

Title: Nanofiber Webs via Spunbonding

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ABSTRACT:

In this report, we compare the usability of three polymers Excevall, AQ55 and AQ65 as sea in islands-in-the-sea configurations. We discuss the possibilities of removal of these polymers from the fabric and the problems encountered. The method used for removing Exceval from 36 I/S 50/50 PP/Exceval fabric is suggested. Based on experimental data for melting viscosity of AQ55, AQ65 and PP, the possibility of spinning AQ polymers with PP is discussed. The problems associated with the spinning of AQ55 with PP are discussed and possible solutions are offered.

The tensile properties of I/S PP/AQ55 drawn and freefall fibers before and after sea removal are investigated. Potential problems dealing with solidification and attenuation of PP during processing with AQ55 is confirmed experimentally. The diameter of the fibers before and after removing the sea polymer is also reported.

INTRODUCTION:

Background:

The common definition of nano is “the precise manipulation of individual atoms and molecules to create layer structures” [1]. In the fiber industry, nanofibers are materials made up of conventional and newly emerging polymers and with end uses as a typical textile product. The size of nanofibers is varied in range of 50 to 500 nanometers (comparing to human hair 20000 to 30000 nanometers). Nanofibers can not be seen without visual amplification. Research into production of polymeric nanofibers began almost twenty years ago. The main focus was on developing an improved barrier fabric. The short term solution was meltblown fabrics that can enhance liquid retention and decrease water contact angle [2]. With the addition of nanofibers, it is expected that air resistance and breathability are improved. Nanofiber webs are expected to provide good barrier properties, small pore size and large surface area. The manufacturing

techniques associated with polymeric nanofibers are electrospinning (40 to 200 nm or larger if so desired), melt blowing (500 nm to 10 microns) and through the use of splittable bicomponent fibers or those that depend on dissolution of one component, e.g., islands-in-the-sea (200 nm to 5 microns or larger if so desired). Compared with electrospun and meltblown nanofibers, fibers produced by splitting and dissolution techniques can be more economical and lead to stronger fibers, with broader polymer selection and without practical restraint on size and shape.

Bicomponent (conjugate) fibers consist of two polymers with different chemical or physical properties extruded from the same spinneret simultaneously. The wide study of bicomponent fibers was caused by discovering multiphase region in wool. Photomicrographs of cross-sections of wool fibers showed that they are composed of two components adhere strongly to each other and rotate spirally around each other as they run the length of fiber [3]. The difference in the shrinkage of these two components leads to the helically crimped configuration of the wool fiber. Wool is, in fact, a natural bicomponent fiber. This discovery confirmed the rationality of idea of creating chemical bicomponent fibers as a new way of producing self-crimping synthetic fibers. The first patent on bicomponent fiber was obtained by “IG Farbenindustrie” in 1943 (patent # 325339). The first commercial bicomponent application was introduced in the mid- 1960s by DuPont [4].

In general, bicomponent fibers can be divided into several groups according to the component distribution within the cross sectional area: side-by-side, sheath-core and matrix-fibril. Side-by-side bicomponent fibers consist of two components laying layer wise and divided along the length of fiber into two more or less distinct regions. Sheath-core bicomponent fibers made up of two components in such way that one of components (core) is fully surrounded by a second component (sheath). Matrix-fibril bicomponent fibers contain many fine fibrils of one polymer that are dispersed in the matrix of another. Matrix-fibril bicomponent fibers are spun from mixture of two polymers in some proportion, where one polymer is suspended in form of droplets within the second melt. The presence of fine fibrils in the matrix is usually increasing the fiber modulus, reducing the moisture regain, improving the texturing capability and reducing dyeability.

Splittable fibers are materials in which components are chosen in such way that they have very small degree of adhesion to each other and can be separated chemically or otherwise. This technique can be used to make ultra microdenier products in the size range from 0.1 to 0.02 denier per filament (dpf). The technology of producing ultra microfibers or even nanofibers involves spinning and processing of bicomponent fibers in range of 2 to 5 dpf (12 to 20 microns), after which the fibers are split into smaller fibers with denier of 0.1 or even less [5]. Several approaches can be used here such as islands-in-the sea, splittable pies and tipped fibers.

