To the Graduate Council:

I am submitting herewith a thesis written by John H. Beard entitled "Development and Characterization of Polytrimethylene Terephthalate (PTT) Based Mono- and Bicomponent Meltblown Nonwovens." I have examined the final 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 Textile, Retail and Consumer Sciences.

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Development and Characterization of Polytrimethylene Terephthalate (PTT) Based Mono- and Bicomponent Meltblown Nonwovens

A Thesis Presented for the Master of Science Degree
The University of Tennessee, Knoxville

John H. Beard

August, 2001

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DEDICATION

I dedicate this work to Lezlee, my soon to be lovely wife for her support and encouragement during the rough times and good times, my family for sticking their necks out and helping me. Dong Zhang and Christine Sun for taking me under their wings and showing me the ropes and being my friends when it counted. To everyone at TANDEC for friendship and Teresa Crabtree for everything she has done for me.

ACKNOWLEDGEMENTS

When I began this endeavor, I wanted to show the world what kind of researcher they were getting. I soon realized that the project would become a time consuming animal that would bite back if you let go of your grip. I was fortunate in the sense that this project fell into my lap and that the folks I would be working with are great people who have a great sense of humor and a capacity to care about their workers.

Not many people know what poly trimethylene terephthalate is. I had no clue in January 2000. Now after 18 months I believe I know what it is and why I am doing this research. To the collective knowledge of the University of Tennessee, no one in the United States has conducted research on meltblown PTT before. I am the first.

I hope that when this thesis is read an enlightenment of new knowledge will be created. My one hope is that something can be learnt from my work as to what PTT is and where it can be used.

I want to express my sincere thanks to the fine folks at TANDEC for all the help on the project whether it is a conversation on the process machinery or showing how to test the webs. I want to thank Dr. Dong Zhang, Dr. Christine Sun, and Dr. Kermit Duckett for being on my committee. Shell Chemical Company funded this research thus they should be thanked for the fiscal input. I also want to thank the University of Tennessee for allowing me the chance to take a gamble on myself and giving me the opportunity to better myself.

ABSTRACT

Mono and Bi-component polytrimethylene terephthalate (PTT) based meltblown fiber webs were developed on the Reicofil® side-by-side bi-component meltblown line at Textiles and Nonwovens Development Center (TANDEC) located on the campus of University of Tennessee, Knoxville. Main process variables studied were melt temperature, melt throughput, air flow rate, air temperature, and Die to Collector Distance (DCD).

PTT and bico PTT/PP webs were characterized for fiber diameter, bulk density, air permeability, hydro-head, tensile properties, heat shrinkage, and drape ability. PTT/PP webs displayed a non-round cross-sectional and more twisted fiber structure via SEM (Scanning Electrical Microscope). Average fiber diameter of 1.71 to 4.76 μm was achieved with the melt throughput from 0.3-1.5 g/hole/min, melt temperature from 270 to 305 °C (520 to 580 °F), air temperature from 230 to 310°C (450 to 530 °F), air flow rate from 12.5 to 29.2 SCFM/in, DCD from 11 to 19 inches. It was found that the PTT polymer grade for this study was quite suitable for the meltblown process. PTT/PP bi-component webs show greater resistance to heat shrinkage and enhanced barrier properties.

The PTT fibers have a greater flexural rigidity due to the molecular structure. It seems that the extra CH_2 makes the polymer structurally better than PET. PET has a Tm of 265°C, which is greater than the Tm of PTT, 232°C. This translates to longer heat up times and higher energy costs for meltblowing processing. PTT outperforms PET in heat shrinkage. PET acts as if the very mention of heat, it wants to shrink. PET appeared more absorbent, hydrophilic polymer; thus, the moisture content in PET was considerably

more than with PTT. These condensation polymers should be dried prior to the meltblowing process.

We have discovered that PTT is a dimensionally stable polyester fiber when heated, although PTT had some shrinkage from 110°C to 130°C. The most shrinkage documented was at 150°C, at approximately 15%. PTT at 190°C and 200°C showed stability with only minimal shrinkage. The PTT fibers that were affected the most at 190° C were the largest fibers. 100% PTT sample 4-3, 5.47μm, shrunk approximately 10% whereas 100% PTT sample 4-1, 4.76μm, had zero shrinkage at 200°C. The smaller diameter fibers tend to be more heat stable.

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Chapter I. History

PTT (Poly Trimethylene Terephthalate) was first discovered and patented in 1941 by John Rex Whinfield and James Tennant Dickson **[10]**. The two British scientists were conducting research on polyesters at Calico Printing Ink during the time of World War II. From their British patent PET (poly ethylene terephthalate) and PBT (poly butylene terephthalate) became popular polymers even unto this day.

PTT (poly trimethylene terephthalate) is a mixture of Terephthalic Acid (PTA) and 1,3 Propanediol (PDO) thus, a thermoplastic, aromatic polyester. Even though PTT was first discovered and processed in 1941, it was not until the 1990's that PTT could be processed at a reasonable cost. Shell Chemical Company, based in Houston, Texas, has patented the process for making PDO, which consequently is a key product for making PTT. This new PTT is trademarked by Shell as CORTERRA. Shell also licenses CORTERRA to those who want to process PTT.

PTT has been determined to fit itself into the polyester group between PBT and PET. Some of the character traits of polyesters are electrical insulation, toughness, moisture resistance, chemical resistance, and dimensional stability **[7]**. PTT is more heat stable than PET and PBT. Many properties show desirable traits that would fill a niche market well. PTT has a lower melting point than does PET. PET crystallizes very slowly; thus, it is poor in molding or other applications where a certain time is required. PTT crystallizes slower than PBT. PBT crystallizes too fast and loses modulus due to the T_g being 25°C. PTT has a soft hand. Some of the uses for PTT are films and nonwovens applications. PET is a rival or a controlling factor in these areas due to cost. PET is

cheaper to produce. More recently, PTT raw material costs have declined to the point where it is competitive with PA-6.

PTT does very well in the carpet industry, monofilaments, thermoplastic applications, textile, and engineered thermoplastics. Because of this niche market, or should it be said this void area, PTT is being looked at for diaper material, hospital gowns, and other applications where pathogenic protection is desired.

There have been numerous experiments on PTT. Whether PTT has been used as a toothbrush filament or as a structural member, this polymer is finding a way into the American home and the international market **[13]**. Many patents are based upon PTT, whether it is a process patent or the product itself. Not much is known of PTT as a meltblown nonwovens product. Extensive research has been conducted on the heat capabilities of PTT **[1]**. Spunbond research has been previously conducted and documented on PTT here at UT Knoxville and in Florida at Hills, Inc **[12]**.

PTT is produced by hydroformylation. Ethylene oxide is combined with Syngas, a Shell Chemical product, to make 1,3 Propanediol (PDO). This PDO is then mixed with poly terephthalic acid (PTA) unto a direct esterification under pressure and then followed by a melt polymerization between 250°C - 275°C under vacuum **[10]**.

Chapter II. PTT As A Carpet Fiber [5, 8, 10]

PTT is an aromatic polyester created from the closed-chain class of organic compounds or benzene derivatives. If one combines Figure 2 to Figure 3 then, that process would equal Figure 1, which is the repeat unit for PTT. PTT has some of the qualities desired from PA, nylons, and PET. Thus far PTT has been directed toward the carpet industry and the textile industry. Spunbonding offers a unique look at PTT.

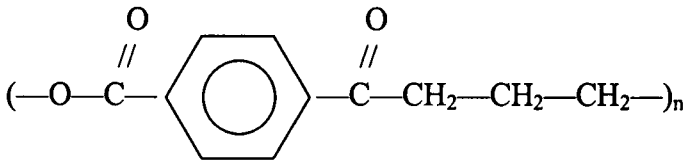


Figure 1. (Repeat unit of chemical structure of PTT)

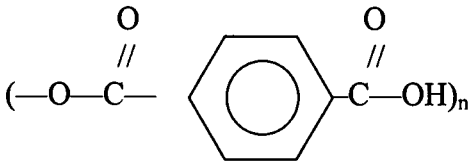


Figure 2. (Repeat unit of terephthalic acid)

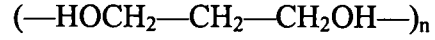


Figure 3. (Repeat unit of 1,3 Propanediol) **[10]**

For spunbonding, polyproplyene (PP) is preferred because of the low cost in which to purchase the polymer. PP is a good choice because many items or products can be extruded or spunbond from it. The major problem with PP is the inability to sterilize effectively. The soft hand that is desired from a yarn, fiber, etc. is not present in PP.

In keeping with the spirit of PTT, the polymer was processed as a 50/50 mixture of methylene chloride and trifluoroacetic acid to give an intrinsic viscosity of 0.9 dl/g. PTT was spunbond at Hills Inc. in West Melbourne, Florida and at TANDEC on the campus of the University of Tennessee in Knoxville, Tennessee. In this particular experimental research Exxon 3445 PP was used as a control specimen. PTT partial oriented yarn was drawn into a textured yarn. It exhibited facile extrusion at speeds of 600 m/min to 5000 m/min. When drawn into textured yarns, the drawing speeds range from 450 m/min to 1100 m/min.

During the experiment it was noted that PTT has a spin speed to POY (partially oriented yarn) properties similar to PET POY. However, PTT POY fibers spun at 3000 m/min had a lower elongation and lower tenacity to that of PET POY fibers at 3000 m/min. These property features equal to a softer web and a more pliable web compared to PET. At slower spin speeds, <3000 m/min, PTT is noticeably more amorphous. At faster speeds PTT is more crystalline. PET POY develops modulus as it increases crystallinity whereas PTT modulus remains virtually constant. The spunbond line at Hills Inc., which is the ASON® line, operates at a faster speed than does the Reifenhauser® 2 line at TANDEC. Webs on the ASON® line crystallize quicker and more plentifully. The Reinfenhauser® 2 line at TANDEC cannot duplicate the crystallinity found on the ASON® line. It is believed that a more amorphous spunbond web is to be desired.

There were several comparison tests performed on PP and PTT utilizing established test methods. The first test was tensile properties: tenacity versus spin speed. This shows the relationship between speed and the ability to remain tough, having

structural integrity. It was shown that tenacity increased with speed. PTT crystallizes at higher speeds making the web stiffer and less pliable.

The second test was a comparison of percent elongation versus spin speed. The question asked is this, "Will the stretch of the web decrease with spin speed?" Just as crystallization is apparent in higher speeds so does elongation reduction show to be true. These two physical properties are connected together.

PTT and PTT w/ TiO_2 were used for comparison to PP. To study the affects on the webs, (PTT and PP), different machine conditions were used to control the web. The melt pump was only altered slightly so variability was minimized. The third test between PTT and PP was a thermal mechanical test (TMA). The test proved that PTT was/is more stable than PP. PP showed an area of shrinkage before melt temperature.

In comparison of spunbond lines, increase the spinning speed and the capacity for smaller fibers increases. With this phenomenon, the Reifenhauser® 2 at TANDEC is not able to produce extremely low deniers. Reifenhauser 2 can produce webs that are more amorphous than the webs on the ASON® line **[2]**.

PTT spunbond fibers showed a favorable softness as that of the mechanically spun fibers. PTT can compete against PP. Gamma radiation tests on PTT fared well. PP doesn't handle Gamma radiation or UV radiation nearly as well. PTT was tested at 2.5 and 5.0 Mrad (an ultraviolet measurement). It can be assumed that if PTT can readily accept pigment (dye) then it should also resist UV ray degradation by the treated dye. So far this is a theory and continues to be studied.

Chapter III. PTT As An Engineered Thermoplastic

PTT historically has been very expensive to manufacture. Shell Chemical Company has opened the door to a more economical PTT polymer trademarked as CORTERRA. PTT is in direct competition with PET and PBT as the consumer's choice for aromatic polyester. PTT has been tested with glass-filled formulations proving that the polymer can compete as an engineering thermoplastic against similar PET, PBT, and PA-6 formulated ETP scenarios.

In discussing engineering thermoplastics (ETP), we can divide this population into three distinct groups- crystalline polymers, amorphous polymers, and exotic, high-temperature polymers. Crystalline polymers range in Tm from 200°C to 270°C. These polymers include PA-6. PA-6,6, aromatic polyesters and aliphatic polyketones **[3]**. Amorphous polymers have a Tg above 150°C. The polymers are PC (polycarbonate), polyarylates, polyphenyleneoxide, and polyethersulfones. The exotic polymers have Tm above 270°C and Tg above 200°C. The polymers are polyetherimides, polyimides, liquid crystal polyesters, polysulfides, polybenzimidazoles, and aromatic polyketones.

To be an ETP, it is compared by or compared to many opponents. They are engineering thermosets, metals, and other traditional materials used in the marketplace. PTT can compete favorably in the ETP realm due to the structure of the polymer, its stability, high temperatures, and the ability to rebound from flexural loads. PTT is compared to PET, PBT, PA-6,6 in the amorphous polymer bracket.

Several polymers with a genuine intrinsic viscosity, and varying mixtures of compounds were tested. It was reported that the highest material (labeled IV) displayed the best balance of virgin polymer properties. The lowest (IV) polymer was the most effective in the glass-filled products. This particular polymer was used as the basis of the report given.

To compound the PTT, a 25mm Berstorff ZE co-rotating screw extruder was used. The screw configuration was the least damaging to the polymers. The barrel in which the compounding occurred was maintained at a range of 230°C to 250°C. The resulting melt temperature was approximately 265°C. After compounding, the polymer strands were quenched in cold water and dried thoroughly before injection molding **[3]**.

The injection molding was done on an Arburg Allrounder 221-55-250 mold machine. Mold temperatures ranged from 220°C - 260°C. Injection pressures were 670-1100 p.s.i., holding pressures were 450-600 p.s.i. , and mold temperatures of 70°- 80°C. The cycle times for the virgin polymers (not compounded) were 40 seconds and 30 seconds for the glass-filled formulations. All the tests were performed on samples equalized at 50% humidity and 23°C (room temperature) following ASTM procedures.

PTT was pitted against PA-6, which is the principal and largest volume nylon polymer. PTT was compared to PC (polycarbonate) which is the largest volume amorphous polymer used in ETP applications. In virgin applications, uncompounded, it can be seen that PA-6, and PC are superior to polyesters by their physical properties. The market reflects this. PA-6 has excellent stiffness and PC has outstanding impact strength. In a mono-state, nylon's (PA-6) problem has been dimensional stability in a moist

environment. This situation has led to niche markets for polyesters and polyacetals not affected so much by moist environments **[3]**.

