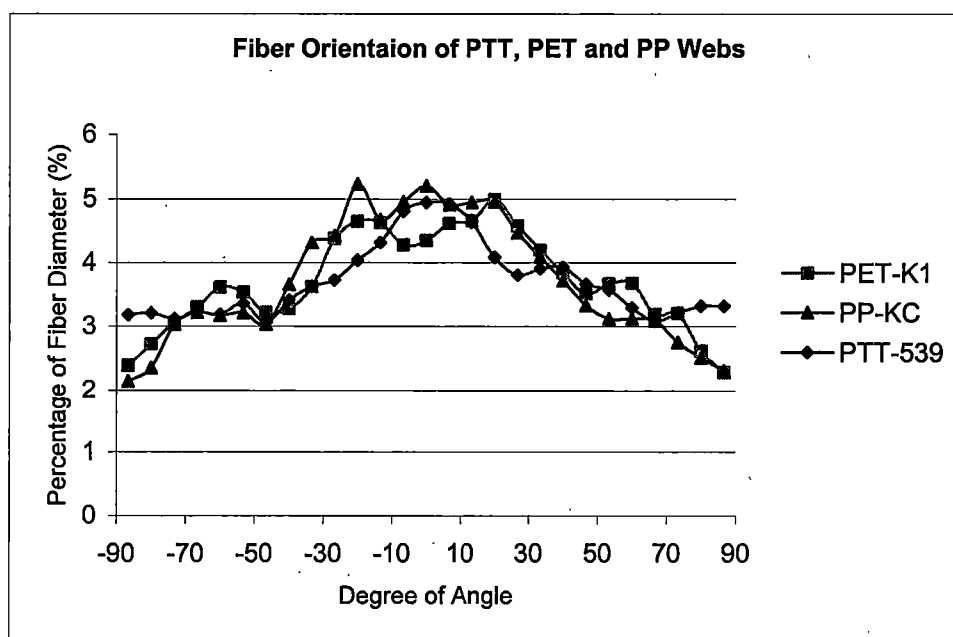


Fig 4.10 Fiber Orientation of PTT, PET and PP Webs

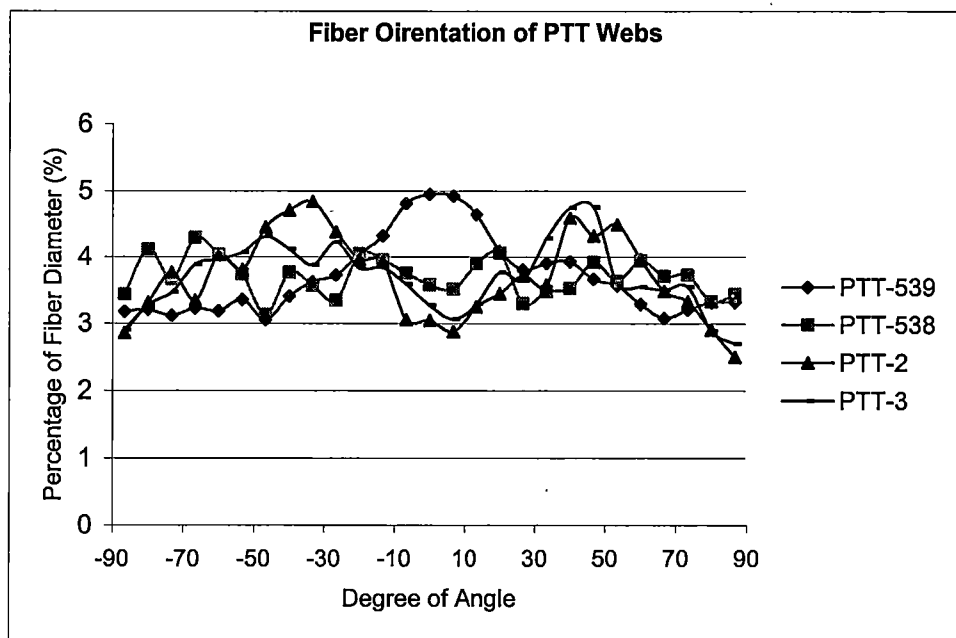


Fig 4.11 Fiber Orientation of PTT Webs

Table 4.1 Orientation Ratio of MD to CD

Sample ID	N1/N2	N1 (%)	N2 (%)	Orientation Angle (degree)
PTT-539	1.4	27.8	19.4	41.4
PET-K1	1.7	27.4	16.3	40.2
PP-KC	2	29.9	15.2	38.5
PTT-538	1.1	22.9	21.8	45
PTT-2	1.1	19.8	18.7	44.3
PTT-3	1.1	20.9	18.8	44
PET-K3	1.5	26.5	17.8	41.2
PP-KA	2.4	33.4	13.8	36.4
PP-KB	2.2	31.2	14.1	37.6
PET-N	3.5	39.7	11.4	32.8
PP-N	1.9	30.2	16.2	39
PP-R143	1.4	26.6	18.7	41.5
PP-R149	1.4	27.8	19.4	41.9

assume the melt throughput is same for the same spunbonding line, the heavier web will be less orientated along the machine direction because the spin belt moves slower for the heavier web than the lighter web. PTT-538 and PTT-539 were produced on the same spunbonding line. However, the basis weight of PTT-538 is about twice as much as PTT-539. Orientation ratio (PTT-539 vs. PTT-538: 1.4 vs. 1.1) indicates that fibers were more orientated in the MD in PTT-539 than those in PTT-538. On the other hand, PTT-2 and PTT-3 were produced on the same spunbonding line based on the similar basis weight. The fiber orientation distributions of these two webs are quite similar. The orientation ratio of these two webs is 1:1.

Fig 4.12 shows the fiber bundle diameter distribution of PTT, PET and PP webs. The fiber bundle diameter distributions of PTT and PET webs are similar. However, there are more small fiber bundles (about 20 μ m diameter) for PP than PET and PTT webs. There are no large fiber bundles (over 100 μ m diameter) for these three webs.

Fig 4.13 shows the fiber bundle diameter distribution of four PTT webs. The fiber bundle diameter distributions of PTT-539 or PTT-539 are different from that of PTT-2 or PTT-3. However, the fiber bundle diameter distributions of webs produced on the same spunbonding line are similar.

4.4. Tenacity and Break Extension

The tenacity and break elongation in the both machine direction and cross-machine direction were plotted in Fig 4.14(A). The extension and tenacity ratio were plotted in Fig 4.14(B).