Using splittable pies technique, after the bicomponent fibers are spun, down-stream processing (mechanical drawing combined with hydroentanglement techniques) is applied to fibers, causing their splitting. The other approach to make an ultra fine fiber is to use tipped fibers where the one polymer is placed in on the tip on a trilobial or delta cross section fiber. After spinning the fibers are twisted and wet heat is applied. The polymer on the tips of the fiber breaks apart into microfibers approximately 0.2 denier and spiral around core polymer.

fluids. This property can be useful for application of AQ polymers in the production of the diapers. They also have high bond strength required by application as adhesive to resist shock, stress, high humidity, extreme temperatures encountered in transportation and storage. *Eastek* 1200 provides excellent water resistance, alcohol resistance, fast drying rates, and a consistently low coefficient of friction. Water dispersibility is also one of the most important properties of AQ polymers. The pellets of polymers can be dispersed directly in hot, deionized water (pH 5-7) without assistance of cosolvents, surfactants and others. Dispersion requires constant agitation at the temperature 85 °C from 20 to 30 min and at the temperature 60 °C for 90 minutes. The water-dispersibility of AQ polymers is due to presence of polar hydrophilic sodiosulfo groups distributed randomly along the polymer backbone. Usual molecule of AQ55 has approximately 5-8 ionic sodiosulfo substituents. The other feature of AQ polymers is their ability to be recycled. Adding ionic species to aqueous medium will cause the polymer particles to overcome their repulsion, coagulate and form separate phase. It is possible then to precipitate out the polymer.

Typical applications of AQ polymers are:

- adhesives: for bonding wood, paper, plastics, nonwoven assemblies, textiles and others ;
- cosmetic film-former: hair spray, mousse, setting lotion, moisturizers, nail polish;
- personal care: hand creams, sun screens;
- household products: shoe polish, air fresheners;
- ink and others.

Exceval is a copolymer of polyvinyl alcohol (PVA) and ethylene-vinyl alcohol (EVA). It has melting point $T_m = 150-230$ °C (our case $T_m=207$ °C), glass transition temperature $T_g = 55$ °C and crystallization temperature $T_c= 187$ °C. This polymer is biodegradable (can be decomposed by microorganism), water soluble and has good gas barrier properties. Dissolved in water it has a stable viscosity at low temperatures, easy film forming properties, good affinity with cellulose and compatible with starches. The dissolving of polymer pellets require continuous agitation at the temperature 80-90 °C from 1 to 2 hours.

Typical applications of Exceval are:

- stabilizer for emulsion polymerization of vinyl acetate monomer to prevent gel formation
- surface sizing agent fro paper
- textile sizing agent
- nonwovens (micro fibers)
- oxygen barrier material for food packaging
- raw material for melt-blown non-woven fabrics with hydrophile property.

The major use for nano (micro) fibers produced through spun bonding process by using islands-in-the-sea bicomponent fiber technology is in ultrasuede and other synthetic leather products since a collagen bundle fiber of genuine leather is about 0.01-0.001 dpf. Other possibilities are in the filtration field, for medical or semiconductor applications, which require the ultimate in superfine filtration [4]. Other applications of nanofibers include barrier fabrics, wipes, personal care, and pharmaceutical applications. Many of air filters are made from webs of nanofiber filter media [6].

OBJECTIVES

Creating the finest fibers from bicomponent fibers by dissolving sea polymer becomes an important task. The issues remain the selection of sea polymers and the manner in which it affects spinnability, its removal and its cost. With respect to fiber formation, the issues are how well the fiber is formed. Fabric properties will ultimately define the type and the count of islands to be used.