PET, an aromatic polyester, has the greatest potential to be used as a virgin polymer. PET has a high modulus and T_g of 80°C, but it has the poorest crystallization kinetics and due to its slowness, cannot be molded as a virgin with acceptable molding cycles. In keeping with the aromatic polyester family, PBT has a very fast rate of crystallization in molding and a short cycle time but rapidly losses modulus with temperature since T_g for this material is 25°C.

PTT is a better fit due to its T_g of approximately 50°C plus PTT doesn't require excessive temperatures to melt. All this is contingent of PTT in a mono-state. For all the property information, see **Table 1**.

Table 1. Physical and Electrical Properties of Unreinforced ETP Polymers [6]

Physical Property	**PET**	**PTT**	**PBT**	**Nylon 6,6**	**PC**
Tensile strength, MPa	72.5	67.6	56.5	82.8	65
Flexural Modulus, GPa	3.11	2.76	2.34	2.83	2.35
HDT @ 1.8 MPa, C	65	59	54	90	129
Notched Izod Impact, J/M	37	48	53	53	640
Specific Gravity	1.4	1.35	1.34	1.14	1.2
Mold Shrinkage, m/m	0.03	0.02	0.02	0.015	0.007
Melt Temperature, °C	265	225	228	265	
Tg, °C	80	45-75	25	50-90	150
Dielectric Strength, V/mil	550	530	400	600	380
Dielectric Constant 1 MHz	3	3	3.1	3.6	3
Dissipation Factor 1 MHz	0.02	0.015	0.02	0.02	0.01
Volume Resistivity Ohm-cm	1.00E+15	1.00E+16	1.00E +16	1.00E+15	8.20E+16

(Physical properties of 100% virgin polymers.) (1 MPa = 144 p.s.i.)

Chapter IV. Heat Capacity of PTT

PTT like all other polymers and engineered mediums have a quantity known as heat. Not all polymers have the same heat rate, heat capacity, or thermal energy values. What is heat capacity? How important is knowledge of heat capacity? The answer lies in the stability of the polymer for melting, extrusion, induction, and so forth.

From the paper titled "Heat Capacity of Poly (trimethylene terephthalate), the heat capacity of PTT has been measured using adiabatic calorimetry, standard differential scanning calorimetry (DSC), and temperature-modulated differential scanning calorimetry (TMDSC). The heat capacities of the solid and liquid states of semicrystalline PTT are reported from 5 to 570 K. The semicrystalline PTT has a glass transition temperature of 331 K. Between 340 and 480 K, PTT can show exothermic ordering depending on the prior degree of crystallization. The melting endotherm of semicrystalline samples occurs between 480 and 505 K, with a typical onset temperature of 489 K (216°C). The heat of fusion of the semicrystalline samples is about 15kJ/mol. For 100% crystalline PTT the heat of fusion is estimated to be 30 ± 2 kJ/mol. The heat capacity of solid PTT is linked to an approximate group vibrational spectrum and the Tarasov equation is used to estimate the heat capacity contribution due to skeletal vibrations ($\theta 1$ = 550.5 K and $\theta 2 = \theta 3$ = 51 K, N (skeletal) =19). The calculated and experimental heat capacities agree to better than ±3% between 5 and 300 K. The experimental heat capacities of liquid PTT can be expressed by C l/p (exp) = 211.6 + 0.434 T J K^{-1} mol^{-1} and compare to ±0.5% with estimates from the ATHAS data bank using contributions of other polymers with the same constituent groups. The glass

transition temperature of the completely amorphous polymer is estimated to be 310- 315 K with a ΔCp of about 94 J K^{-1} mol^{-1}. Knowing Cp of the solid, liquid, and the transition parameters, the thermodynamic functions enthalpy, entropy, and Gibbs function were obtained. With these data one can compute for semicrystalline samples crystallinity changes with temperature, mobile amorphous fractions, and resolve the question of rigid-amorphous fractions **[1]**.

While this is taken directly from the paper, it reflects on the urgency to know what PTT can do as far as melting, how to process, and to what extent one can work with it. I am most concerned with the melt temperature and glass transition temperature in order to process this polymer on a meltblown processing line. The above paragraph may be a lot of noise to contend with, but the rudiment ideas stand out.

PTT belongs to the aromatic polyesters such as PET, and PBT. PTT has been determined through research that the molecular mass is 206.2. During this research, PTT was found to have an equilibrium melt temperature of approximately 237°C and a glass transition temperature range of approximately 42°C to 72°C.

Thermogravimetry (TGA) showed that PTT is thermally stable. The initial weight loss was detected at around 300°C. A second weight loss of 2% was recorded on heating the polymer at 20 K/min in nitrogen at 350°C; at 400°C a weight loss of 23% occurred. At 450°C the polymer fully decomposed, leaving about 8% of total weight in residue. Vibrational motion of the solid PTT has been determined to be a factor in measuring heat capacity **[1]**.

During the TGA experiment PTT was measured in a 50/50 mixture of methylene chloride and trifluoroacetic acid at 30°C and analyzed as obtained and after various thermal treatments. PTT was heated above its Tm and quenched outside the calorimeter by immersing in liquid nitrogen with different speeds of transfer.

Three calorimeters were used for measurement of the transition behavior and heat capacities: A DSC 820 from Mettler Toledo Inc. (Toledo, OH), a MDSC 2920 from TA Instruments, Inc. (New Castle, DE) for TMDSC measurements, and an adiabatic calorimeter for low-temperature measurement. The DSC and TMDSC are of the heat-flux type. Heat capacities of PTT from 5 to 330 K were measured with an adiabatic vacuum calorimeter. Liquid helium and nitrogen were used to obtain low temperature in the cooling system. Heat capacities were calibrated with benzoic acid standard before the measurements of PTT. The precision was estimated to be ± 0.5% from 5 to 330K.

The measurement of heat capacity by standard DSC was carried out a heating rate, {q}, of 10 K/min After steady state was attained, the heat capacity was determined from the following equation:

$$mc_p = K\frac{HF}{q}$$

where K is determined as a function of temperature from the sapphire calibration, HF is the heat flow (proportional to the temperature difference, ΔT, between reference and sample) and {q} being the heat rate. The effect of calorimeter asymmetry and differences between empty references and sample calorimeters are eliminated by using the empty pan run as a baseline for the heat flow amplitudes. For measurements of heat

capacity by standard DSC, 20-30 mg of sample were employed. For transition measurements the samples weighed 2-6 mg. The smaller samples permitted keeping the steady state closer to the melting peak. If one uses a sample size larger than 10 mg then there could possibly be overflow after the Tm has occurred.

When the DSC was used at 10 K/min, the transition temperature, T_g, of the semi-crystalline PTT occurred at 331K with a ΔCp of 40 J K^{-1} mol^{-1}. The semi-crystalline PTT shows a much smaller ordering exothermal between 460 and 480 K. The melting endotherm occurs between 480 and 505 K with an onset temperature (actual phase change) T_f = 489 K (216°C) **[1]**.

Above the glass transition, rigid-amorphous and crystalline phases may exist in addition to the mobile-amorphous phase. The heat capacity after the glass transition, however, does not follow the expected semi-crystalline path; rather it increases further and, finally, displays a pre-melting peak at approximately 450 K.

Chapter V. Process Conditions

Previous research has been conducted on spunbond nonwoven webs at TANDEC at UT Knoxville and at Hill, Inc. in West Melbourne, FL. Some rudimentary research on meltblown webs was conducted at TANDEC. However, a study of meltblown PTT based mono and bi-component webs have not been exclusively researched until now. Comparisons have been drawn up between the polyester group to which PTT belongs. Characterization of PTT meltblowns are in the form of DSC, MFR, hydro-head, air permeability, drape ability, elastic recovery, tensile properties, heat shrinkage, and SEM-fiber diameter.

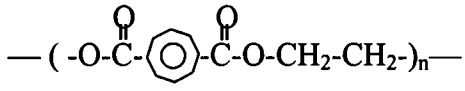
The beginning of the research focused on two types of PTT and one PET sample. The first PTT sample was of a virgin origin cataloged by Shell Chemical Company as VFR 50008. The second PTT sample had TiO_2 compounded to lower the luster. TiO_2 also increased the workability of the PTT and it was cataloged by Shell as VFR 50009. The PET sample used is cataloged as Wellman PET. This PET sample represents the main competitor for PTT.

The performance of meltblown webs depends on many processing conditions, but the primary control variables that influence the web properties are melt temperature, air temperature, melt throughput, air flow rate, and die-to-collector distance (DCD). In addition, the resin itself and die geometry parameters such as die orifice diameter, L/D ratio, nosetip angle, air gap, and nosetip setback, etc. have notable effects on the web formation. A series of mono PTT and bico PTT/PP samples were produced on the Reicofil® line based on the designed trials based on Surface Response Methodology [29] (SRM) with the die orifice diameter as 0.4mm and Air Gap/Setback as 0.8mm/1.0mm.

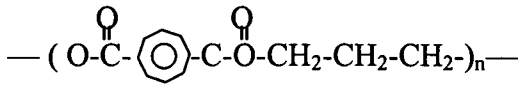
The PTT polymer 50009 was processed on Reicofil 24-inch wide Bico Meltblown Line at TANDEC in March 2000. The webs were thin and not uniform, due to the spinneret die holes being partially plugged during that experimental period even though a new die tip was replaced. The plugged holes created uneven webs and streaks oriented in the machine direction. The soft hand was apparent but static electricity was evident. Though the basis weights of the 37 samples were in a range of 34.3 to 48.4 g/cc. In April 2001, a new set of PTT mono and bico webs were processed at TANDEC. These webs faired better due to the spinneret holes being clean.

PTT is being compared to PET, PBT, and PA-6. These polymers are very similar in composition with the exception of PA-6. The chemical structures are:

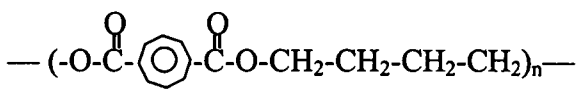
PET



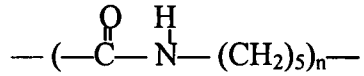
PTT



PBT



PA 6



PET, PBT and PTT are all aromatic polyesters. The main difference between the three is an extra CH_2 group in the repeat unit. PET has two CH_2, PTT has three CH_2, and PBT has four CH_2 groups. PA-6 or nylon-6 by its trade name is a close competitor due to the physical properties common to the aromatic polyesters. Currently PET is still the most economical polymer to purchase but PTT has dropped its cost to that of the PA-6. This cost reduction should help to market PTT even better. All the testing conducted on PTT is to show how well PTT competes with the most popular polymers in the field. PTT has been touted as the new polymer to replace PET with its soft hand, superior dying capability, greater heat resistance, low static charge, and stain resistance.

Each test is separated into its own section for ease of reading and analyzing. Characterizations from the first PTT (3/2/2000), PTT (2/5/2001), and the other polymers can be viewed in their appropriate sections. See **Tables 2-3** for process conditions for the March 2000 processing. See **Table 4** for process conditions for April 2001 processing. **Tables 5-8** show the characterizations of the research.

Table 2. Processing Conditions A) Mono PTT Meltblown Webs (Shell VFR 50009)
(Air gap/Setback = 0.8mm/1.0mm, March 2, 2000)

Sample No.	Melt Temp		Through-Put g/h/min	Air Temp		Air Flow Rate (SCFM)	DCD (in.)
	(°F)	(°C)		(°F)	(°C)		
1	520	271	0.9	490	254	500	15
2	535	279	0.6	470	243	400	17
3	535	279	0.6	510	266	600	13
4	535	279	0.6	470	243	400	13
5	535	279	0.6	510	266	600	17
6	535	279	1.2	470	243	400	13
7	535	279	1.2	510	266	600	17
8	535	279	1.2	470	243	400	17
9	535	279	1.2	510	266	600	13
10	550	288	1.2	490	254	500	15
11	550	288	0.9	490	254	500	15
12	550	288	0.9	490	254	300	15
13	550	288	0.9	490	254	700	15
14	550	288	0.9	450	232	500	11
15	550	288	0.9	530	277	500	19
16	550	288	0.9	490	254	500	15
17	550	288	0.9	490	254	500	15
18	550	288	1.5	490	254	500	15
19	565	296	0.6	470	243	400	13
20	565	296	0.6	510	266	600	17
21	565	296	0.6	470	243	400	17
22	565	296	0.6	510	266	600	13
23	565	296	1.2	470	243	400	17
24	565	296	1.2	510	266	600	13
25	565	296	1.2	470	243	400	13
26	565	296	1.2	510	266	600	17
27	580	304	0.9	490	254	500	15

Table 2. Processing Conditions B) for Bico PTT/PP Meltblown Webs
(Shell VFR 50009/Exxon 3546G, Air gap/Setback = 0.8mm/1.0mm)

Sample No.	PTT/PP Ratio	Melt Temp (°C)		Air Temp (°F)	Air Temp (°C)	Throughput g/h/m (PET, PE)	DCD (in.)	Air Flow Rate (SCFM)
		PTT	PP					
1a	75/25	274	257	500	246	0.9	12	400
1b	75/25	274	257	500	246	0.9	14	500
1c	75/25	274	257	500	246	0.9	12	600
1d	75/25	274	257	500	246	0.9	19	500
2a	50/50	274	257	500	246	0.6	8	300
2b	50/50	274	257	500	246	0.6	10	400
2c	50/50	274	257	500	246	0.6	12	500
3a	25/75	274	257	500	246	0.3	10	400
3b	25/75	274	257	500	246	0.3	10	300
3c	25/75	274	257	500	246	0.3	8	300