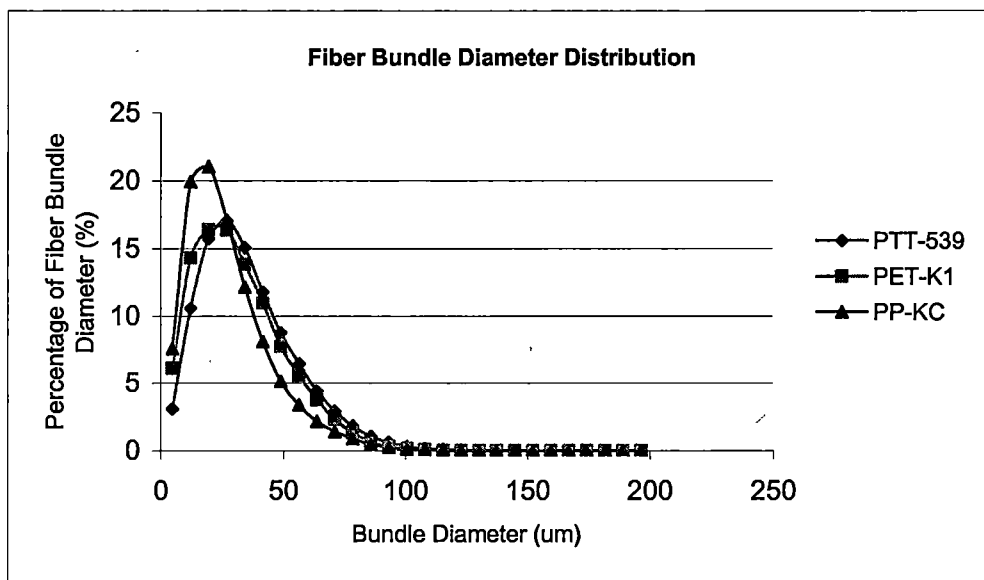


Fig 4.12 Fiber Bundle Diameter Distribution of PTT, PET and PP Webs

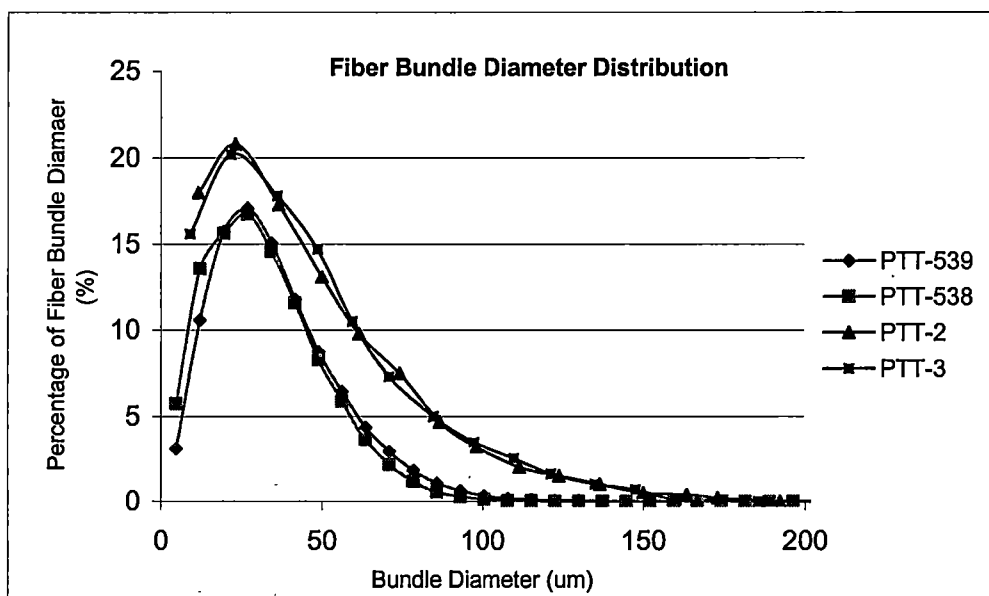
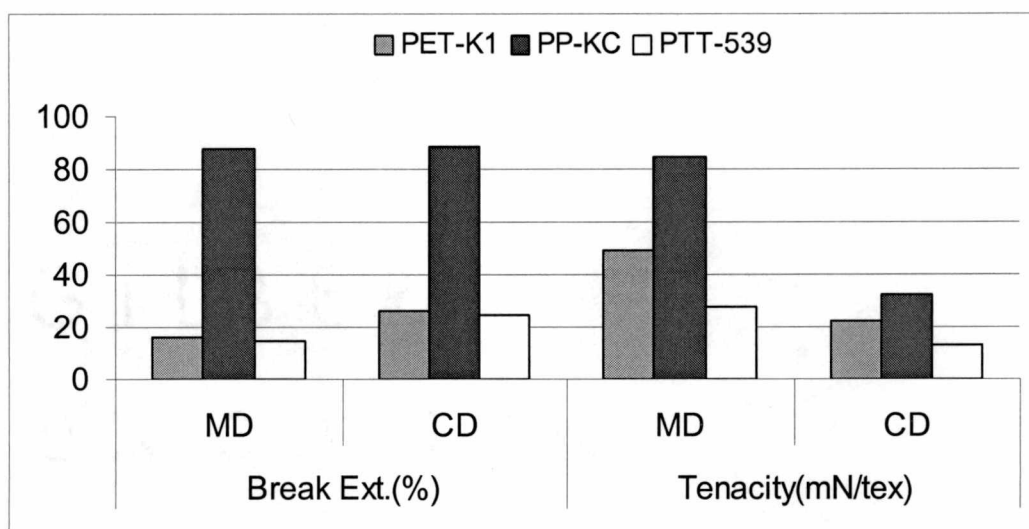
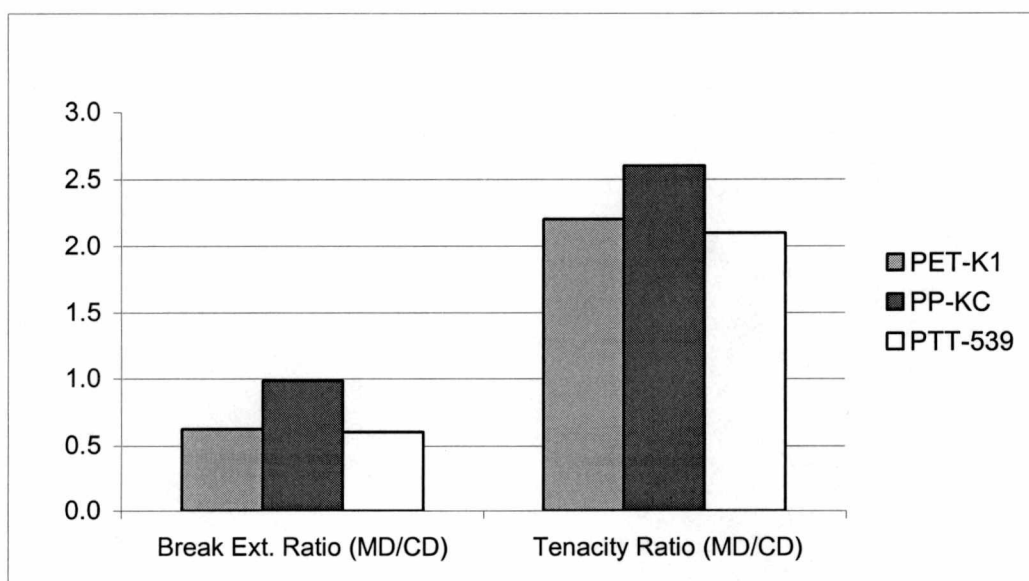


Fig 4.13 Fiber Bundle Diameter Distribution of PTT Webs



(A)



(B)

Fig 4.14 Break Extension and Tenacity.
(A) PTT, PET and PP Webs, (B) Ratio of PTT, PET and PP Webs

As can be seen from the Fig 4.14(A), PTT forms a weaker fabric than PP and PET at the same basis weight, with a lower elongation to break. It has been reported that tensile properties in various web directions correlated with the fiber orientation distribution. It is generally known that CD tensile strength is lower than MD strength in many nonwoven webs and Fig 4.14(A) shows that this was the case for the three spunbonded webs we evaluated. The reason for this is that fibers in webs are oriented more in the MD.

Fig 4.14(B) shows that PP web has the highest tenacity ratio, while PTT web has the lowest tenacity ratio because fibers in PP web are the most orientated in the MD and fibers in the PTT web are the least orientated in the MD.

The break extension and tenacity of PTT webs are shown in Fig 4.15. PTT-539 has the higher tenacity in the MD because fibers in PTT-539 are more orientated in the MD than those in the other webs.

One strange result obtained is the break extension ratio of MD to CD of PP web. Since the PP web was most orientated in the machine direction, it was supposed to have the lower break extension in the MD than CD. However, the result showed that the break extensions in the MD and CD were identical. In order to explore the possible reason, we compared the web uniformity in the MD and CD. The MD and CD uniformity spectra of these three webs were plotted in Fig 4.16, Fig 4.17 and Fig 4.18 respectively. The web uniformities of these three webs differ from one another. For PP and PTT webs, the webs were less uniform in the MD than in the CD. For PET web, the web uniformities in the MD and CD are similar.