The objectives of this study may be summarized as:

Overall objectives:

- ◆ Determine the feasibility of I/S technology for creating sub-micron spunbonded nonwovens.
- ◆ Investigate the interaction of two polymers during processing by developing the computational model of solidification and attenuation of the I/S bicomponent fibers.
- ◆ Determine the interaction between island count and polymer composition and their influence on:
 - Fiber size
 - Spinnability
- ◆ Selection of the “correct” sea polymer with respect to:
 - Spinnability
 - Ease of removal
 - Cost
 - Environmental impact
 - Fiber formation
 - Fabric properties

Experimental objectives:

- ◆ Determine the role of the following on fiber and fabric properties:
 - Island count
 - Polymer type
 - Polymer ratio
 - Processing conditions
- ◆ Determine following properties for both free-fall and drawn fibers:
 - Size
 - Modulus
 - Failure stress
 - Failure strain
 - Crystallinity
 - Orientation
- ◆ Determine following properties for fabric:
 - Modulus

- Failure stress
- Failure strain
- Barrier properties

Secondary objectives:

- ♦ Determine the best known method for sea polymer removal
- ♦ Investigate the influence of number of islands and polymer composition on:
 - Fiber size
 - Fiber properties
 - Fabric properties

EXPERIMENTAL APPROACH:

The melting viscosity of PP and AQ55, AQ65 was measured by using Thermo Haake Minilab Equipment. The values were obtained in range of temperatures from 180 to 280 °C. The shear rate 71.06 [1/sec] (120 rpm screw speed) was used.

Bicomponent fibers 36 I/S PP/Exceval in proportion 50/50 islands/sea and 36 I/S PP/AQ55, 36 I/S PP/AQ65, 108 I/S PP/AQ 65, 18 I/S PP/AQ55 in ratios of 50/50, 25/75 and 75/25 islands/ sea were obtained. 18, 36, 108 I/S PP/AQ55, AQ65 were made at NCRC's Partners Laboratory. The PP/Exceval samples were made at Hills.

The web contains AQ55 and AQ 65 polymers was bonded by using calender and hydroentanglement systems. To calender the web the temperature range from 121⁰C to 170⁰C and pressure from 800 PSI to 1300 PSI were used. The water jet pressures used in hydroentanglement system were in a range from 30 to 225 bars.

To dissolve the sea polymer (Exceval and AQ55, 65) a Jet Dyeing machine was used. While typically use for dyeing of conventional fabrics, this machine is ideal for the removal of the sea because it provides agitation in the form of water jet streams. The characteristics of the machine used are: liquor content 6-20 liters, fabric content 100–1500 g, fabric speed 4-30 m/min, temperature 20-150⁰C (4 bars), and heating up speed 2-4⁰C.

The fabrics contain Exceval polymer were subjected to Jet Dyeing machine at different times (5, 15, 30 min) and different temperatures (80, 110, 130⁰C). The initial temperature was in range of 25-30⁰C. Seven samples of 36 PP/Exceval were obtained and examined. 36, 108 I/S PP/AQ65 calendered and hydroentangled web was subjected to jet dyeing machine at temperatures 80, 90, 110, 120 °C and time 15,20,30,60,120 min. In total 10 samples were obtained. 36 I/S 50/50 PP/AQ55 calendered fabric was subjected to jet dyeing machine at temperature 80⁰C and 15 min. 18 I/S 50/50 PP/AQ 55 calendered and hydroentangled fabric was subjected to jet dyeing machine at temperature 80, 90 and 110⁰C and time 15, 20, 30 min.

The fiber diameter from washed spunbond samples 36 I/S PP/Exceval was defined by taking ESEM pictures and then measuring the diameter of fibers manually by using image analysis system. Optical microscopic pictures of washed and unwashed 18 I/S PP/AQ55 fibers (drawn and freefall) were taken and the fiber diameter was measured using the same technique as mentioned above. The linear density D_L (den) of fiber then was calculated:

$$D_L = 9000\pi R^2 \rho \quad (1)$$

R^2 - fiber diameter in meters,
 ρ - density of fiber in g/m^3 .