Table 3. Characterization of PTT Mono and Bico Webs

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. ($ft^3/ft^2/min$)	Hydro-head (cm)
				Ave. (μm)	C.V. (%)					
3/2/00-1	43.0	0.375	0.115	4.76	27.4	0.12	58.54	1.08	314.6	12.03
3/2/00-2	43.4	0.511	0.085	3.84	18.1	0.08	88.10	0.71	318.8	13.10
3/2/00-3	43.2	0.369	0.117	2.86	25.6	0.24	30.95	2.15	128.6	19.20
3/2/00-4	46.0	0.445	0.103	2.88	30.8	0.07	49.04	0.59	161.6	15.93
3/2/00-5	48.0	0.430	0.112	2.87	22.8	0.20	33.18	1.61	132.8	17.07
3/2/00-6	43.9	0.446	0.098	3.77	28.2	0.16	52.98	1.41	321.0	9.43
3/2/00-7	43.4	0.428	0.101	2.87	26.6	0.20	40.28	1.78	266.0	10.23
3/2/00-8	44.8	0.540	0.083	3.83	25.4	0.13	68.06	1.12	411.6	9.13
3/2/00-9	43.3	0.385	0.112	2.41	23.6	0.28	34.58	2.50	212.2	9.60
3/2/00-10	48.4	0.441	0.110	2.56	40.9	0.25	29.80	2.00	85.8	13.77
3/2/00-11	45.1	0.445	0.101	2.98	22.3	0.14	41.64	1.20	186.2	9.97
3/2/00-12	43.0	0.518	0.083	3.99	27.5	0.14	81.60	1.26	346.6	9.13
3/2/00-13	44.3	0.381	0.116	2.39	21.6	0.37	26.50	3.23	132.4	11.23
3/2/00-14	42.0	0.378	0.111	2.99	28.8	0.26	27.06	2.39	149.0	11.53
3/2/00-15	46.0	0.522	0.088	2.67	31.0	0.13	50.88	1.09	201.2	9.90
3/2/00-16	44.5	0.422	0.105	3.24	19.7	0.14	38.18	1.22	170.0	10.00
3/2/00-17	42.2	0.406	0.104	3.18	20.0	0.17	33.22	1.56	168.8	9.93
3/2/00-18	42.4	0.419	0.101	3.22	29.6	0.19	34.60	1.73	239.4	9.67
3/2/00-19	43.4	0.394	0.110	3.04	22.4	0.15	20.36	1.34	158.4	9.50
3/2/00-20	44.8	0.398	0.113	2.02	25.2	0.24	26.98	2.07	110.5	11.40

Table 3.Continued

Sample ID	Basis Wt (g/m2)	Thickness (mm)	Bulk Den. (g/cm3)	Fiber Diameter		Peak Load (kg)	Break Ext. (%)	Tenacity (mN/tex)	Air Perm. (ft3/ft2/min)	Hydro-head (cm)
				Ave. (mm)	C.V. (%)					
3/2/00-21	46.2	0.436	0.106	3.36	19.0	0.11	25.14	0.92	112.8	9.90
3/2/00-22	44.4	0.376	0.118	2.21	22.1	0.42	15.52	3.65	71.4	11.77
3/2/00-23	42.8	0.491	0.087	3.00	23.2	0.10	32.02	0.90	192.8	9.13
3/2/00-24	41.7	0.371	0.112	2.22	22.4	0.36	24.70	3.33	133.4	11.03
3/2/00-25	39.8	0.404	0.099	2.83	20.5	0.16	32.72	1.55	203.4	9.53
3/2/00-26	43.1	0.428	0.101	2.51	27.4	0.24	30.26	2.15	150.8	10.30
3/2/00-27	42.8	0.429	0.099	3.01	20.0	0.29	30.18	2.62	128.2	10.50
3/2/00-bico1a	39.8	0.363	0.110	3.00	22.5	0.24	12.10	2.33	110.2	38.77
3/2/00-bico1b	40.4	0.370	0.109	2.47	26.4	0.30	10.42	2.87	90.2	40.37
3/2/00-bico1c	36.5	0.329	0.111	1.71	21.4	0.32	11.82	3.39	80.3	42.00
3/2/00-bico1d	42.5	0.463	0.092	2.27	25.6	0.16	20.32	1.45	82.3	31.77
3/2/00-bico2a	34.3	0.307	0.112	2.22	24.9	0.53	37.02	5.97	95.6	41.30
3/2/00-bico2b	36.5	0.355	0.103	2.12	23.9	0.36	15.74	3.81	82.2	45.87
3/2/00-bico2c	38.6	0.389	0.098	1.92	24.1	0.29	14.04	2.90	85.2	45.17
3/2/00-bico3a	42.1	0.392	0.107	1.98	26.1	0.39	10.96	3.58	53.1	59.10
3/2/00-bico3b	38.6	0.355	0.111	2.34	30.7	0.46	15.10	4.60	73.5	52.10
3/2/00-bico3c	35.5	0.319	0.116	2.20	23.4	1.18	12.32	12.84	62.5	59.17

Table 4. Processing Conditions for PTT and PTT/PP Meltblown Webs (Air gap/Setback = 0.8mm/1.0mm, Feb 5, 2001)

Sample Number	Polymer ratio (PTT/PP)%	PTT Temp (°C)	PP Temp (°C)	Air Temp (°C)	Throughput g/h/m	DCD (inchs)	Air Flow Rate (SCFM)
3-1	75/25	262	246	257	0.9	8	350
3-2	75/25	262	246	257	0.9	10	350
3-3	75/25	262	246	257	0.9	10	400
3-4	75/25	262	246	257	0.9	10	450
3-5	50/50	262	246	257	0.6	8	350
3-6	50/50	262	246	257	0.6	10	350
3-7	50/50	262	246	257	0.6	10	400
3-9	25/75	262	246	257	0.3	8	350
3-10	25/75	262	246	257	0.3	10	350
4-1	100	262	246	257	1.2	8	300
4-2	100	262	246	257	1.2	10	400
4-3	100	262	246	257	1.2	10	500

Table 5. Properties of PTT Mono-and Bico Meltblown Webs

Sample ID	Sample Description	BW (g/m^2)	Bulk Den (g/m^3)	Hydrohead (cm)	Air Perm ($ft^3/ft^2/min$)	Fiber Dia (μm)	Drape		Elasticity Recovery%
							Side A %	Side B%	
3-1	75/25 PTT/PP	44.02	0.118	49.22	70.86	2.4	49.98	58.47	*failure*
3-2	75/25 PTT/PP	45.88	0.126	42.08	87.3	3.51	40.52	43.54	3.74
3-3	75/25 PTT/PP	46.97	0.117	42.97	77.68	3.06	41.59	48.9	3.4
3-4	75/25 PTT/PP	47.74	0.114	49.85	76.18	3.63	39.73	50.62	4.4
3-5	50/50 PTT/PP	43.09	0.116	51.64	71.52	3.67	54.95	67.15	1
3-6	50/50 PTT/PP	44.8	0.111	50.11	71.38	3.27	48.89	55.16	3.28
3-7	50/50 PTT/PP	43.25	0.106	55.21	65.88	3.38	44.61	57.09	2.2
3-9	25/75 PTT/PP	39.22	0.104	61.07	53.48	3.93	59.89	75.22	0
3-10	25/75 PTT/PP	39.37	0.094	61.33	52.02	4.11	50.03	62.35	6.8
4-1	100% PTT	55.34	0.145	21.55	104.6	4.76	56.02	65.7	5.6
4-2	100% PTT	55.03	0.126	24.61	105.8	5.52	52.04	65.53	3
4-3	100% PTT	50.69	0.134	28.18	74.88	5.47	54.64	56.26	0

Table 6. Tensile Properties of PTT Mono-and Bico Webs

Test Name	Sample Description	Basis Wt (g/m^2)	Thickness (mm)	Bulk Den. (g/cm^3)	Peak Load (kg)	Break Ext. (%)	Tenacity (mN/Tex)
3-1	75/25 PTT/PP	44.02	0.373	0.118	0.44	16	0.8
3-2	75/25 PTT/PP	45.88	0.363	0.126	0.29	15.6	0.5
3-3	75/25 PTT/PP	46.97	0.4	0.117	0.36	19.5	0.6
3-4	75/25 PTT/PP	47.74	0.42	0.114	0.32	17	0.6
3-5	50/50 PTT/PP	43.09	0.372	0.116	0.51	25.9	1
3-6	50/50 PTT/PP	44.8	0.405	0.111	0.3	17.5	0.5
3-7	50/50 PTT/PP	43.25	0.407	0.106	0.4	14.5	0.8
3-9	25/75 PTT/PP	39.22	0.378	0.104	0.57	18.5	1.2
3-10	25/75 PTT/PP	39.37	0.419	0.094	0.43	17.3	0.9
4-1	100% PTT	55.34	0.383	0.145	0.56	28.1	0.8
4-2	100% PTT	55.03	0.438	0.126	0.26	36.3	0.4
4-3	100% PTT	50.69	0.379	0.134	0.38	34.2	0.6

Table 7. Properties of Samples Prior to Heat Shrinkage

Sample ID	Sample Description	BW (g/m^2)	Bulk Den (g/m^3)	Bending (cm)	Hydrohead (cm)	Air Perm ($ft^3/ft^2/min$)	Fiber Dia. (μm)	Drape (%)		Elasticity Recovery (%)
								Side A	Side B	
I	100% PBT	52.3	0.142	4.47	46.41	57.1	3.34	76.73	81.9	0.4
II	75/25 PBT/PP	21.46	0.123	3.65	68.3	63.1	3.41	n/a	n/a	0
III	25/75 PBT/PP	23.7	0.132	4.73	71.4	53.8	3.24	60.9	69	10
IV	50/50 PBT/PP	50.6	0.132	3.58	55.8	58.9	3.12	88.27	89.5	3.8
V	100% PA-6	52.2	0.124	3.12	16.32	86.3	3.08	53.75	55.13	0
VI	75/25 PA-6/PP	52	0.146	3.64	48.69	67.42	3	n/a	n/a	5.4
VII	50/50 PA-6/PP	53.2	0.126	4.02	42.34	69.86	3.45	75.27	81.73	2
VIII	25/75 PA-6/PP	50.8	0.12	4.47	49.96	55.45	2.78	n/a	n/a	0
XIII	100% PET	53.8	0.099	3.12	14.79	150	10.49	56.37	55.1	13
XIV	75/25 PET/PP	49.4	0.114	3.44	31.65	154.19	3.78	79.4	80.83	6.8
XV	50/50 PET/PP	50	0.108	4.47	34.09	110.08	4.21	89.13	91.5	11.4
XVI	25/75 PET/PP	52.3	0.116	4.53	39.47	64.1	4.49	85.3	92.53	9.8

Table 8. Tensile Properties Prior to Heat Shrinkage

Sample ID	Sample Description	Basis Wt (g/m^2)	Thickness (mm)	Bulk Den. (g/cm^3)	Peak Load (kg)	Break Ext. (%)	Tenacity (mN/Tex)
I	100% PBT	52.30	0.368	0.142	0.28	12.7	0.4
II	75/25 PBT/PP	21.46	0.174	0.123	0.67	5.5	11.9
III	25/75 PBT/PP	23.70	0.179	0.132	0.50	10.6	6.9
IV	50/50 PBT/PP	50.60	0.383	0.132	1.11	26.6	8.3
V	100% PA-6	52.20	0.420	0.124	0.60	32.2	1.1
VI	75/25 PA-6/PP	52.00	0.356	0.146	1.26	29.6	9.0
VII	50/50 PA-6/PP	53.20	0.422	0.126	1.43	44.4	9.7
VIII	25/75 PA-6/PP	50.80	0.423	0.120	1.62	46.8	8.8
XIII	100% PET	53.80	0.543	0.099	0.42	32.5	0.6
XIV	75/25 PET/PP	49.40	0.433	0.114	0.93	43.1	5.7
XV	50/50 PET/PP	50.00	0.463	0.108	1.24	32.2	9.3
XVI	25/75 PET/PP	52.30	0.451	0.116	1.39	26.1	10.0

Chapter VI. Characterization by DSC

The Mettler Toledo model DSC 25 digital scanning calorimeter was used to determine the melt temperature phase change and the glass transition temperature of the PTT and its competing polymers used in the experiment. Several PTT samples were tested along with PET, PBT, PA-6. The heating rate of DSC was 10°C/min in a temperature range from 25°C to 300°C. The measured thermal Properties of the polymers are shown in **Table 9**.

Table 9. Thermoplastics Used in Fiber Production

Properties	PTT (Shell VFR 50009)	PET (Wellman)	Nylon 6 (BASF B 3)	PBT (Ticona Celanex 1300A)
Melt Point (°C)	232	265	226	228
Glass Trans (°C)	45-65	80	40-87	29
Density (g/cc)	1.33	1.40	1.13	1.41

Chapter VII. Characterization by MFR

The Tinius Olsen Model MP 987 Melt Indexer was used to establish a melt flow rate value for the polymers used in this PTT research and other comparisons. Melt flow rate (MFR) or melt flow index (MFI or MI) was measured to help determine the processing temperature. MFR is widely used in plastic industry to describe the fluidity of a polymer melt. It is a simple flow value of the amount of material extruded at a standardized temperature through a die under pressure from a set mass over a period of 10 minutes. The melt at the higher temperature is easier to flow and corresponds to higher MFI. We did the test under the processing temperature. For meltblown processing a MFR of 400-1200 is not too uncommon. The resin is usually not recommended for the industrial production if the MFR value is too low (<100) at the processing temperature, or the processing temperature should be increased to improve its flow ability as long as no oxidation occurs.

The MFR testing process was tedious to say the least. Each sample was tested five times, as specified in ASTM D1238-89, in order to obtain an average. The first tests were conducted on samples as they were. The measured MFR was unusually high due to moisture in the polymers (Table 10), indicating that the melt spinnability was destroyed to a large extend by the polymer degradation and oxidation. The decision was made to analyze the moisture content of the samples using a moisture content analyzer at TANDEC and determine a drying session for the samples. PTT VFR 50009 was determined to have moisture content of 0.277%. PTT VFR 50008 had moisture content of 0.364%. Wellman PET had moisture content of 0.464%. Whereas the moisture

content normally has to be not greater than 0.03% for the meltblown production. It was determined that we should dry the polymers. First, the drying conditions were set for three hours at 100°C. This proved insufficient so a new batch of samples was dried for three hours at 120°C. The drying session proved to be the correct procedure. In normal industrial production, polymers are dried then processed through the meltblown production line. One can compare **Table 10** with **Table 11** and see how drying affects the polymers.