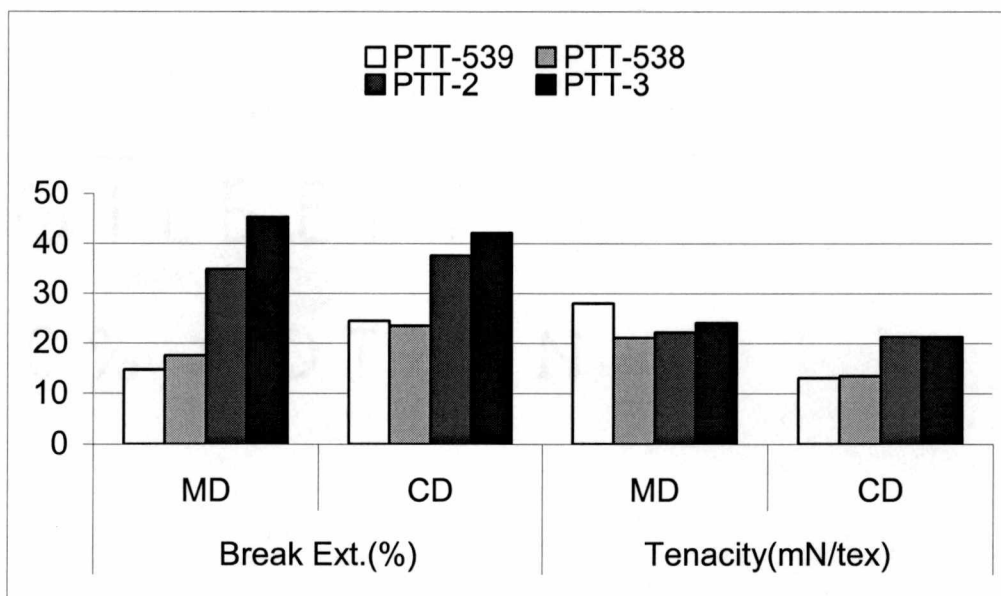


Fig 4.15 Break Extension and Tenacity of PTT Webs

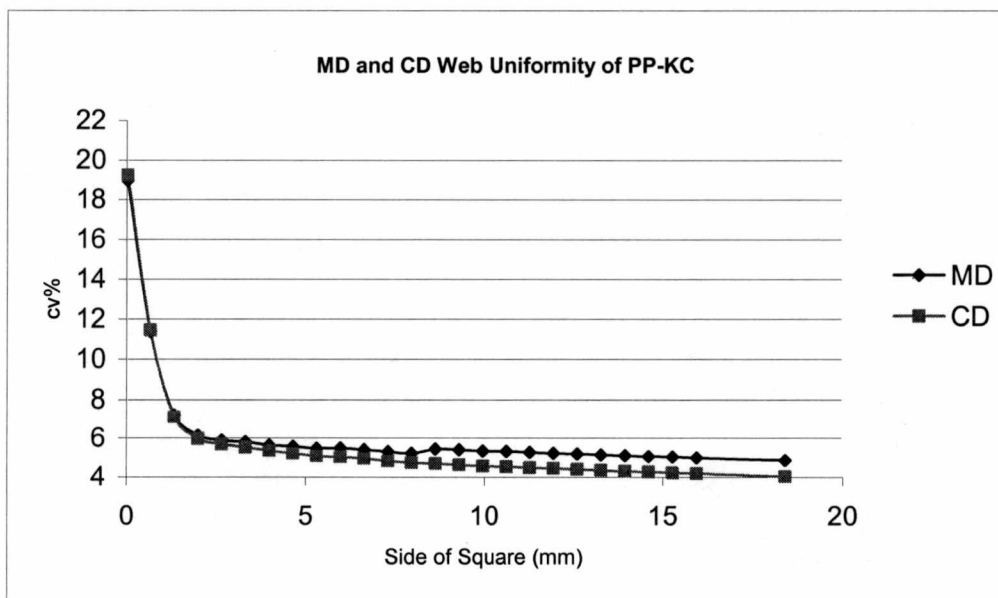


Fig 4.16 MD and CD Web Uniformity Spectra of PP-KC

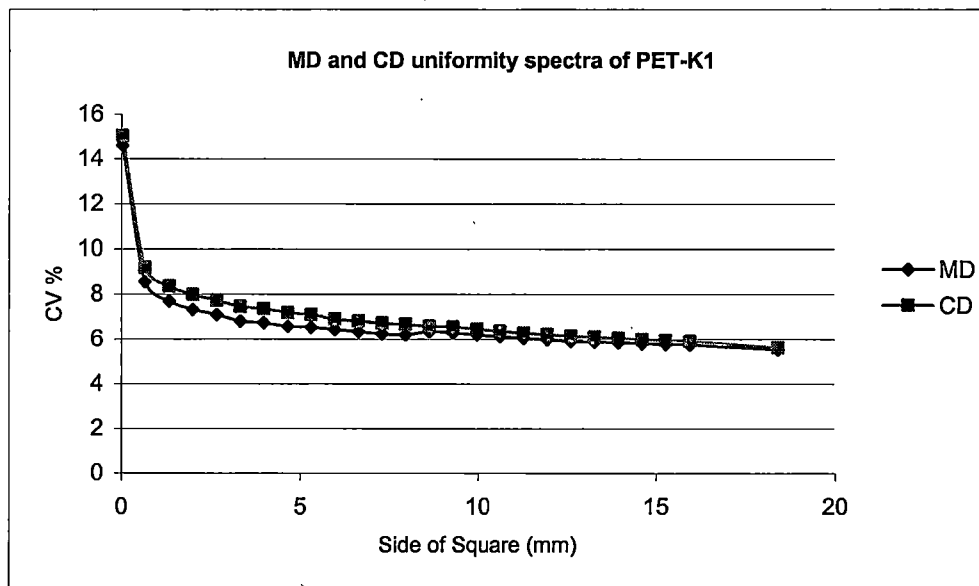


Fig 4.17 MD and CD Web Uniformity Spectra of PET-K1

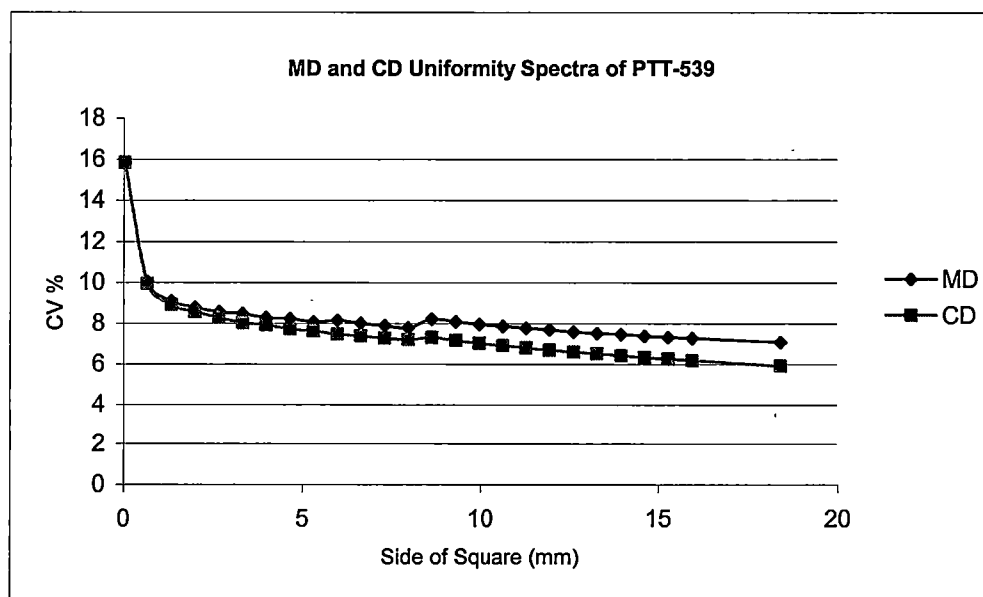


Fig 4.18 MD and CD Web Uniformity Spectra PTT-539

Fig 4.16 indicates that PP web was less uniform in the MD than in the CD. Consequently, when a force was applied to draw the web, the web was relatively easier to break in the MD than CD. This may be one of the possible factors that affect the extension ratio.

As can be seen from Fig 4.18, PTT web was also less uniform in the MD than in the CD. One may question why PTT web still has the lower extension ratio in the CD than MD. Since PTT-538 was produced on the same spunbonding line as PTT-539, Fig 4.19 was used to illustrate the bonding pattern of PTT-539. The distance between the centers of two nearest bonding points in the MD and CD was measured. The distance ratio of CD to MD is about 1.6. That means there are more bonding area in the MD than CD in PTT-539. From this point of view, the web is easier to break in the CD than MD. The effect of uniformity in the MD and CD counteracts with the effect of bonding pattern. This may be the reason of PTT web having the lower extension ratio in the CD than MD.

On the other hand, the bonding patterns of PP and PET webs are isotropic. That means the bonding pattern do not cause the performing difference in the MD and CD for these two webs.

4.5 Air Permeability

Air permeability is related to the structural characteristics of nonwovens. Fig 4.20 shows the air permeability of PTT, PET and PP webs. Fig 4.21 shows the air permeability of four PTT webs. PTT web has the highest air permeability compared to PET and PP webs. The air permeability of PET and PP were similar. From Fig 4.20 we can see that PTT web has larger fiber diameter compared to PET and PP webs. This is one of the possible