The tensile properties of washed and unwashed 18 I/S PP/AQ55 fibers (drawn and freefall) were determined by using standard test method D3822-01. Twelve specimens from each sample of 18 I/S 75/25 PP/AQ55, 50/50 PP/AQ55 and 25/75 PP/AQ55 were used. Each specimen was mounted centrally in clamps of a tensile testing machine (Instron) and a force applied until the specimen breaks. A constant time to break 20 ± 3 s, gage length 1 inch, break sensitivity 90% and loading rate 0.1 in/min were used in test method. Values for the breaking force and elongation of the test specimen were obtained. Average values for the breaking force, elongation-at-break, tenacity, initial modulus and secant modulus at 10% of elongation were calculated. Breaking tenacity was calculated by using formula:

$$Y = \frac{F}{D_L} \quad (2)$$

F - breaking force (gf),
 D_L - linear density of the fiber (denier),
 Y - breaking tenacity (gf/den).

To calculate initial modulus, the maximum slope was located and a line tangent to the stress-strain curve was drawn. The stress and the corresponding elongation with respect to the stress axis were measured. Initial modulus J_i (gf/den) was calculated by using formula:

$$J_i = \frac{S}{\varepsilon_p} \quad (3)$$

S - determined stress on the drawn tangent line (gf/den),
 ε_p - corresponding strain with respect to the drawn tangent line and determined stress.

To calculate the secant modulus the stress for a specified elongation, such as 10 %, was determined and the point on the stress-strain curve P_2 was labeled. Then a second point, P_1 at a specified elongation, such as 0 % elongation was labeled. A straight line through points P_1 and P_2 intersecting the zero-stress axis was drawn. Secant modulus J_s (gf/den) was calculated as:

$$J_s = \frac{S}{\varepsilon_p} \quad (4)$$

S - determined stress on the constructed line (gf/den),
 ε_p - corresponding strain with respect to the constructed line and determined stress.

RESULTS:

To determine the best method of sea polymer removal that gives the finest fibers the ESEM images of fibers in washed and unwashed samples 36 I/S PP/Exceval were taking (Fig.1, 2).

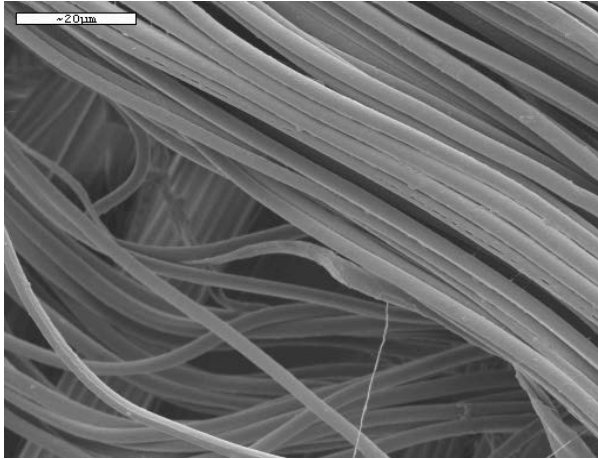


Fig.1. 36 I/S PP/Exceval, 110⁰C, 30 min

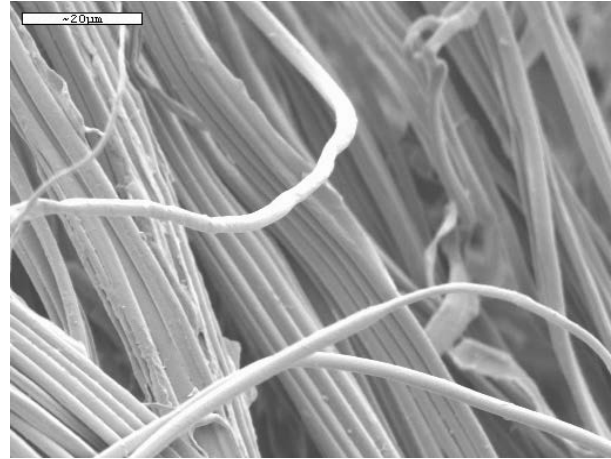


Fig.2. 36 I/S PP/Exceval, 130⁰C, 30 min

The results of fiber diameter measuring for samples 36 I/S PP/Exceval are depicted in Fig.3.