In order to calculate the MFR by using the Tinius Melt Indexer we use a formula suited to the polymer. PTT is very similar to PP. ASTM does not have a MFR value for PTT so we use a ratio to solve the formula. PP has a constant value of 801. PE has a constant value of 828. The formula for MFR is thus:

$$MFR = (801/\text{time elapsed}) \times \frac{\text{(density of test melt)}}{\text{(density of PP melt)}}$$

Thus by looking at **Table 11** we can see that PTT has a slightly higher MFR at 270°C than does PET at 300°C. If one desires to compare at the same temperature, then PTT wins hands down with a MFR of 885 and 869, respectively. PET only has 375 MFR at 300°C thus PTT can perform better as a meltblown than PET at a cheaper cost due to the energy needed to sustain a high MFR.

Table 10. MFR Measured Before Drying

Polymer	MFR (g/10min)	Temp (°C)
PTT (Shell VFR 50008)	2155	300
PTT (Shell VFR 50009)	1385	300
PET (Wellman)	1949	300

Table 11. MFR After Drying at 120°C at 3 hours

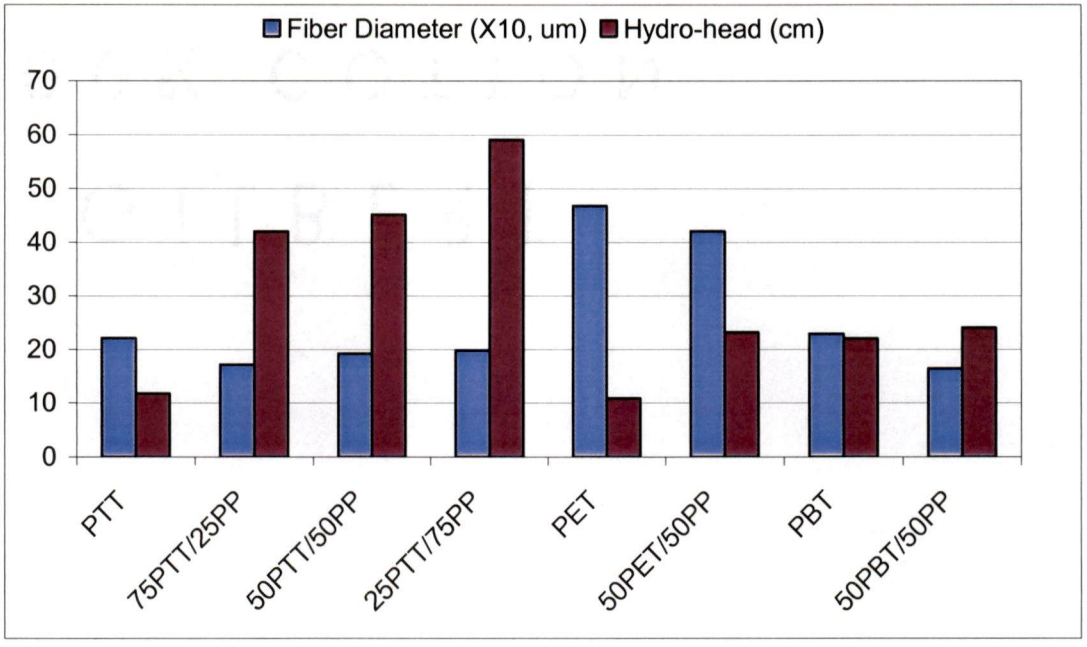
Polymer	Temp °C	MFR (g/10min)	Moisture Content Before Drying	Moisture Content After Drying
PTT (Shell VFR 50009)	270	383.6	0.218%	0.030%
	300	884.8		
PTT Shell VFR 50008	270	353.6	0.364%	0.007%
	300	868.8		
PET (Wellman)	300	375.2	0.464%	0.000%
Nylon 6 (BASF B3)	280	85.0	0.510%	0.030%
PBT (Ticona Celanex 1300A)	280	216	0.350%	0.020%

Chapter VIII. Characterization by Hydro-Head

The Textest FX 3000 hydro-head tester was used for the research conducted on the PTT (3/2/2000), PTT (2/5/2001), PET, PBT, PA-6 samples. The purpose of such testing is to provide data showing were the nonwoven web begins to take on water. The hydro-head test will tell us the barrier property of the web.

By following INDA Standard Test IST 80.6 (98), the unit measures the water pressure on the web in milli-bars. During the test when three separate bubbles appear the test is stopped. The pressure reading is documented and converted to inches. The conversion from mbars to centimeters is, 1.02 x (milli-bar value). In **Figure 4**, the increase of polypropylene, PP, shows an increase of hydro-head thus making the barrier property of the bico web better than the single component web. There is not enough conclusive evidence across the Figure to determine if hydro-head is affected by fiber diameter. However, one can see that the smaller fibers of the PTT/PP bicomponents have a greater hydro-head effect. The PET fibers were not so forgiving.

PTT (3/2/2000) and PTT (2/5/2001) have similar hydro-head values. If one increases the fiber diameter size then the hydro-head decreases. This is explained by the larger fibers not meshing closely with its neighbor thus making the web more prone to fluid entrapment and fluid flow.



(Melt throughput = 0.6 g/hole/min)

Figure 4. Hydro-head and Fiber Diameter for Bico Meltblown Webs

Chapter IX. Characterization by Air Permeability

The German made Textest 3300 Air Permeability Tester was used for the air permeability testing. The purpose of Standard Test Method, ASTM D737-96 Air Permeability of Textile Fabrics is to determine how much air passes through a nonwoven web. Depending on the amount of air exchange, a nonwoven can be an effective barrier or an excellent filter. The breathability of a nonwoven is a selling point for marketing plans.

Fiber size and fiber configuration affects the flow of air through the web. Look at the characterization table and the corresponding fiber diameter sizes. By looking at **Figure 7**, page 40 in chapter XIV one can see how air permeability can be affected by fiber diameter and polymer composition before and after heat shrinkage. For further explanations on air permeability see chapter XIV.

Chapter X. Characterization by Drape (drape ability)

The drape behavior of a fabric is one of those selling points a clothing manufacturer exploits for marketing of its product. For PTT meltblown research, drape of the nonwoven web is a characteristic that was researched. A drapemeter tester was used for the experiment. By following the guidelines stipulated by the manufacturer of the drape meter and by EDANA 90.1-76 Nonwovens Drape Test, tests were performed on three individual 10" diameter web samples. Each side was documented, tested and used to determine if a pattern existed between the collector belt side and the exposed side.

PTT (2/5/2001) samples and PET, PBT, PA-6 samples were tested. See Figure 7 for details. We found that the collector side, called "A", truly had a different drape as does the open side, which was called "B". The collector side showed less drape due to the compression and compaction of the fibers onto the collector belt.

In formulating a value for drape, the formula used was based on the manufacturer's ratio of weights.

$$F = \frac{(W_3 - W_2)}{(W_1 - W_2)} x100$$

The drape is determined by the weight of the tracing paper used in the test. Here is how it works. A 10" diameter nonwoven web is the sample. Cut three pieces per sample for the test. Make an incision in the center of each piece. The drape meter tester requires tracing paper to be used. Cut a 10" diameter circle of tracing paper for each side

tested. This would be six pieces cut. Label the tracing paper pieces as W1. Cut a four-inch piece of tracing paper and place a small hole in the center. Label it W2.

Place the nonwoven sample onto the platform of the drapemeter. Place W2 on top of the sample. Place W1 on top of the drapemeter aligning it centered over the sample. Let the sample drape for five minutes. When the time is up, turn on the light and trace the web's silhouette onto W1. Remove W1 and weigh it documenting the value. Now cut the silhouette out and label the new piece as W3. Dispose of the outside edge, weigh W3 and document the value. Weigh W2 and document the value. Values for W1-W3 will be used in the formula above.

When F is solved that means a percentage of weight has been draped over the edge. **Table 5**, page 21 gives the drape percentages of the PTT (2/5/2001). It is important to remember that when one looks at the values in the chart, it depicts how much the web did not drape. The greater the percentage value, the less the nonwoven web draped. See **Figure 5** for a better look at drape on the PTT (2/5/2001) samples. The bottom line on the Figure is the collector side down. That is to say that when the test was performed the collector side was placed face down on the instrument.

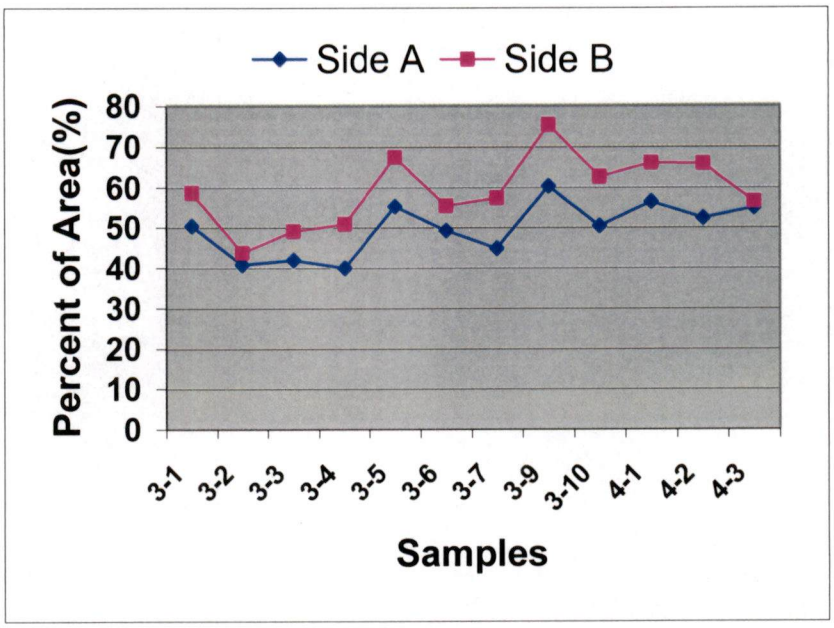


Figure 5. Drape of PTT and Bico PTT Meltblown Webs

Chapter XI. Characterization by Elasticity Recovery

The flexural rigidity of a web tells us how a sample can return to its original position. Elastic Recovery (E.R.) describes how much the web can withstand before permanent deformation. E.R. tests were conducted on the PTT (2/5/2001) samples. PTT (3/2/2000) samples were not suitable due to the web construction via plugged spinneret holes.

The E.R. is based upon the standard tensile test of meltblown webs via United Tensile Tester. The test begins as usual with exception of the web being marked prior to testing. The test concludes when the extension reaches 10%. Remove from the tensile tester and allow the sample to rest for at least two minutes before measuring the length. Theoretically, a new length occurs after the test.

To determine the E.R., take five samples and measure the (Δ) change in length. If the length change is0.014 averaged then multiply by 100 for the E.R. percentage. E.R. translates to percentage of original length that is permanently deformed. In other words, E.R. could be the percent of length that is changed. When a sample has an E.R. of zero (0) then that means simply that at 10% tensile extension the web has an elastic behavior.

By nature, meltblown webs and polymers have visco-elastic properties that show permanent change to the web structure. **Table 12** details the results of the E.R. test.

Meltblown webs are notorious for having low tensile properties. They also exhibit low recovery. Several samples were tested to the ten percent mark and were found to break. However, at low tension the webs do show some recovery or memory of the original length.

Table 12. Elasticity Recovery of PTT Based Meltblown Webs

Sample #	Sample Description	E.R. (%)	New length (in)
3-1	75/25 PTT/PP	Failed	n/a
3-2	75/25 PTT/PP	3.7	5.2
3-3	75/25 PTT/PP	3.4	5.2
3-4	75/25 PTT/PP	4.4	5.2
3-5	50/50 PTT/PP	1.0	5.1
3-6	50/50 PTT/PP	3.3	5.2
3-7	50/50 PTT/PP	2.2	5.1
3-9	25/75 PTT/PP	0.0	5.0
3-10	25/75 PTT/PP	6.8	5.3
4-1	100% PTT	5.6	5.3
4-2	100% PTT	3.0	5.2
4-3	100% PTT	0.0	5.0

Chapter XII. Characterization by Tensile Properties

The United Tensile Tester was used to conduct tests upon the PTT (3/2/2000), PTT (2/5/2001), and PET, PBT, PA-6 samples. For a view of the characterization results see **Tables 2-4**, pages 16-20, in chapter V. Tests were performed under the guidelines of ASTM D5035-95 Standard Test Method for Breaking Force and Elongation of Textile Fabrics (Strip Method).

Mono web samples (100%) have greater break extension than webs containing a bicomponent. With the introduction of PP, the break extension dramatically drops. The bico webs do not have the luxury of stretching before breakage.

However, with the increase of PP, the webs become stronger but brittle. PP is the limiting factor for elongation, but the deciding factor in strength. The next section deals with tensile properties after heat shrinkage. There one can see a change in behavior of the web.

To facilitate the tensile test required a ten-pound load cell to be attached to the United Tensile Tester. The ten-pound load cell is ideally suited to meltblown because one rarely exceeds ten pounds of force when pulling apart the web sample. The sample pieces were cut into ten inch by one-inch strips. However, if a six-inch strip was only accessible then the clamp distance could be adjusted prior to operation. Normally five samples in the machine direction and eight samples in the cross direction are used in tensile testing. With meltblown, cross direction samples are not necessary because of the web structure. Ten samples in the machine direction are sufficient. The samples are

tested then averaged giving data in break elongation, peak load, and tenacity. This data is used to give an explanation of why the web acted the way it did.

Chapter XIII. Characterization by Heat Shrinkage

Previous research has shown the importance of determining the melt temperature, glass transition and the overall heat capacity of the polymer. As pertaining the physical characterization, shrinkage is a phenomenon that needs to be made available. In regard to PTT as a fabric, the nonwoven PTT webs were tested to see how much the sample areas shrunk.

One of the first things that a consumer does is to check the content label of the clothing. Whether it is 100% cotton, cotton blend, or polyester we look for information alluding to shrinkage. If the label denotes pre-shrunk cotton then we are confident the shirt will not change its size. If the garment says 100% cotton then we anticipate shrinkage after the first wash. Cotton blends will not shrink along with polyester, nylon types. We are confident when we read the label.

PTT, or should we say CORTERRA, has been described as heat stable. It was decided that PTT would be heat shrunk to gauge the difference between heat shrunk PTT and heat shrunk PET, PBT, PA-6.

We can see that PTT is truly more stable than PET. One explanation is that the repeat unit of PTT has one more CH_2 group allowing for a more rigid alignment than the PET molecular unit. See **Figures 6-13**, below on PTT (3/2/2000). With the increase of PP into the bico webs, we see some shrinkage appear. At 190°C PTT (2/5/2001) mono samples faired very well.