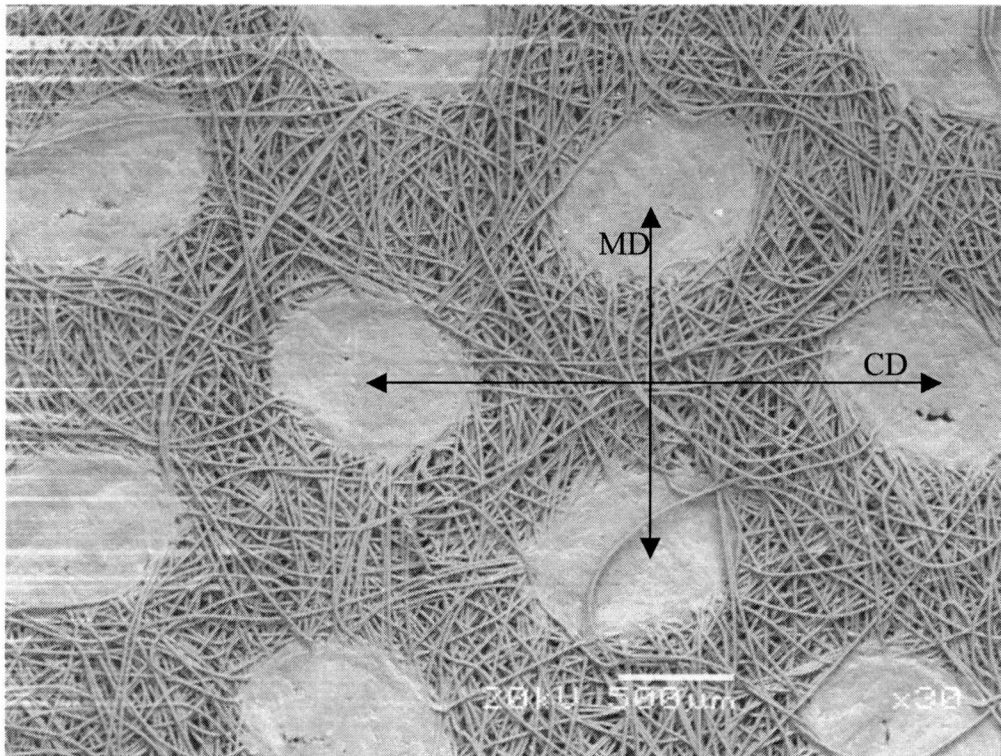


Fig 4.19 SEM Micrograph of PTT-538 Showing the Bonding Pattern

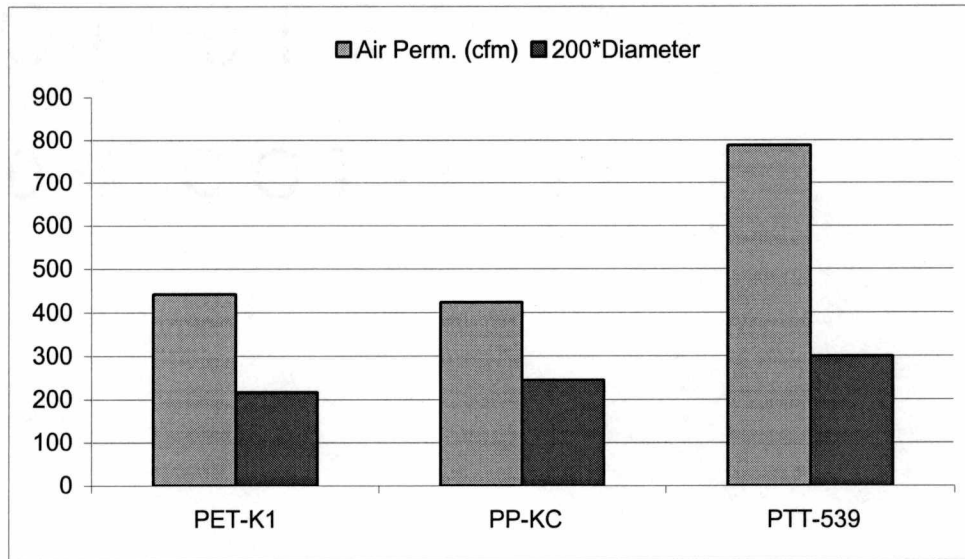


Fig 4.20 Air Permeability of PTT, PET and PP Webs

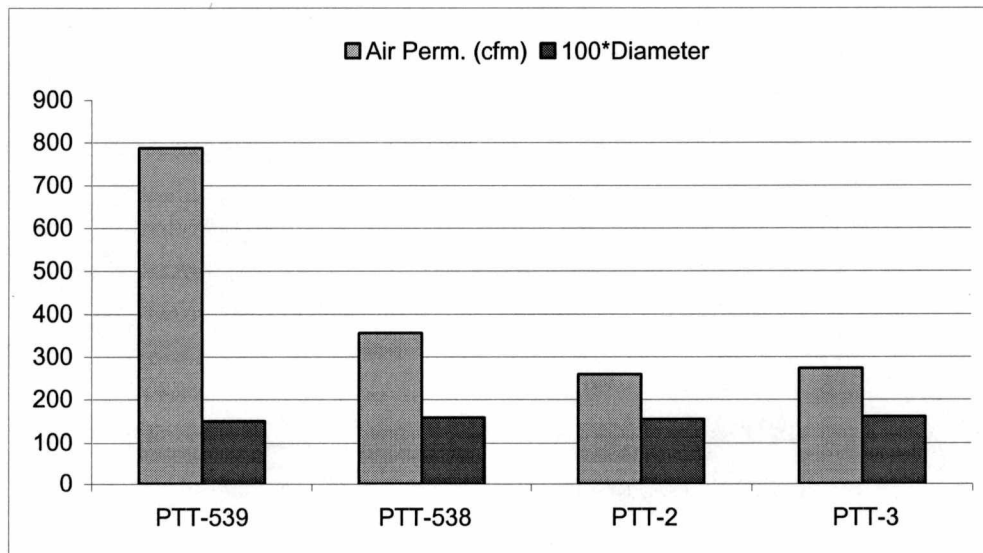


Fig 4.21 Air Permeability of PTT Webs

factors that affected the air permeability. The percentage of bonding area is another factor that affects the air permeability of the web. The percentage of bonding area in PET web is about 25% of the total area. While the percentage of bonding area in PP and PTT is about 22% of the total area. The bonding patterns of these three webs are shown in Fig 4.22, Fig 4.23 and Fig 4.24 respectively. Since PP web has the larger fiber diameters and smaller percentage of bonding area than that of PET web, one question is why PP web has similar air permeability as PET web. The answer may be that fiber bundle diameter distribution of PP web is different from PET web. There are more small fiber bundles (about 20 μ m diameter) in PP web than PET. These small fiber bundles enhance the barrier property of PP web.

4.6 Hydrostatic Pressure and Liquid Strike-through-time

Fig 4.25 shows PP web has the highest hydrostatic pressure because PP is more hydrophobic compared to PET and PTT webs. Our previous discussion also showed that there are more small fiber bundles (about 20 μ m diameter) in PP web than PET and PTT webs. These small fiber bundles enhance the barrier property of PP web. PP web requires higher pressure to let the liquid pass through the web. Table 4.2 shows the test results of liquid strike-through-time. From this table, we can see that the liquid almost could not pass through PP web without any pressure. On the contrary, liquid could easily pass through the PTT web within a relatively short time. PET requires more time to let liquid pass through than PTT does.

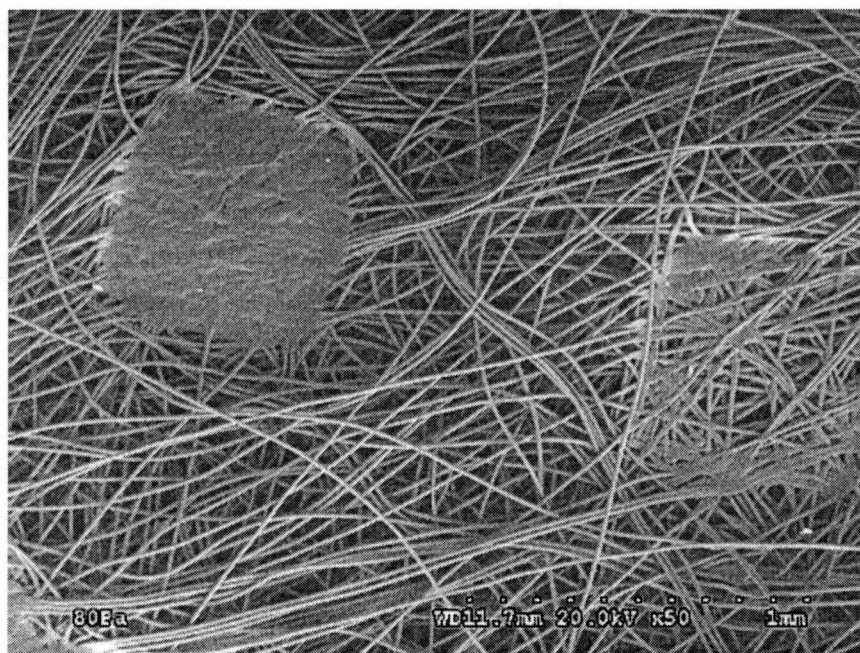


Fig 4.22 SEM Micrograph of PP-KC

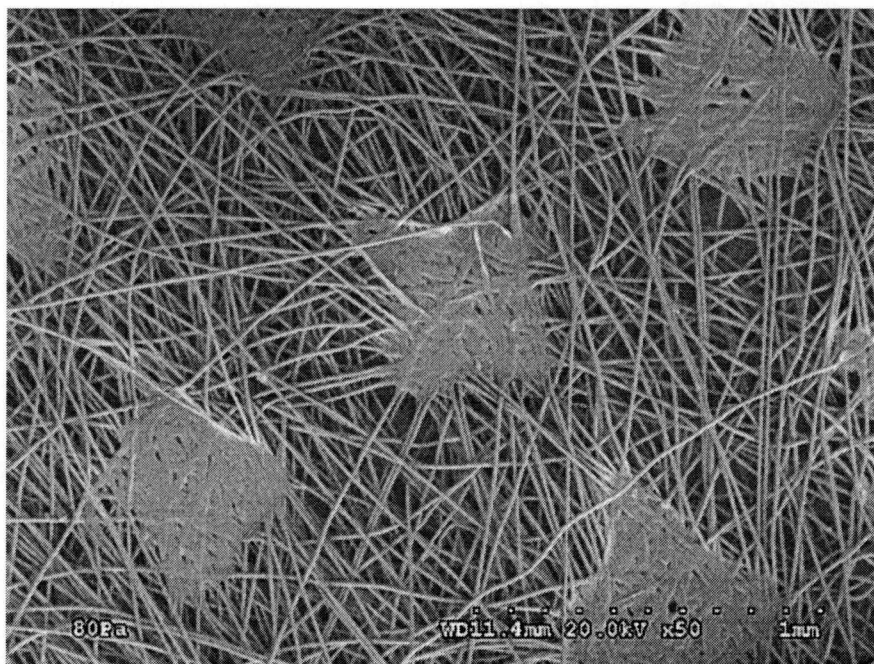
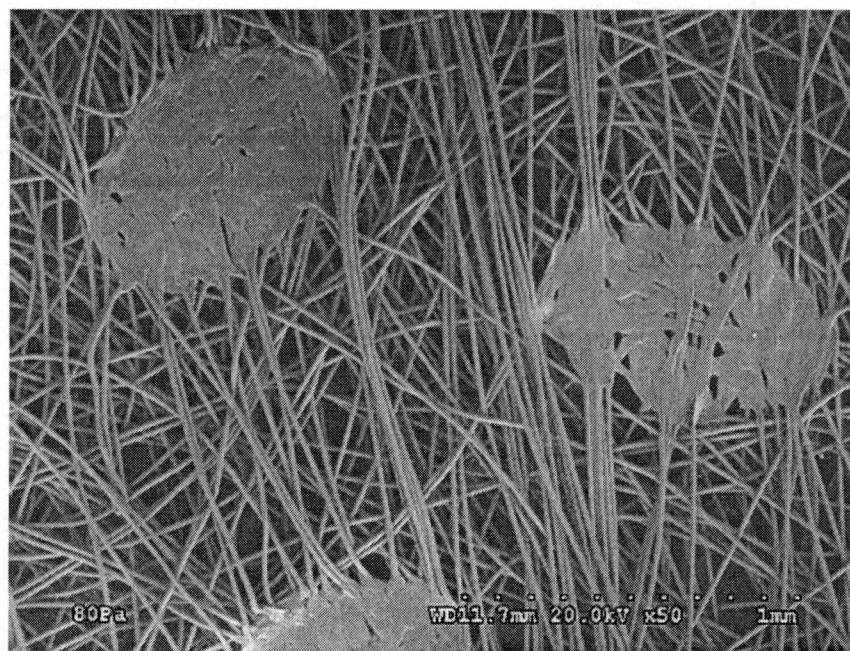


Fig 4.23 SEM Micrograph of PET-K1



**Fig 4.24 SEM Micrograph of PTT-539
Before Heat Treatment**

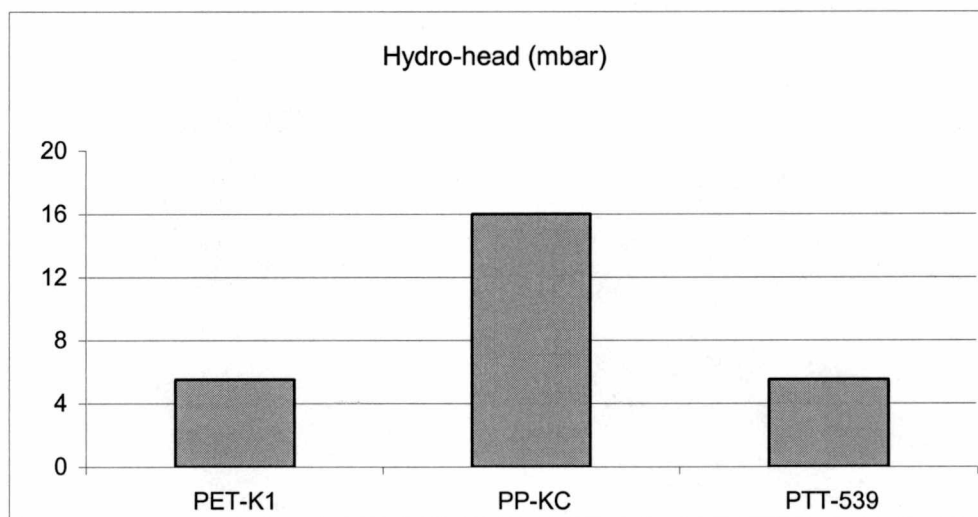


Fig 4.25 Hydrostatic Pressure of PTT, PET and PP webs

Table 4.2 Liquid Strike-through-time of All Webs

Sample ID	Liquid Strike-through-time (second)
PET-K1	27.12
PP-KC	>1000
PTT-539	7.24
PET-K3	4.18
PP-KA	31.25
PP-KB	63.14
PET-N	5.69
PP-N	>1000
PP-R143	82.2
PP-R149	>1000
PTT-538	>1000
PTT-2	>1000
PTT-3	>1000
PTT-14	>1000
PTT-16	>1000
PTT-1.3	>1000
PTT-2.1	>1000

Fig 4.26 shows the hydrostatic pressure of PTT webs. PTT-538 has the higher hydrostatic pressure than PTT-539 because the basis weight of PTT-538 is twice as much as PTT-539. The heavy basis weight enhances the barrier property. The hydrostatic pressure of PTT-2 is higher than that of PTT-3 even though the basis weight of PTT-2 is slightly lower than that of PTT-3. This result may be explained by the web uniformity analysis.

Fig 4.9 shows that PTT-2 web is less uniform than PTT-3 web. There are more weak points or areas in the non-uniformed web than the relatively uniformed web, making liquid easier to pass through non-uniformed web.

Liquid strike-through-time also shows that liquid could pass through the thin PTT web within relatively short time. However, liquid could not pass through the thick PTT webs within relatively short time without any pressure.

4.7 Elastic Recovery

Fig 4.27 shows that PTT has excellent elastic recovery. The stretched PTT web 100% returned to its original length. PET and PP also show good elastic recovery. But compared to PTT, there is a small permanent deformation. The elastic recovery of the web may be due to the web structure since there is some flexibility between the fibers in the web. When a force was applied to draw the web, the fibers tended to move in the direction of the force. Another factor that contributes to the web elastic recovery is the fiber property. One of the unique properties of the PTT fiber is its excellent elastic recovery. PTT's glass transition temperature is above room temperature making it settable and giving its crimp stability. Its molecular arrangement is such that the polymer

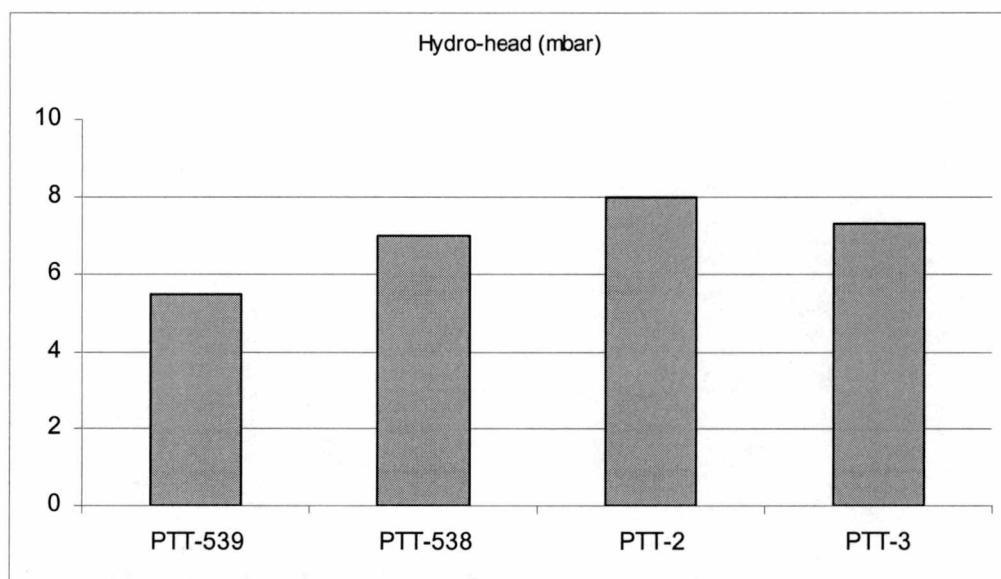


Fig 2.26 Hydrostatic Pressure of PTT Webs

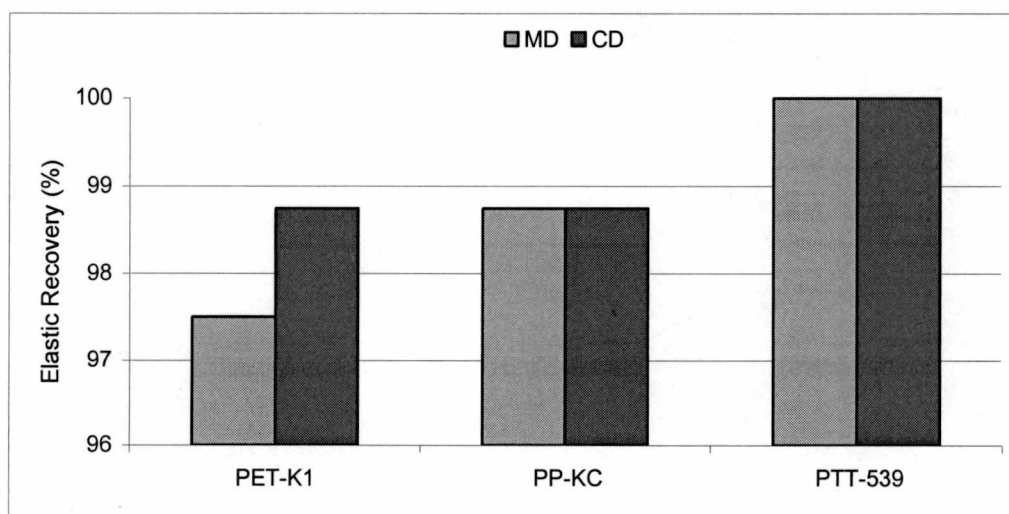


Fig 4.27 Elastic Recovery of PTT, PET and PP Webs

chains deform like a spring along the fiber axis when stress is applied and substantially recovers when the stress is removed [36]. PET undergoes irreversible deformation under stress although it is settable. PP is neither settable nor does it have good recovery [37]. PTT fiber tends to return to its original position after elongation. This may be why the PTT web has excellent elastic recovery.

Fig 4.28 shows the elastic recovery of PTT webs. Although PTT fiber has good elasticity, the spunbonded PTT webs show the different elastic recovery based on the individual processing conditions. This indicates the elastic recovery of the web is related to the web structure as well as fiber property.

4.8 Stiffness

Fig 4.29 shows the flexural rigidity of PTT, PET and PP webs. For all the webs, flexural rigidity in the MD is higher than that in the CD. This result can be explained by the fiber bundle orientation analysis. All the webs are more orientated in the MD than in the CD. Therefore, webs are more rigid in the MD than the CD. PTT web is more flexible than PET and PP webs, although PP web has the lowest density. The elasticity and flexibility of PTT fiber may contribute to the flexibility of PTT spunbonded web. The drape coefficient of these three samples is shown in Fig 4.30. This result is consistent with the flexural rigidity.