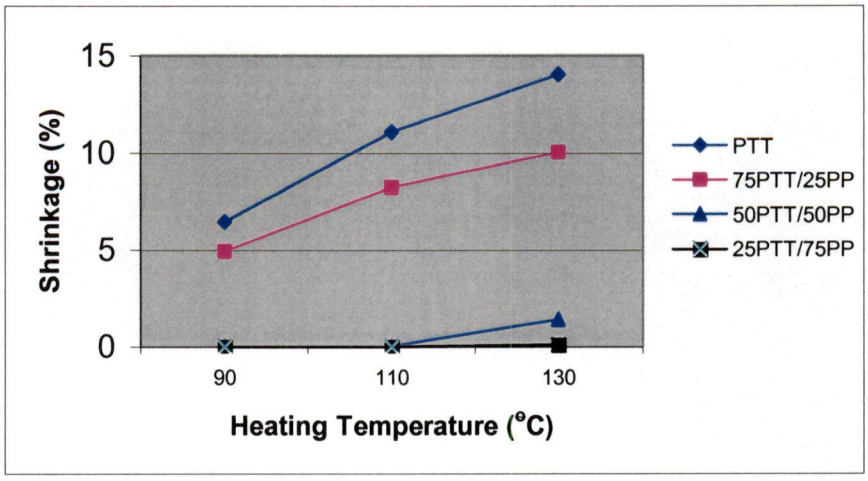


Figure 6. Heat Shrinkage of PTT Mono and PTT/PP Bico Meltblown Webs at 90°C, 110°C, 130°C for 7 Minutes

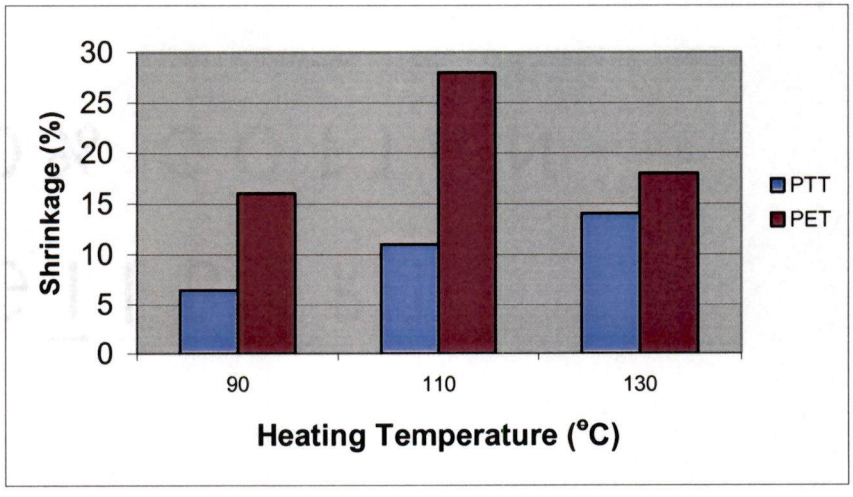


Figure 7. Comparison of Heat Shrinkage for PTT and PET Mono-Meltblown Webs at 90°C, 110°C, 130°C for 7 Minutes .

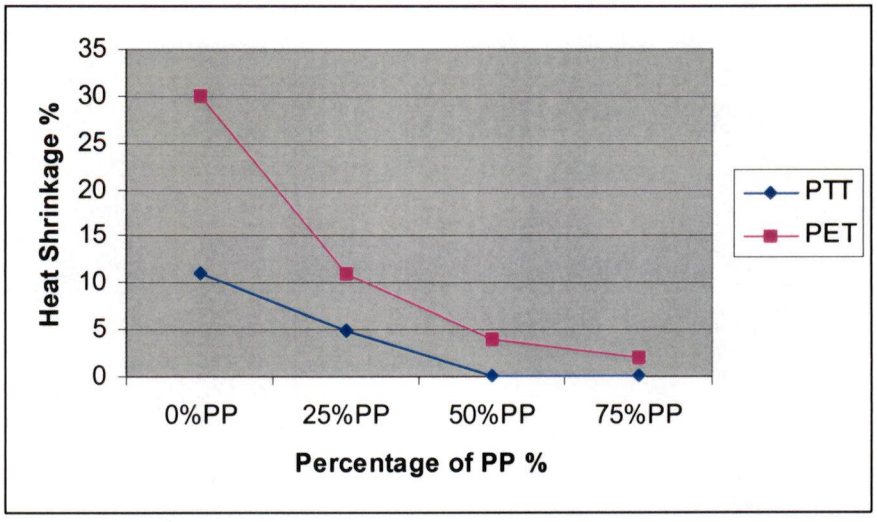


Figure 8. Comparison of Heat Shrinkage for PTT and PET Bico Meltblown Webs at 90 °C for 7 Minutes.

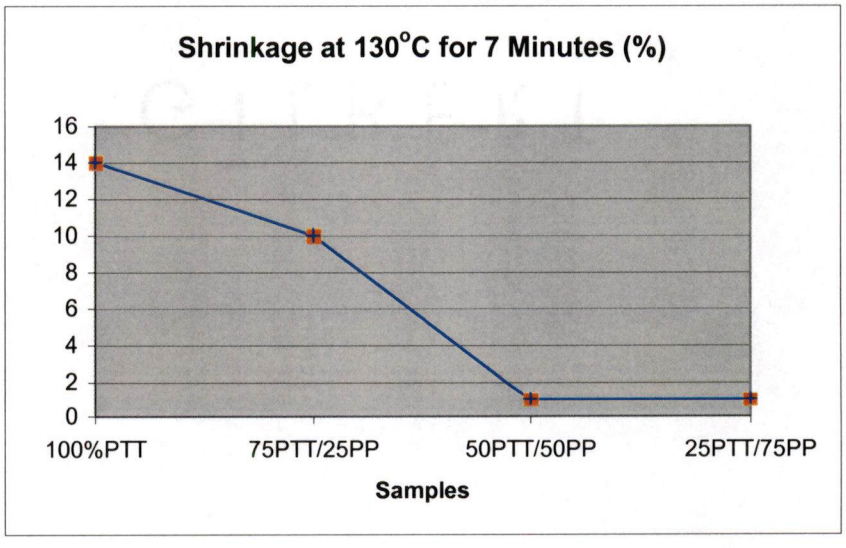


Figure 9. Heat Shrinkage of PTT mono and bico Meltblown webs.

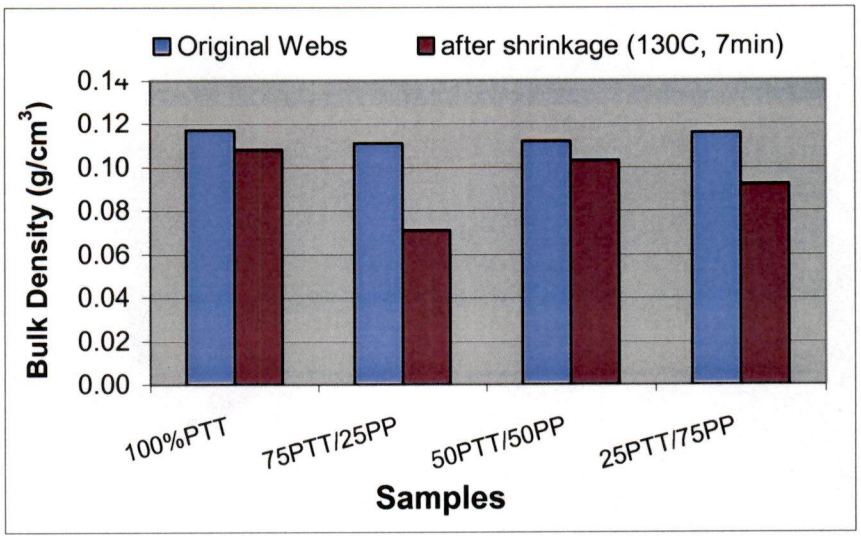


Figure 10. Effects of heat shrinkage on bulk density.

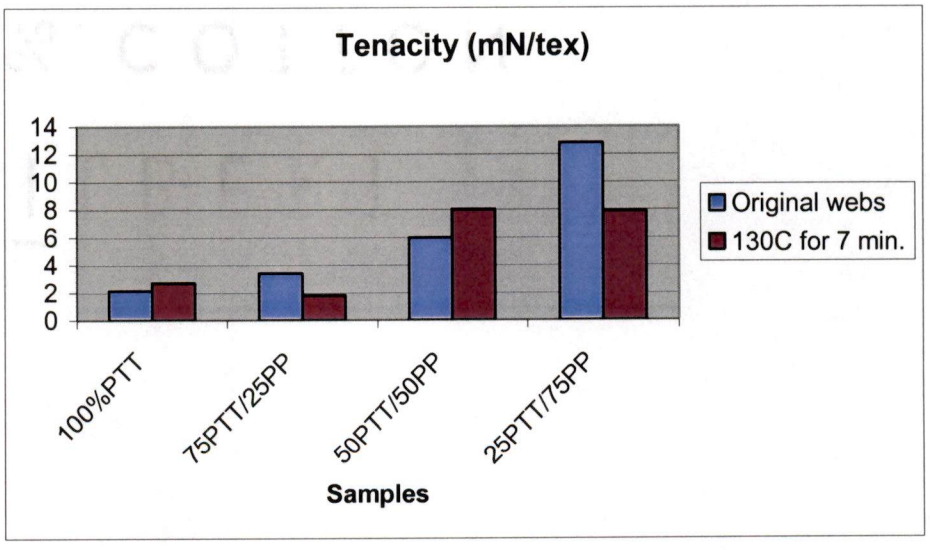


Figure 11.Effects of heat shrinkage on tenacity.

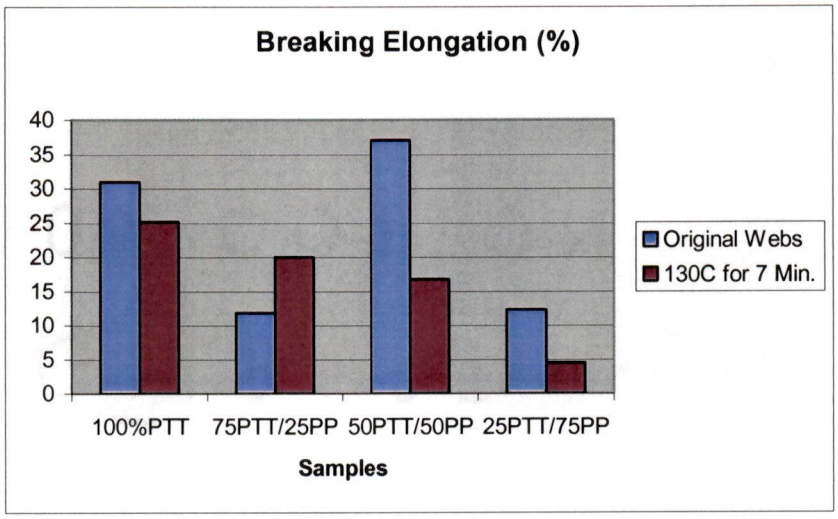


Figure 12. Effect of heat shrinkage on breaking elongation

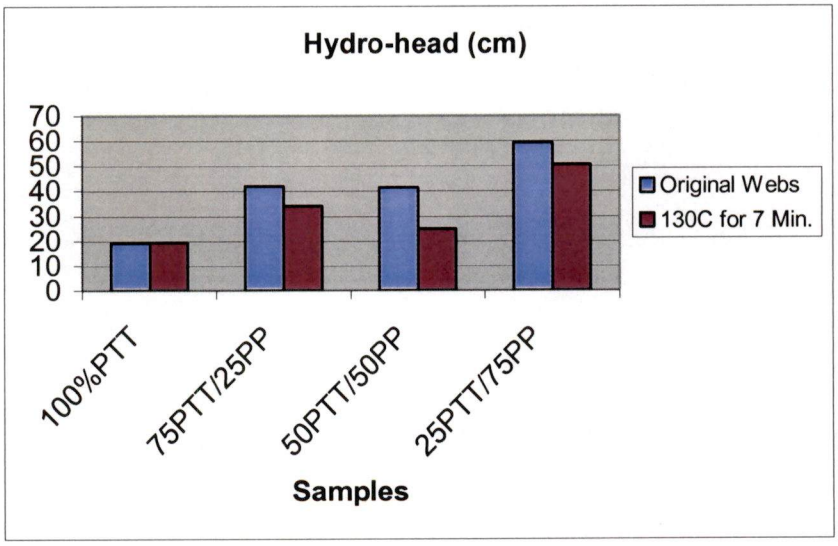


Figure 13. Effects of heat shrinkage on hydro-head

Testing upon an area of 64 in^2 the PTT samples tested at 190°C showed a respectable range of 58.4 in^2 to 64 in^2. The larger fiber diameter sizes have more shrinkage than the smaller fibers. Thus the area with no change in size, of the 100% PTT samples, had the smallest fibers. Heat shrinkage is affected by the polymer and web structure. See **Table 13** for details.