Fig 4.31 shows the flexural rigidity of four PTT webs. PTT-539 was more flexural than PTT-538 because of the lighter basis weight. The flexibilities of PTT-2 and PTT-3 webs

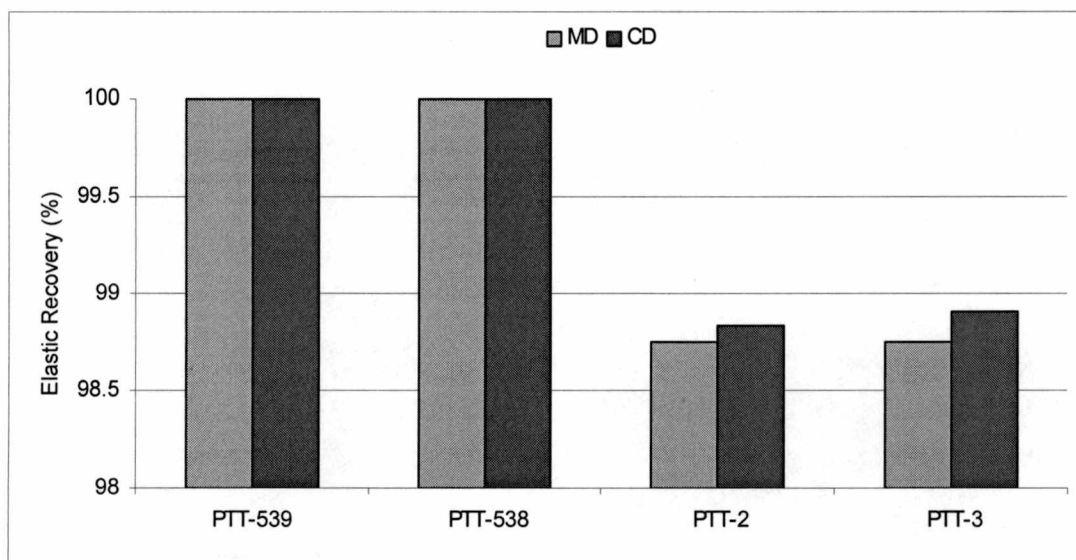


Fig 4.28 Elastic Recovery of PTT Webs

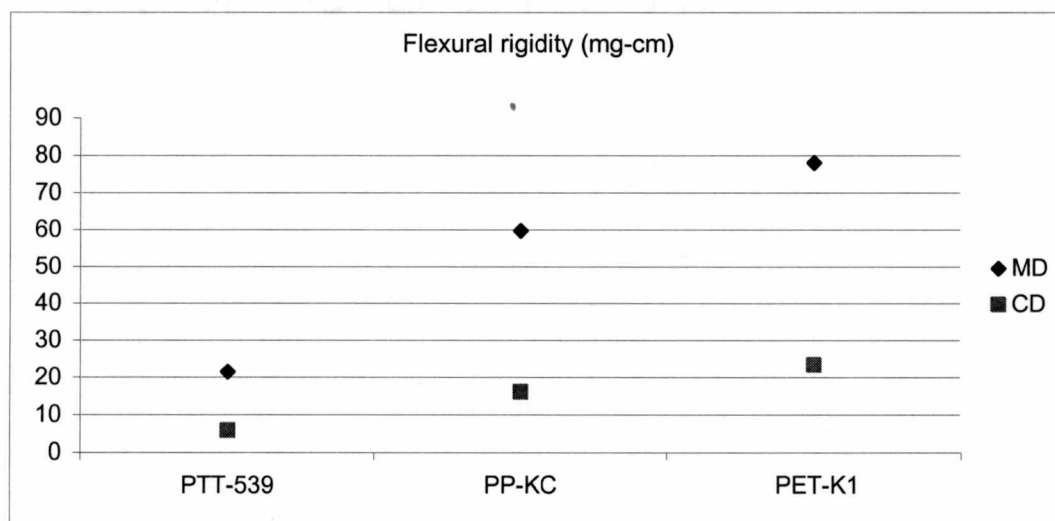


Fig 4.29 Flexural Rigidity of PTT, PET and PP Webs

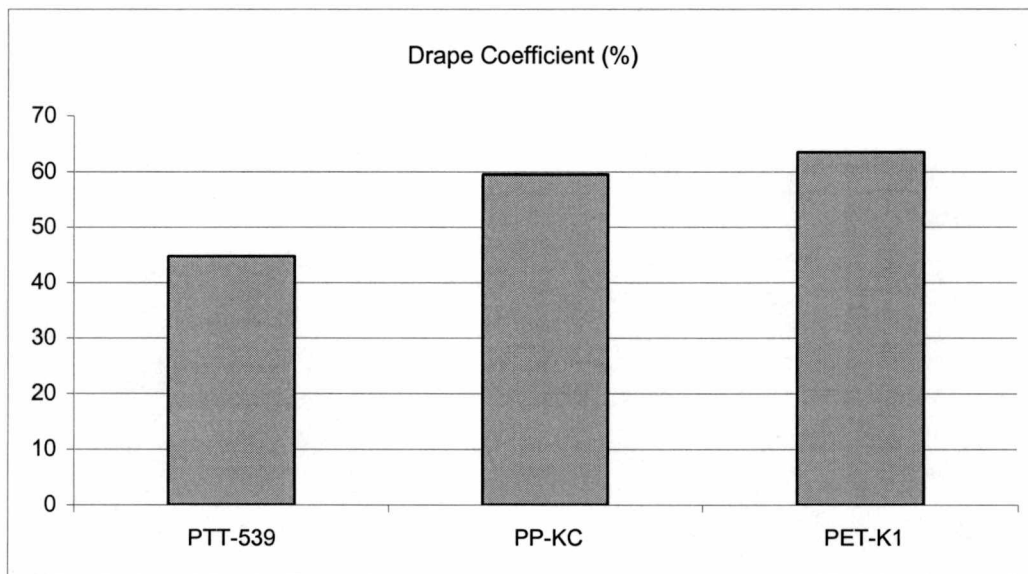


Fig 4.30 Drape Coefficient of PTT, PET and PP Webs

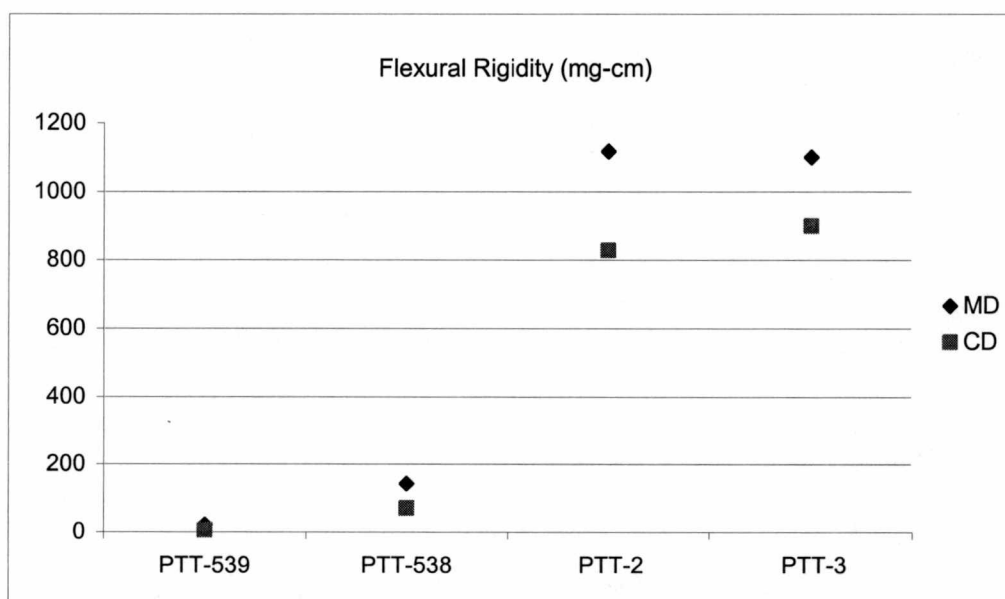


Fig 4.31 Flexural Rigidity of PTT Webs

are similar in MD. PTT-2 was more flexible than PTT-3 in the CD since PTT-2 was lighter than PTT-3.

4.9 Heat Shrinkage

The heat shrinkage measurement is based on the area change of the web. “-” Value means the web shrinks after heat treatment and “+” value means that the web extends after heat treatment. Fig 4.32 shows that the three webs substantially differ in heat shrinkage at different temperatures. All the webs were relatively stable at 90°C. PTT and PET shrank less than 1% while PP did not shrink at 90°C. The shrinkages increased with the temperature for PP and PTT web. However, PTT was more stable than PP at high temperature (150°C). In fact, two spunbonded webs from Reifenhäuser were even melted at 150°C.

An interesting result of the heat shrinkage is that the PTT-2.1 and PTT-1.3 extended after heat treatment. We can see these results from the Fig 4.33. These extensions may be related to the crystallinity at the different temperatures. We conducted the DSC test to verify this possibility. After conducting the DSC tests for all the four samples, we did not find the significant variations for the crystallinity at the different temperatures. The extensions of the PTT-1.3 and PTT-2.1 may be due to the loosening of the fibers at the bonding points and surrounding areas. Fig 4.34 and Fig 4.35 show the SEM micrographs of PTT-2.1. We can see the bonding points and surrounding areas became relatively loose after heat treatment since these two webs were poorly bonded. Consequently, the fibers

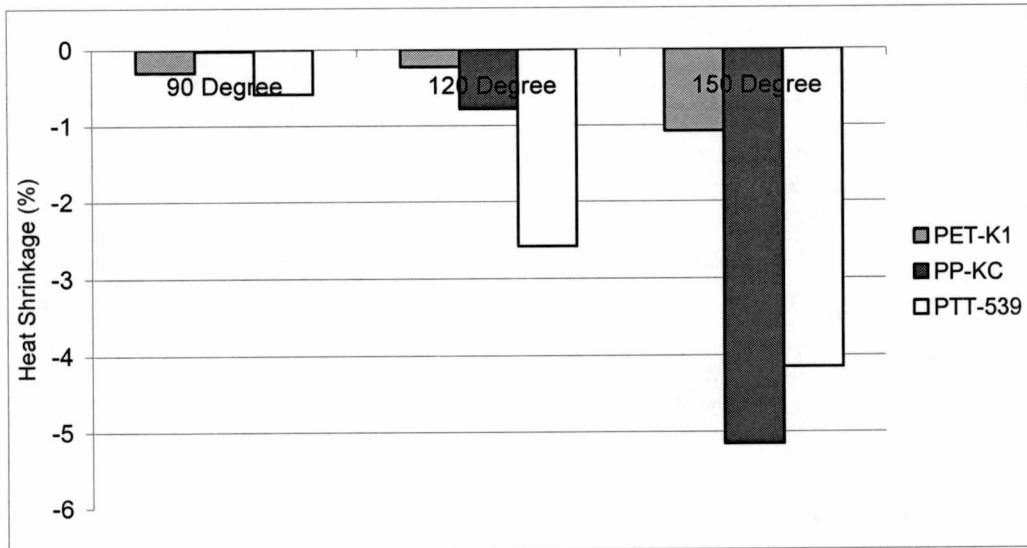


Fig 4.32 Heat Shrinkage of PTT, PET and PP Webs

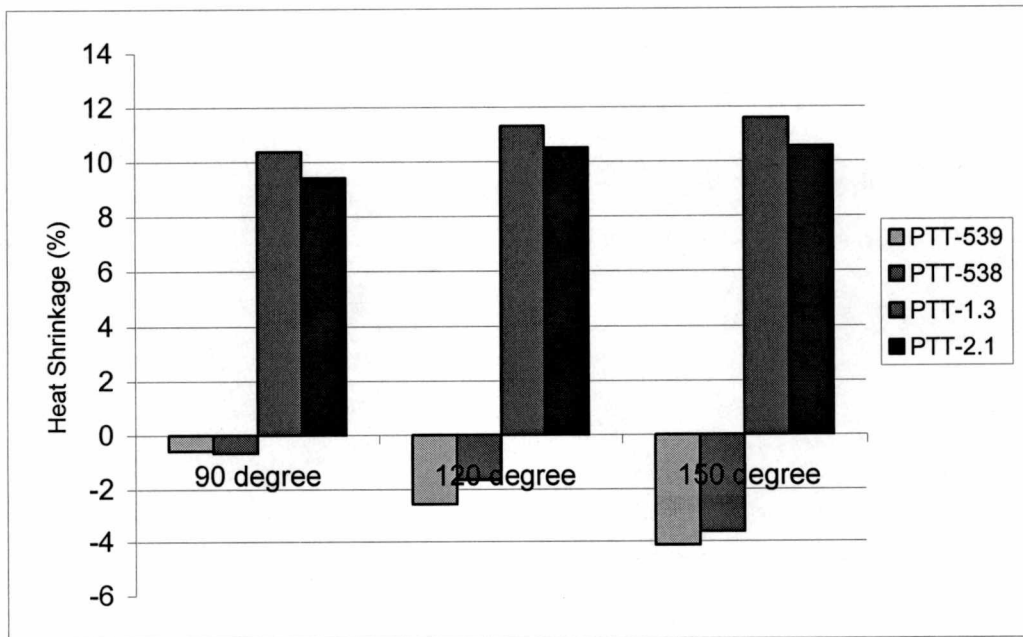


Fig 4.33 Heat Shrinkage of PTT-538, PTT-539, PTT-1.3 and PTT-2.1

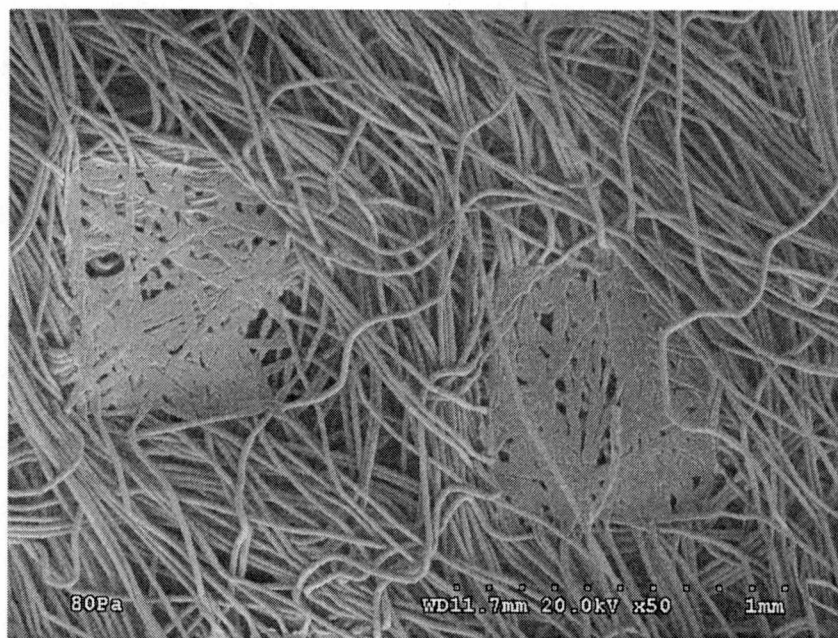
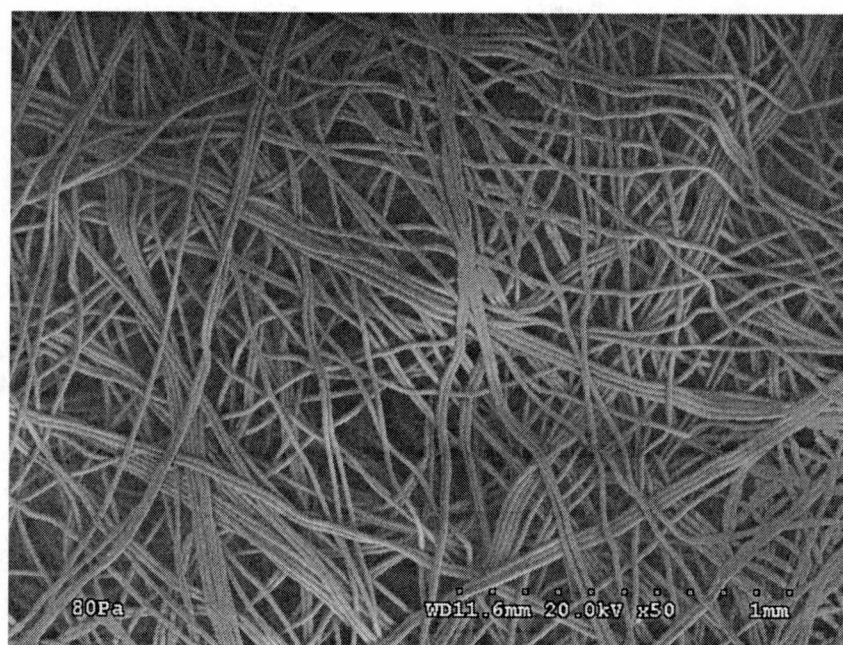


Fig 4.34 SEM Micrograph of PTT-2.1 Before Heat Treatment

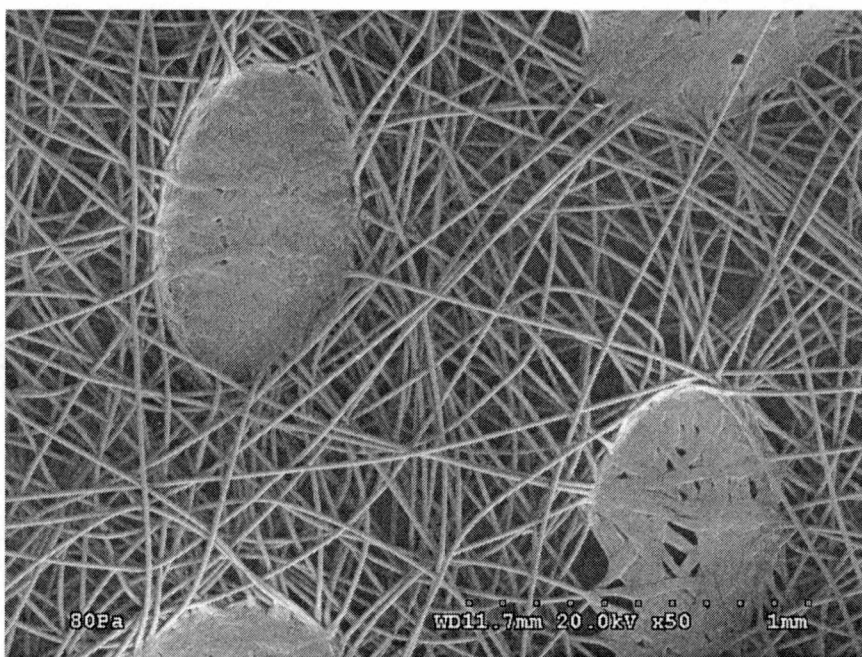


**Fig 4.35 SEM Micrograph of PTT-2.1 After
Heat Treatment at 120°C, 2 Minutes**

tended to relax after the heat treatment. It is important to bond the fiber properly in order to maximize the thermal dimensional stability.

On the other hand, PTT-539 and PTT-538 exhibit little heat shrinkage after heat treatment since these two webs were bonded properly. We could not detect the difference between the images before and after heat treatment from the SEM micrographs as shown in Fig 4.24 and Fig 4.36.

Fig 4.37 shows PTT-2 or PTT-3 was relatively thermal stable that PTT-538 or PTT-539 because PTT-2 or PTT-3 was over-bonded and film-like. The thermal stabilities of PTT-2 and PTT-3 were similar. The slightly different thermal stabilities of PTT-538 and PTT-539 may be related to web structures and processing conditions.