Table 13. Heat Shrinkage-Area change (%)

Sample ID	Sample Description	90°C			110°C			130°C			150°C			190°C		
		3min	5min	7min	3min	5min	7min	3min	5min	7min	3min	5min	7min	3min	5min	7min
3-1	75/25 PTT/PP	0.00	1.56	1.56	2.34	2.34	3.44	4.69	4.69	6.09	6.56	7.34	6.88	n/a	n/a	n/a
3-2	75/25 PTT/PP	2.66	2.66	2.66	5.00	5.47	5.47	6.88	7.66	7.66	5.78	7.66	9.84	n/a	n/a	n/a
3-3	75/25 PTT/PP	5.47	4.69	4.69	6.09	7.66	7.66	8.75	10.3	11.4	12.8	10.9	13.9	n/a	n/a	n/a
3-4	75/25 PTT/PP	6.88	6.09	6.09	8.44	9.84	10.3	12.8	12.8	11.7	14.2	14.2	13.6	n/a	n/a	n/a
3-5	50/50 PTT/PP	2.66	2.66	2.34	4.22	3.13	3.13	6.56	6.09	6.09	10.3	11.7	9.84	n/a	n/a	n/a
3-6	50/50 PTT/PP	4.69	3.44	3.91	5.47	5.47	6.09	8.44	9.84	9.84	12.8	13.3	14.2	n/a	n/a	n/a
3-7	50/50 PTT/PP	4.22	4.22	3.44	6.88	7.66	7.34	9.84	10.6	10.6	15	14.7	14.2	n/a	n/a	n/a
3-9	25/75 PTT/PP	2.34	1.88	1.88	3.44	3.44	3.91	5.00	5.78	6.09	9.84	12.5	10.9	n/a	n/a	n/a
3-10	25/75 PTT/PP	2.34	2.34	1.88	5.00	4.22	5.47	7.97	6.88	6.88	11.7	11.7	11.4	n/a	n/a	n/a
4-1	100% PTT	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1.56	1.56	1.09	0.00	0.00	0.00
4-2	100% PTT	0.00	0.00	0.78	3.13	3.13	3.13	0.00	0.00	0.00	4.22	4.22	4.22	4.69	3.91	3.91
4-3	100% PTT	0.00	1.56	1.56	4.22	4.69	1.56	1.56	1.88	2.34	5.00	5.47	5.00	10.31	8.75	8.75

Chapter XIV. Characterization by Fiber Diameter and SEM

Fiber diameter is an integral part of characterization. Many times the fiber diameter is requested before other tests. The fiber diameter establishes the structure of the web and can explain why the web acts the way it does. It is usual for a meltblown fiber diameter to be 2μm to 20μm.

In meltblown webs, these ultra fine fibers contribute to excellent fluid barriers, and filtration. Fiber diameter is determined by using a microscope with 50x magnification lens. For each sample slide there will be at least 35 measurements taken and processed through the software available at TANDEC to come up with an average value.

Meltblown fibers have a round to non-round character. By the very nature of meltblowing a polymer, one can visualize the polymer being extruded at a high rate of speed and as the fiber journeys through the air it will twist and bent and change shapes until it is collected on the collector belt.

Much debate looms over fiber diameter methods. It is argued that a microscopic view of a fiber's diameter is not a true diameter reading. True, but the fibers in this research show a non-round shape via SEM pictures. It is argued that in order to get a true representation of a fiber diameter one should get a cross-section or use methods of size determinations utilized in Filtration Efficiency tests. To do this size test one must put the sample into a vacuum chamber. The top of the sample should have a pressure reading "x" while the bottom pressure should read "y". The different pressure readings would be incorporated into a formula, thus giving a fiber diameter size.

That is quite a bit of work just to find a fiber diameter. The microscope using a 50x lens comes very close to this form of testing. The microscope is quicker and gives hard evidence and not a pressure change. However, it was decided to show cross-sectional views of PTT and PTT bico fibers to show the round, non-round, and bico make-up of the fibers. Figures 14-16 show in detail fiber alignment and bi-component configurations.

SEM Picture #1

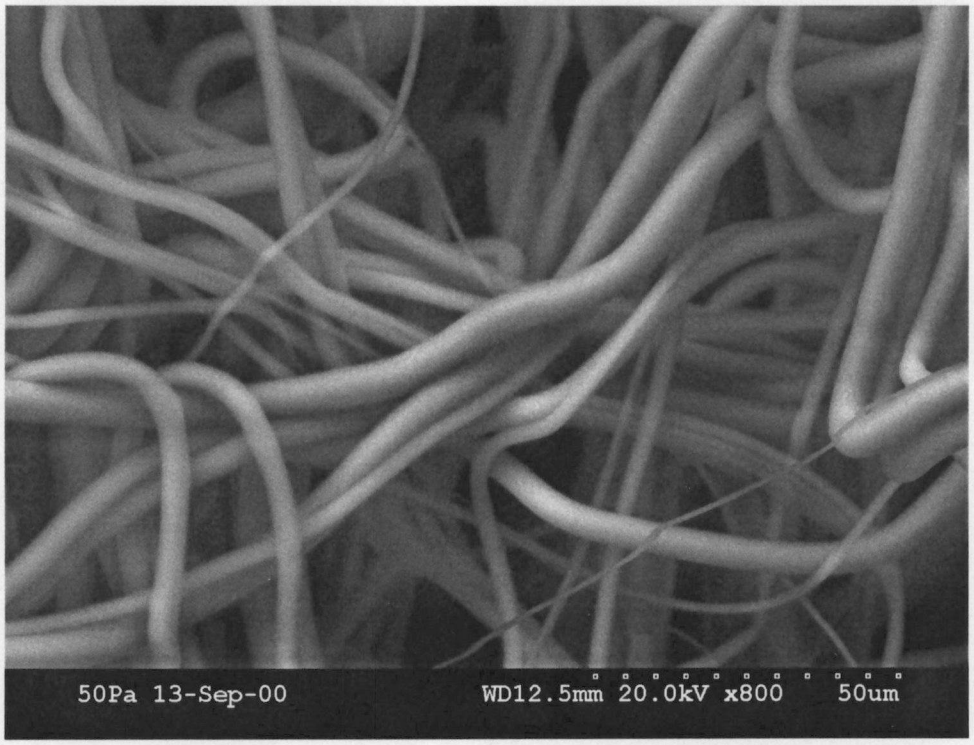


Figure 14. SEM of 100% PTT Meltblown Web

SEM Picture #2.

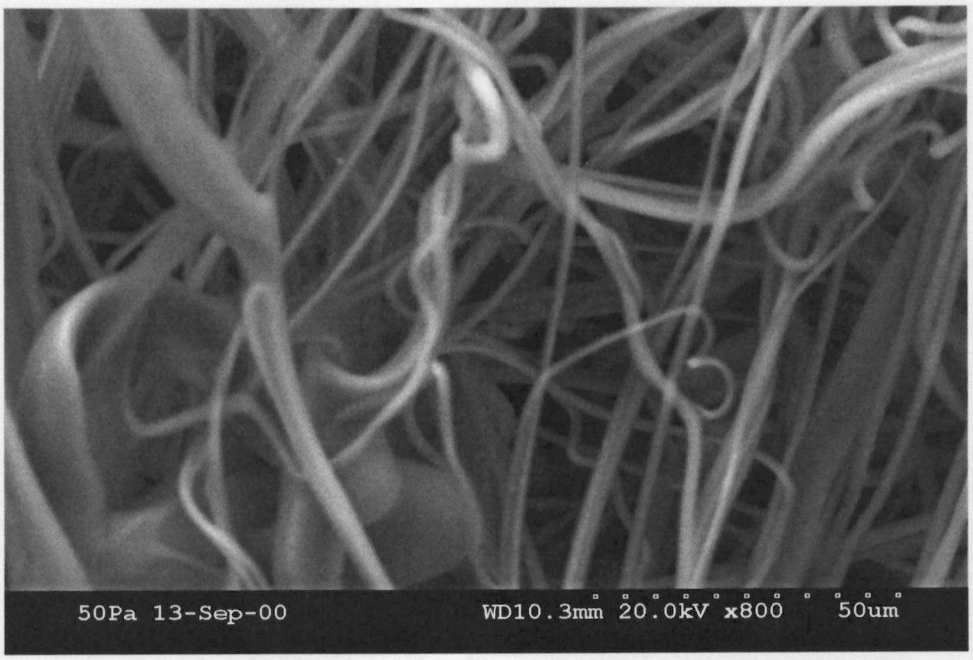


Figure 15. SEM of 50/50 PTT/PP Meltblown Web

SEM Picture #3.

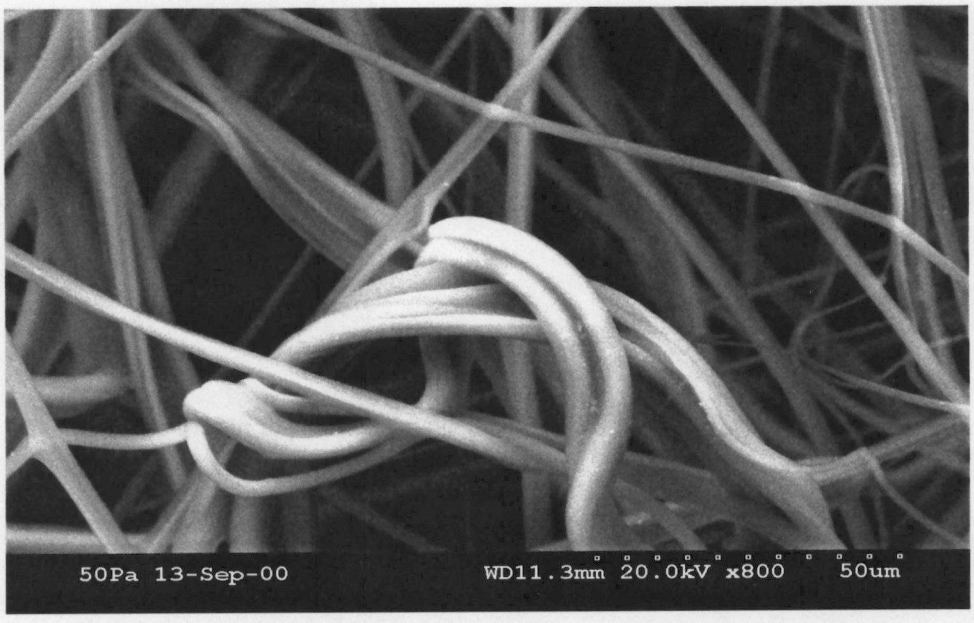


Figure 16. SEM of 25/75 PTT/PP Meltblown Web

The pictures above definitely show side-by-side fibers and non-round fibers. Here is a SEM view of a cross-section, Figure 17, of PTT and PTT bico sample. Notice that PTT in this picture is darker than the PP.

SEM Picture #4

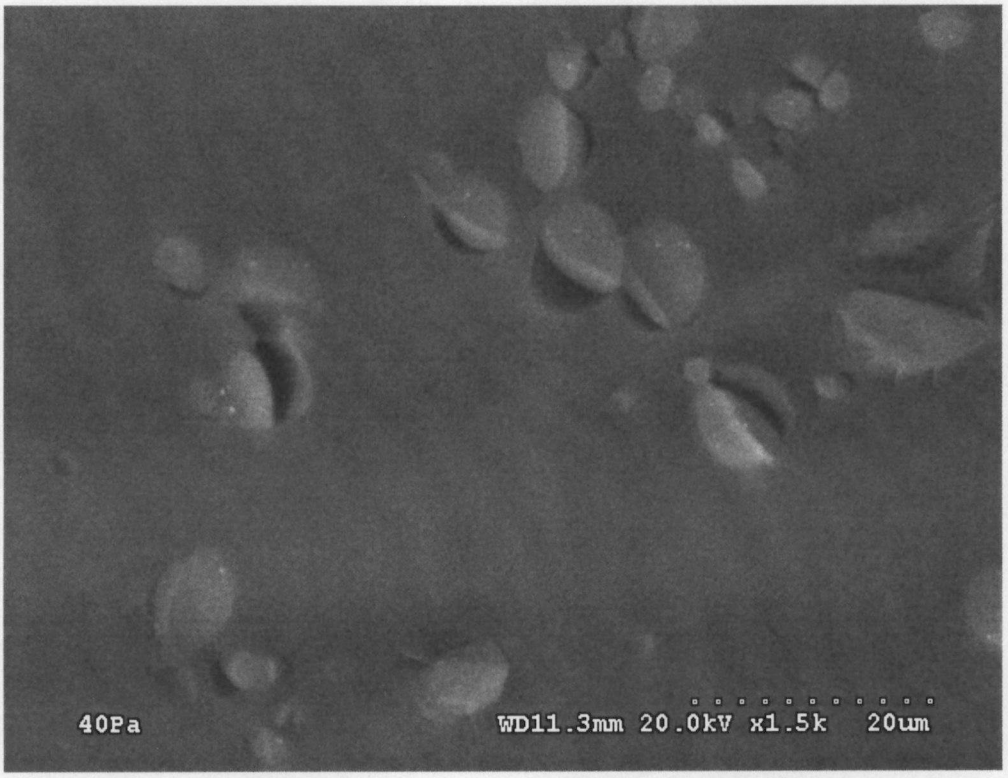


Figure 17. Cross-Section of 75/25 PTT/PP Meltblown Web Fibers

Chapter XV. Making Sense of the Results

PTT shrunk approximately 11% whereas PET shrunk by 28% at 110°C. PET takes a very long time to reach its glass transition temperature. This is why PET is so forgiving when processing because it stays molten longer.

Both PET and PTT behave similarly during heat shrinkage when PP is increased in the bico. PP stabilizes the movement of the polyester fibers. PP is very forgiving in processing. With a T_m of 168°C and a T_g of -17°C, PP crystallizes fast and the energy required to melt PP is not that great. Since PP crystallizes so fast it becomes the backbone of the web when the polymer bico hits the collector belt and cools down. At the lower temperatures (130°-90°C) the increase of PP significantly reduces shrinkage.

However, testing 100% samples gave strong evidence that 150°C is the maximum heat a PP web can endure without melting. Even though the sample gets charred at 150°C the integrity of the web remains intact.

In continuation of PTT comparisons after heat shrinkage, one can observe that the bulk density is reduced after being heated. The fibers are reduced in both length and diameter because of the heat. The web fibers constrict into a new size sample. Tenacity is affected differently because of the PP concentration. There is no broad evidence to confirm that the newly shrunken fibers increase tenacity. The fibers that once were full and firm now have become shriveled and tough. Due to the heat shrinkage, breaking elongation is reduced. By viewing **Figure 12**, page 43 one can see that the breaking elongation dropped dramatically for the 50/50 PTT/PP sample. The 75/25 PTT/PP sample was reversed. A possible explanation to this is the web configuration of the sample. No other valid answer could be found.

After heat shrinkage of the PTT, PTT/PP samples, hydro-head is reduced. That is to say that it takes less pressure to penetrate the web. The 100% PTT sample was virtually unchanged. Since the web does not have the rapidly cooled PP fibers, it behaves as if in unison. That is to say 100% PTT sample shrinks at the same rate instead of two different rates.

In comparison of PTT with PET, PBT, and PA-6 bico webs, without considering heat shrinkage, we can determine that with the increase of PP in the bico web the hydro-head increases, the drape decreases, and air permeability decreases. The mono-webs were characterized showing that the basis weights were very similar so that we can compare apples to apples. See **Figure 22** for results. We found that with similar basis weights, at 100% mono, PBT had the greatest hydro-head value. For the bico samples pertaining to hydro-head, it was viewed that increasing the PP would increase the hydro-head. PA-6 and PET behave similarly at 100%. In **Figures 18-22**, the different properties of bico PTT, PBT, PA, and PET are discussed.

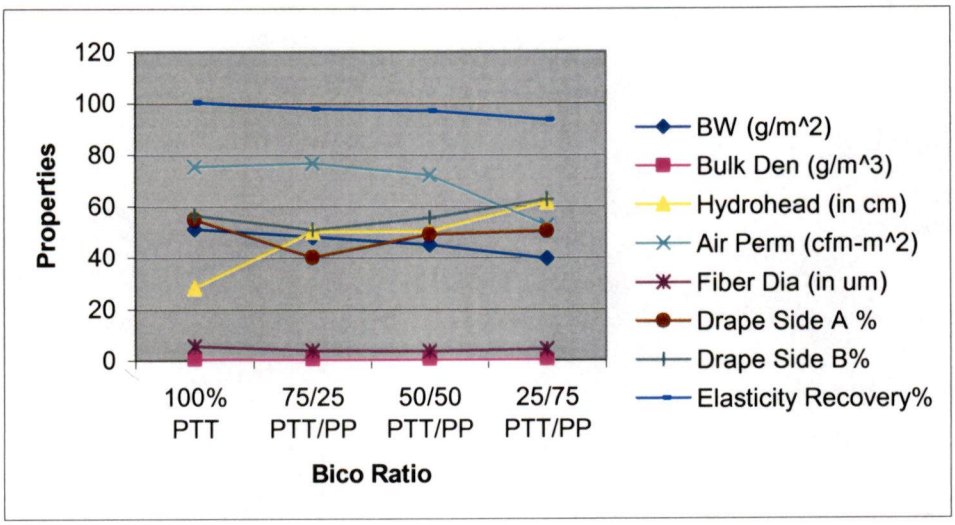


Figure 18. Physical Properties of PTT Bico MB Webs with Different Bico Ratios.

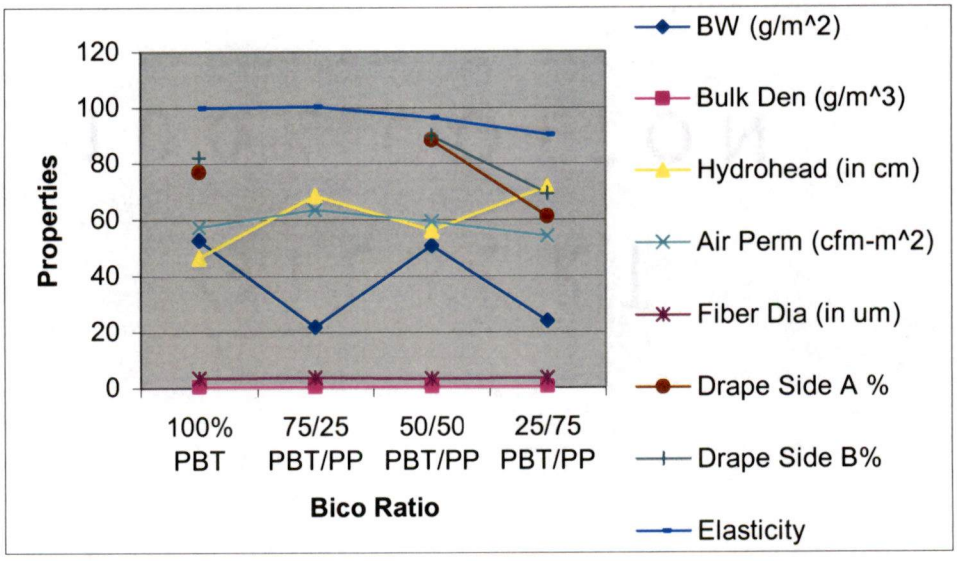


Figure19. Physical Properties of PBT Bico MB Webs with Different Bico Ratios.

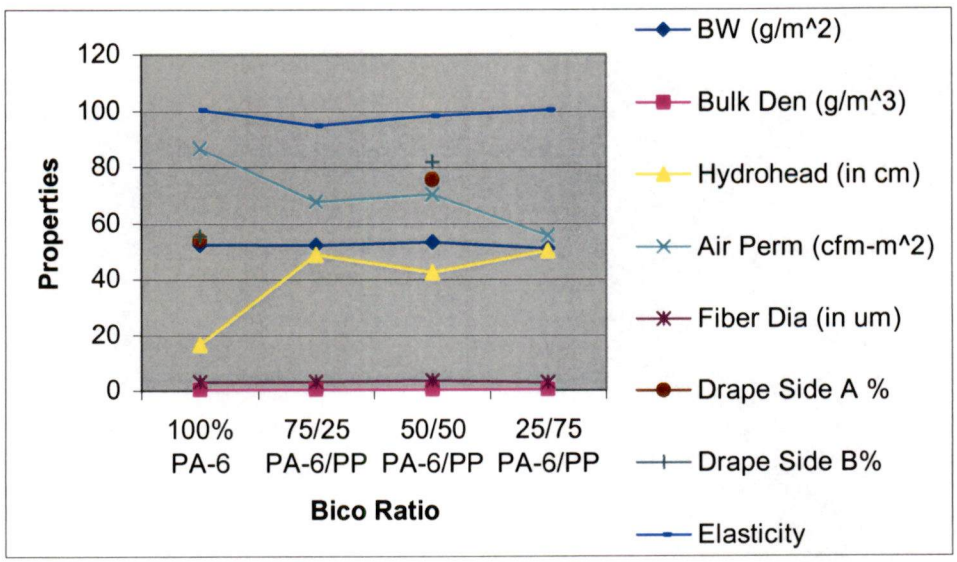


Figure 20. Physical Properties of PA Bico MB Webs with Different Bico Ratios.

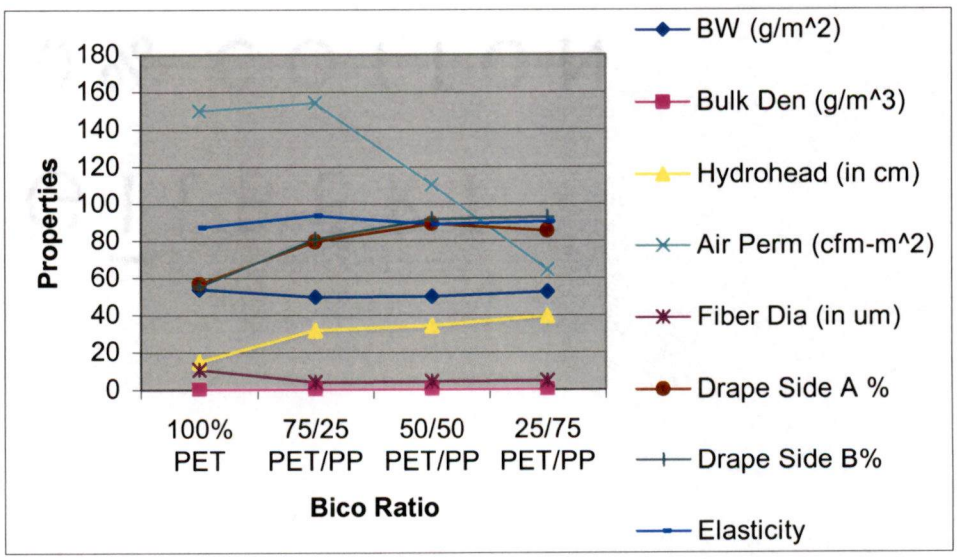


Figure 21. Physical Properties of PET Bico MB Webs with Different Bico Ratios.

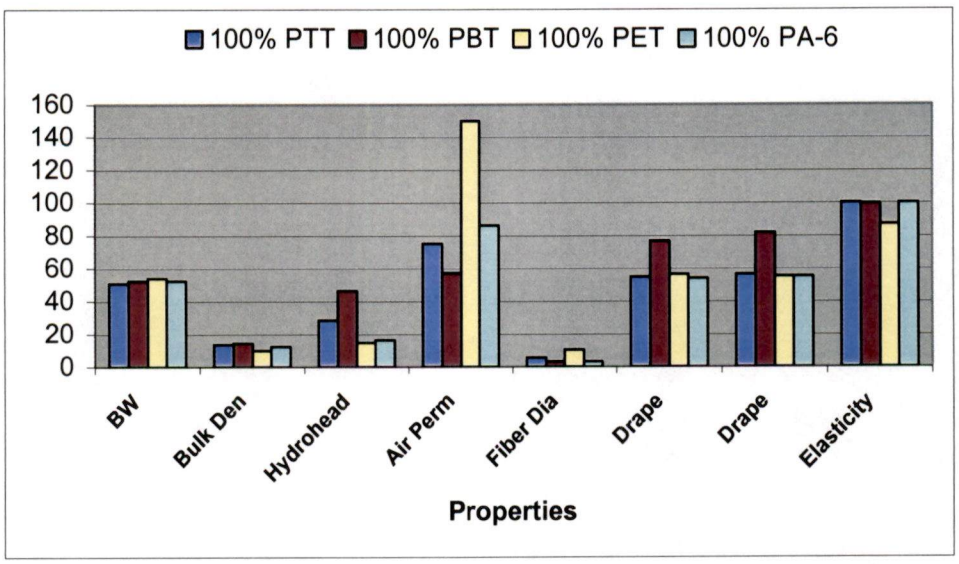


Figure 22. Physical Properties of Different Polymer MB Webs.

The increased ratio of polypropylene causes the webs to behave differently than the 100% webs. By looking at **Figure 23**, we can see how elasticity of the mono-based webs is affected. PTT, PA-6, and PBT have approximately the same initial elastic recovery but PET does not exhibit the same elastic recovery at 100% mono configuration. As the PP is increased from zero to 75% one can see the change taking place. PA-6 manages to regain its elastic recovery beyond 50% PP mix. PTT, PBT, and PET lose elastic recovery due to the PP being the binding agent in the web. The PP makes the meltblown web stiffer. PTT loses elastic recovery from 100% to 25% PP/PTT by approximately 7% due to the PP inhibiting the PTT fibers to move and expand.

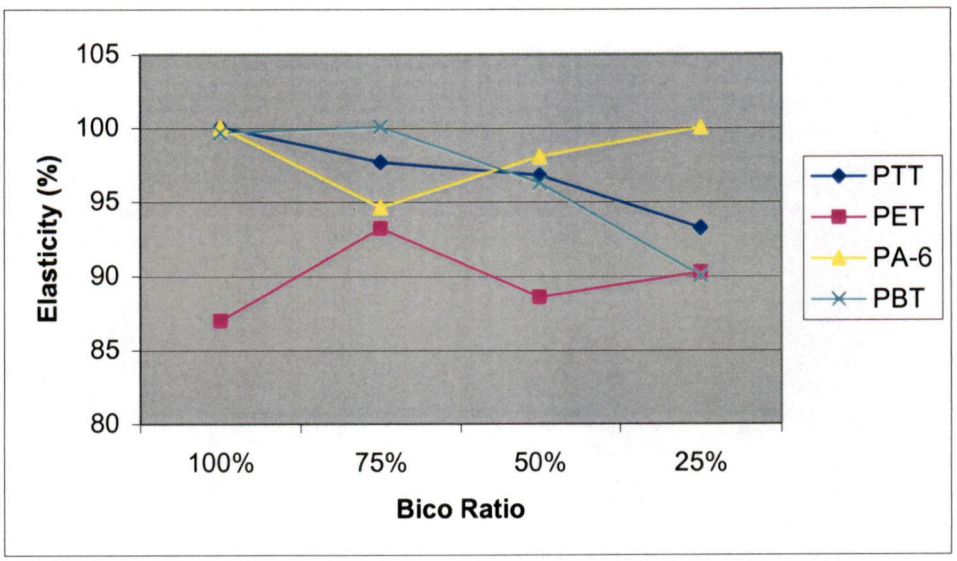


Figure 23. Elasticity of PTT, PET, PA-6, and PBT MB Webs.

Bulk density of the PTT, PET, PA-6, and PBT is slightly affected by the increase of PP in the meltblown web. **Figure 24** shows an approximate fluxuation of 4% between the samples. PTT and PBT drop bulk density when 25% PP is added while PET and PA-6 increase in bulk density. By looking at the figure one can see that each meltblown web has approximately the same bulk density at 100% and at 75% PP. These samples depict a cycle of bulk from 0 to 100%.

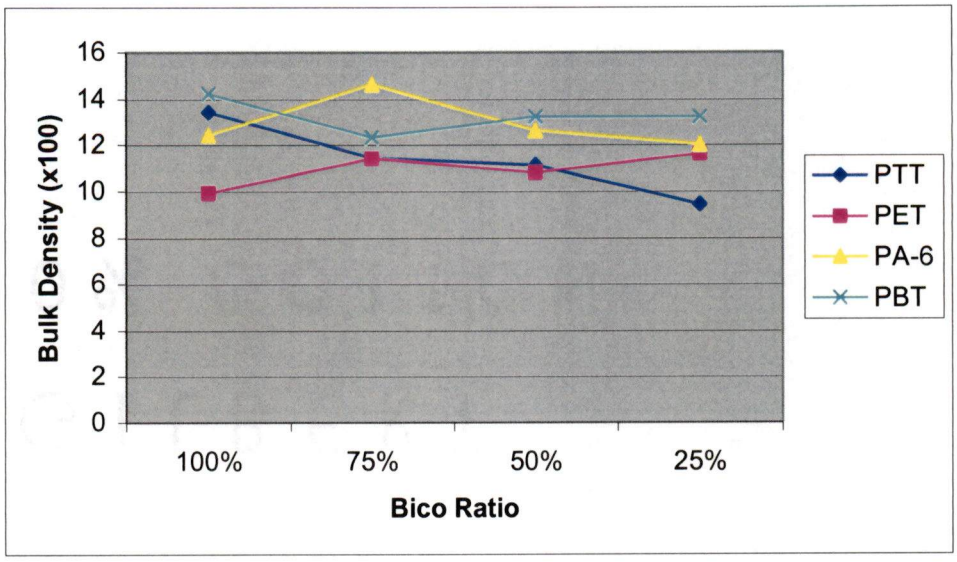


Figure 24. Bulk Density of PTT, PET, PA-6, and PBT MB Webs.