**Fig 4.36 SEM Micrograph of PTT-539 After
Heat Treatment at 120°C, 2 Minutes**

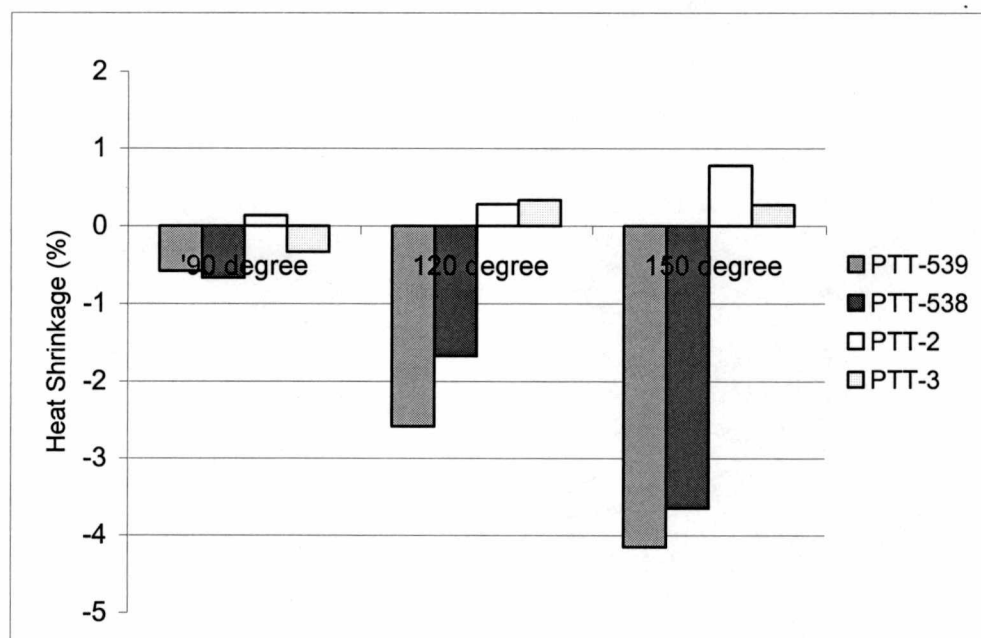


Fig 4.37 Heat Shrinkage of PTT-538, PTT-539, PTT-2 and PTT-3

CHAPTER V

CONCLUSION

PTT spunbonded nonwovens were produced on Reicofil[®] 3 spunbonding line at Refenhäuser, Germany and Reicofil[®] 2 spunbonding line at TANDEC, University of Tennessee, Knoxville. The fundamental properties of the PTT spunbonded nonwovens were investigated. The fundamental properties investigated included basis weight, bulk density, fiber diameter, tensile properties, air permeability, drape ability, stiffness, hydrostatic pressure, liquid strike-through-time, elastic recovery, and heat shrinkage. SEM was applied to examine the web structure. The comparisons among PTT, PET and PTT spunbonded nonwovens were conducted where appropriate. Web uniformity and fiber orientation analysis were also conducted to study the web properties.

From the measurements in this study, PTT spunbonded nonwoven exhibits excellent elastic recovery compared to the PP and PET webs. PTT spunbonded nonwoven is more flexible than PET and PP spunbonded nonwovens. The heat shrinkage of PTT web was greater than that of PP and PET at the temperature 90°C and 120°C. However, PTT web exhibits more heat stability than PP and PET web at a higher temperature (150°C).

The bulk density of PTT spunbonded nonwoven is generally higher than that of PP spunbonded nonwovens. PTT generally forms a weaker spunbonded web than that of PET and PP. PTT was less orientated along the machine direction compared to PP and

PET. PTT web was more uniform than PP web when evaluated on a size scale smaller than 1mm x 1mm, but PTT web was less uniform than PP web when evaluated on larger size scales. PTT web was less uniform than PET web no matter whether it was evaluated on smaller size scales or larger size scales. PTT has the higher air permeability compared to PET and PP. The hydrostatic pressure of PTT is similar to that of PET but lower than that of PP.

From our previous discussion, we can see the web properties are directly related to the processing conditions. The webs produced on Reicofil[®] 3 are different from the webs produced on Reicofil[®] 2. The differences can be seen from the web uniformity and fiber orientation analysis. The webs produced on Reicofil[®] 3 are superior to the webs produced on Reicofil[®] 2. High filament spinning speed is recommended to produce the thin web.

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APPENDICES

APPENDIX I

Characterization of PTT, PET and PP Spunbonded Nonwovens

Sample ID	Basis Wt (g/m ²)	Thickness (mm)	Bulk Den. (g/cm ³)	Fiber Diameter		Peak Load (kg)		Break Ext. (%)		Tenacity (mN/tex)		Air Perm. (cfm)
				Ave. (μm)	C.V. (%)	MD	CD	MD	CD	MD	CD	
PET-K1	22.68	0.144	0.158	10.81	7.64	2.87	1.30	16.1	25.9	48.9	22.1	442
PET-K3	20.08	0.140	0.143	23.17	4.17	2.27	1.12	13.0	20.1	43.7	21.6	1340
PP-KA	10.30	0.138	0.075	12.45	5.07	1.90	0.58	49.4	58.3	71.1	21.7	901
PP-KB	15.43	0.202	0.076	12.17	5.91	3.20	1.04	66.2	71.6	80.2	26.1	613
PP-KC	20.63	0.211	0.098	12.24	4.43	4.50	1.71	87.4	88.6	84.3	32.0	424
PET-N	14.64	0.119	0.123	10.91	8.72	3.09	0.44	30.8	48.8	81.6	11.6	896
PP-N	14.59	0.157	0.093	13.10	5.97	2.78	0.98	55.4	49.3	73.6	26.0	706
PP-R143	17.18	0.197	0.087	14.64	5.31	1.80	1.13	47.9	63.7	40.4	25.4	703
PP-R-149	18.29	0.176	0.104	14.50	2.69	1.52	0.86	57.5	67.7	32.2	18.1	705
PTT-538	42.58	0.279	0.153	15.77	5.25	2.33	1.49	17.5	23.5	21.1	13.5	356
PTT-539	20.26	0.158	0.128	14.99	6.24	1.47	0.69	14.7	24.5	28.0	13.1	788
PTT-2	69.56	0.429	0.162	15.43	9.94	3.99	3.84	34.8	37.6	22.2	21.3	259
PTT-3	73.36	0.494	0.149	15.99	6.97	4.57	4.05	45.3	42.1	24.1	21.3	274
PTT-14	105.62	0.515	0.205	21.12	8.42	4.90	5.09	27.1	22.3	17.9	18.6	224
PTT-16	103.37	0.501	0.206	21.21	10.56	5.99	5.13	29.8	27.5	22.4	19.2	184
PTT-1.3	100.82	0.647	0.156	16.40	8.71	1.52	1.58	65.2	89.3	5.8	6.1	341
PTT-2.1	102.85	0.739	0.139	16.64	11.40	2.15	1.20	38.9	94.0	8.1	4.5	368

APPENDIX I (continued)

Characterization of PTT, PET and PP Spunbonded Nonwovens

Sample ID	Hydro-head (mbar)	Drape Coefficient (%)	Elastic Recovery (%)		Heat Shrinkage (%)			Flexural Rigidity (mg-cm)	
			MD	CD	90°C	120°C	150°C	MD	CD
PET-K1	5.5	63.35	97.50	98.75	-0.30	-0.23	-1.08	78.04	23.42
PET-K3	4.3	N/A	N/A	N/A	0.00	-0.52	-1.08	118.20	48.47
PP-KA	8.0	40.51	98.25	96.25	0.44	0.32	-4.73	16.05	2.48
PP-KB	12.3	52.02	98.75	98.13	0.44	-0.39	-4.73	34.14	5.18
PP-KC	16.0	59.49	98.75	98.75	0.00	-0.78	-5.16	59.69	16.14
PET-N	6.0	65.38	N/A	99.38	0.00	-0.91	-2.19	67.17	6.64
PP-N	9.3	54.51	98.75	99.38	-0.67	-1.00	-9.91	27.54	6.15
PP-R143	8.3	38.55	98.75	98.13	-0.95	-7.91	NA	12.79	4.18
PP-R-149	8.7	30.46	98.75	96.41	-1.35	-12.04	NA	9.68	3.22
PTT-538	7.0	67.68	100.00	100.00	-0.67	-1.67	-3.65	142.82	71.02
PTT-539	5.5	44.81	100.00	100.00	-0.58	-2.58	-4.15	21.50	5.80
PTT-2.1	8.0	N/A	98.75	98.83	0.13	0.28	0.78	1118.30	828.57
PTT-3	7.3	N/A	98.75	98.91	-0.34	0.33	0.27	1101.46	900.59
PTT-14	7.5	N/A	98.75	98.44	0.33	0.50	1	3443.38	2877.22
PTT-16	8.3	N/A	98.63	98.59	0.13	1.00	1	3355.36	2878.29
PTT-1.3	6.2	N/A	99.75	99.06	10.37	11.30	11.59	920.25	559.80
PTT-2.1	5.8	N/A	98.00	100.00	9.41	10.49	10.55	743.67	262.03

VITA

Weili Zhao was born in Liaoning Province, The People's Republic of China, on January 10, 1966. She entered the Tianjin Institute of Textile Science and Technology in 1985 and received her Bachelor of Science Degree in Textile Engineering in 1989. After graduation, she has worked in textile industry. Her working experience includes textiles testing, factory management, quality control, and garment merchandising. In Fall 2000, she was admitted to the Master's program in Textile Science at the University of Tennessee, Knoxville. She was employed as Graduate Research Assistant during her study at the University of Tennessee. She received her Master of Science Degree, with a major in Textiles, Retailing and Consumer Sciences in December 2001.