Hydro-head in **Figure 25** shows that when the PP is increased so does the hydro-head (in centimeters). The PP ratio in the meltblown web makes for a better moisture barrier. Each sample increases hydro-head dramatically at 25% PP then drops slightly at 50/50 then increases again. The reason for such action can be contributed to the distribution of fibers in the web. Only at 50/50 does the meltblown web deviate from the anticipated behavior. The even mixture of the polymers and PP at 50/50 creates more opening between the fibers as does 75% PP fibers. PTT showed a consistent increase in hydro-head with increased PP. PBT is the only rival in the test for best barrier properties. Regardless of polymer, each had better barrier properties with the presence of PP.

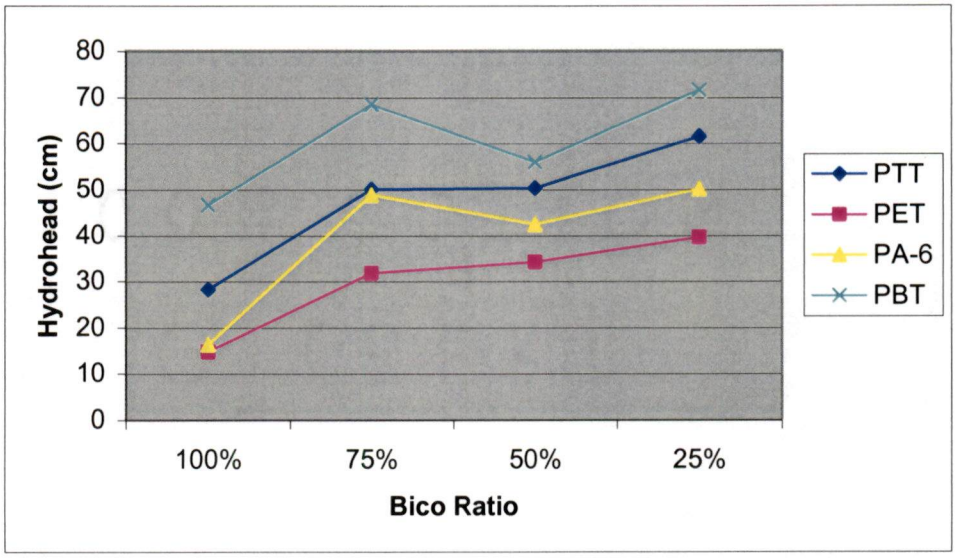


Figure 25. Hydro-head of PTT, PET, PA-6, and PBT MB Webs.

Air Permeability of PTT, PET, PA-6, and PBT are significantly affected by the presence of PP. In **Figure 26**, PET has a reading of 150 cfm/m^2 while at 100% mono-based meltblown web. The PTT, PBT, PA-6 mono-webs were very similar to each other ranging from 55 cfm/m^2 to 80 cfm/m^2. With the introduction of PP one can see that PET was significantly affected. The drop was from 150 to 60 cfm/m^2. PTT, PBT, PA-6 webs weren't affected as much as PET. These three meltblown webs stayed consistent throughout the ratio range. PTT stayed within 25 cfm/m^2 from 0 to 75% PP presence. PTT and PP fibers are very similar in size contributing to the slight change in air permeability. When looking at **Figure 27**, one will notice that PET has the largest fiber diameter while at 100% mono configuration.

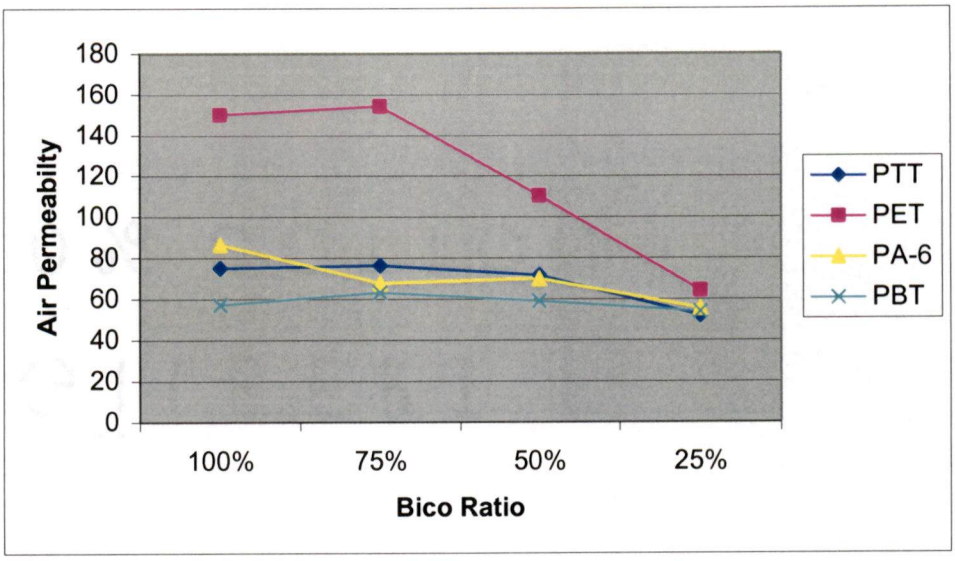


Figure 26. Air Permeability of PTT, PET, PA-6, and PBT MB Webs.

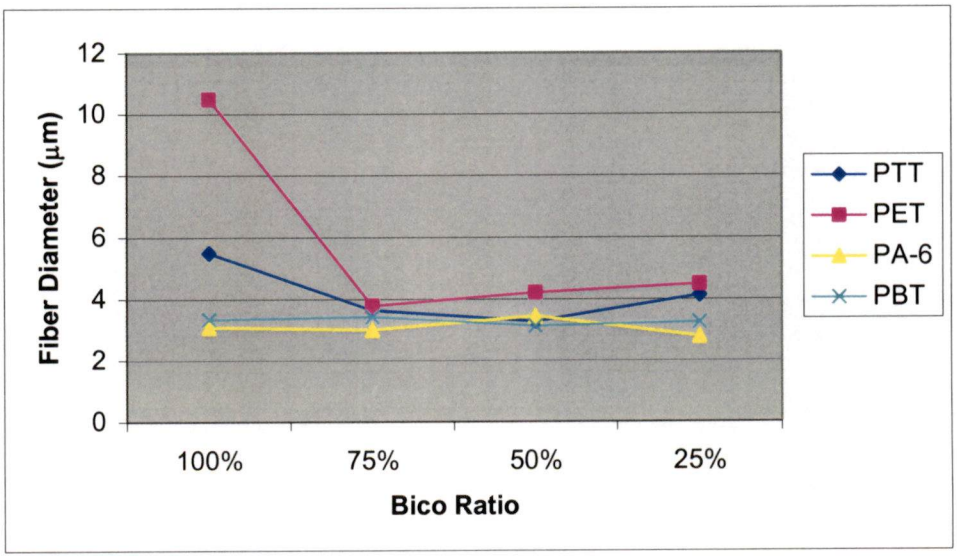


Figure 27. Fiber Diameter of PTT, PET, PA-6, and PBT MB Webs.

Drape ability of PTT, PBT, PET, and PA-6 vary due to the fiber size and PP ratio of the meltblown webs. **Figure 28** shows the percentage of drape per sample. The higher the percentage on the graph, the less the meltblown web drapes. We can see that PET did not drape as much as PTT. We can also see that Side A does not drape the same as Side B. This is why we have **Figures 28** and **29**. Side A is an example of the collector belt side exposed or in the up position. Side B is the collector side down. The reason Side A doesn't drape as well as Side B is because Side A has more compacted fibers and web structure. A meltblown web is a three dimensional item. When the fibers leave the spinneret and make contact with the collector belt, they press into each other more than the last fibers that make it to the belt. Let it be stressed that PTT has better drape than PET.

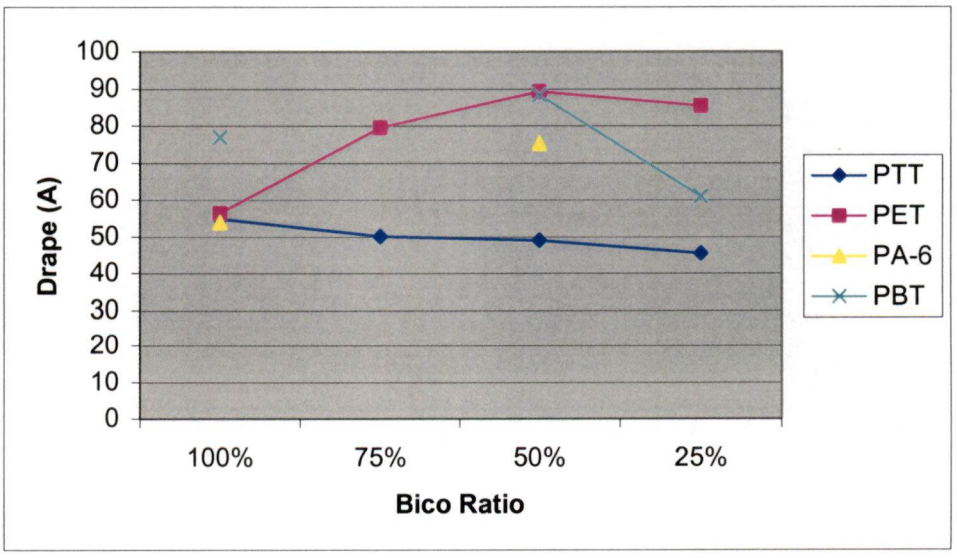


Figure 28. Drape ability of PTT, PET, PA-6, and PBT MB Webs: Side A

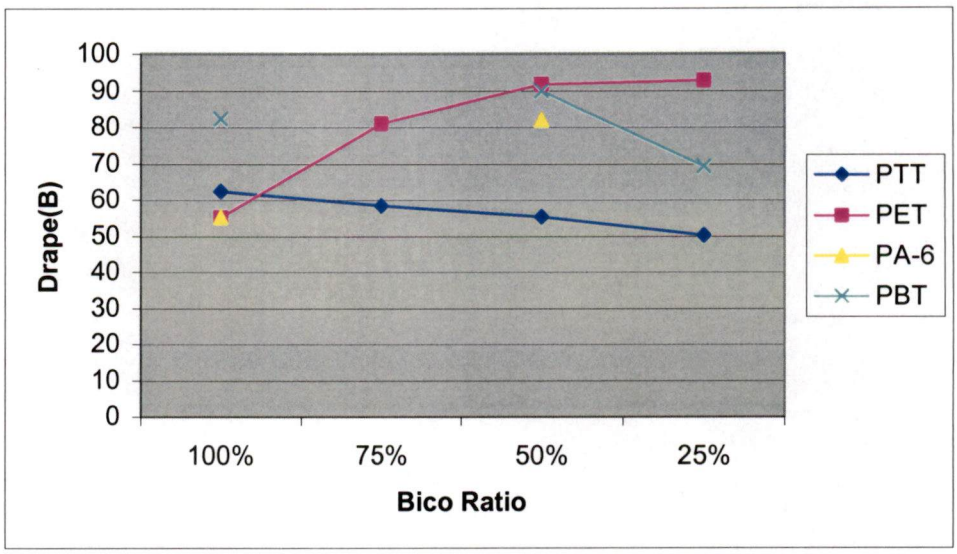


Figure 29. Drape ability of PTT, PET, PA-6, and PBT MB Webs: Side B

Chapter XVI. Conclusions

Poly(Trimethylene Terephthalate) (PTT) resins have been extruded to form quality mono- and bi-component PTT meltblown fiber webs using the Reicofil® side-by-side bicomponent meltblown line. Processing conditions were changed for the web development with the melt throughput from 0.3-1.5 g/hole/min, melt temperature from 270 to 305 °C (520 to 580 °F), air temperature from 230 to 310 °C (450 to 530 °F), air flow rate from 12.5 to 29.2 SCFM/in.Die and DCD from 11 to 19 inches. The obtained fiber diameter was in the range of 1.71 to 4.76 μm. PTT and bico PTT/PP exhibited excellent meltblown processability and web quality. Compared to conventional (mono-component) round and smooth meltblown fibers, the bicomponent PTT/PP webs showed the structure of non-round cross-sectional and twisted fibers. The barrier property and heat shrinkage resistance of the bico webs were notably improved. Results showed the PTT MB webs have a better drapability than any PET, PBT and nylon MB webs. PTT showed good processability for meltblowing process.

PTT is a formidable competitor with PBT, PET, and PA-6. The physical characteristics show us that PTT is in the middle of the hunt. PTT has a lower Tm than PET. PTT has a lower T_g than PET. PTT is stable at high heat. PTT has excellent air permeability, hydro-head, and drape. PTT doesn't crystallize as fast as PBT but it doesn't sacrifice modulus in order to crystallize. PTT is a great choice for garments, but the customer must want quality instead of cheaper raw materials. PTT is super competitive even costing nearly the same as PA-6. If the purchase cost could be reduced

to PET, then that would be a strong selling point. PTT has even been visualized as an Engineering Thermoplastic. There are so many things PTT does well.

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VITA

John Beard was born in Centerville, Tennessee, on October 23, 1970 to Mr. And Mrs. Gary Beard. He is the youngest of two with an older sister. He attended Nashville State Technical Institute earning an Associates Degree in Architectural Engineering in 1990. In August of 1990, he began a drafting/detailing position at Stud Welding, Inc. in Centerville. After two years he decided to go back to school to earn a higher degree. In August 1992, he enrolled at Austin Peay State University in Engineering Technology concentrating on manufacturing processes and principles. While at APSU, he was recognized as "Who's Who in American Universities and Colleges". He also was a student member of the Society of Manufacturing Engineers and president of the campus Church of Christ Student Center. He earned the Bachelor of Science degree in 1995. On May 1, 1996 he was employed by Universal Fasteners, Incorporated, in Centerville, Tennessee as a pressroom engineer. The main function of the pressroom engineer was to keep the presses running efficiently. He contributed to the welfare of the pressroom operator by utilizing new equipment and building items that would be ergonomically better and a benefit to production. On January 1, 2000, he resigned from Universal Fasteners, Inc. and began working on a Masters of Science degree in Textile Science at the University of Tennessee at Knoxville. At UT Knoxville, he was awarded the College of Human Ecology, Textile Science Service Award for outstanding service to CISM and TANDEC during the transition from one building to the new location on 1321 White Avenue, and for selfless readiness to help when needed. He earned his M.S. in August 2001.