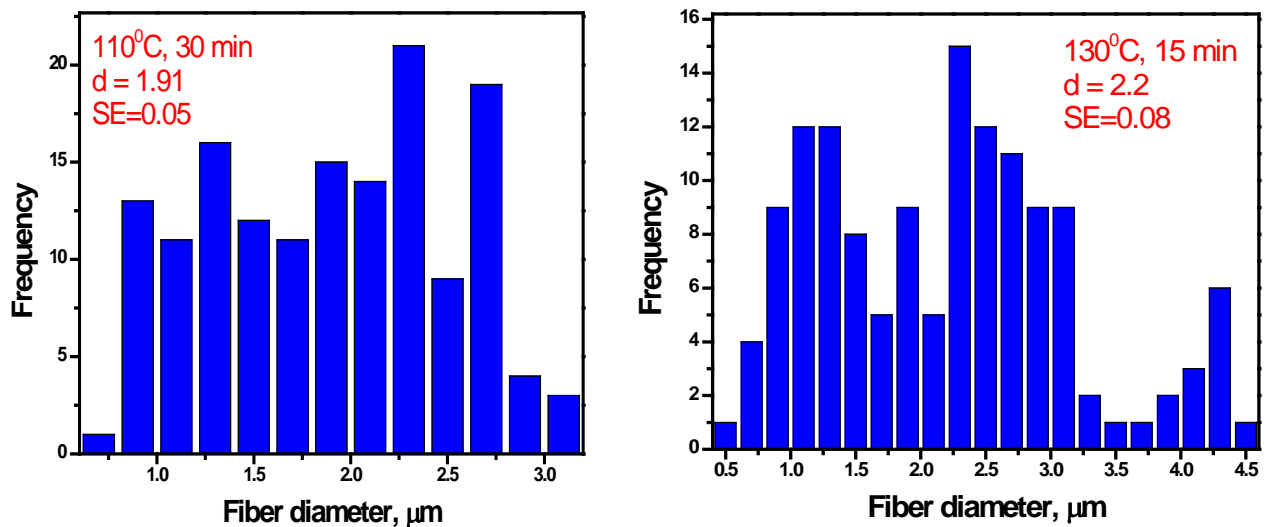


Fig.3. Washed spunbond samples I/S PP/Exceval.

It can be seen that treating 36 I/S PP/Exceval fabric in Jet Dyeing machine at temperature 110⁰C and 30 min gives the finer fiber diameter than treating 36 I/S PP/Exceval fabric in Jet Dyeing machine at temperature 130⁰C and 15 min. At the same time mechanical and physical properties of both samples are approximately the same as it was established in previous work.

To look into the possibility of spinning PP together with AQ55 and AQ65 the study of melting viscosity all these polymers was conducted. The rheological properties of PP, AQ55 and AQ 65 are depicted in Fig 4-7.

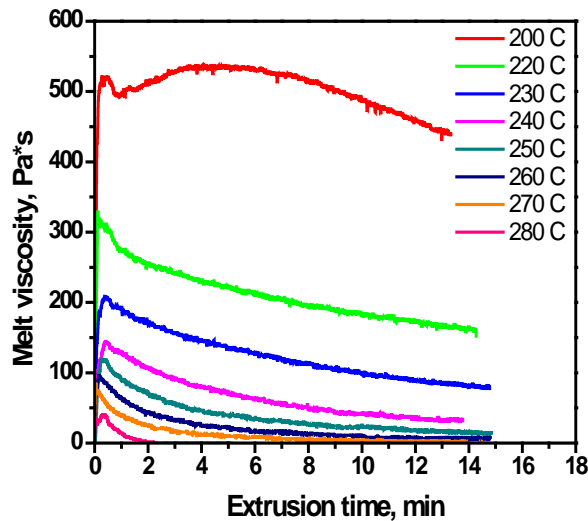


Fig. 4. Melt viscosity of AQ55 vs extrusion time

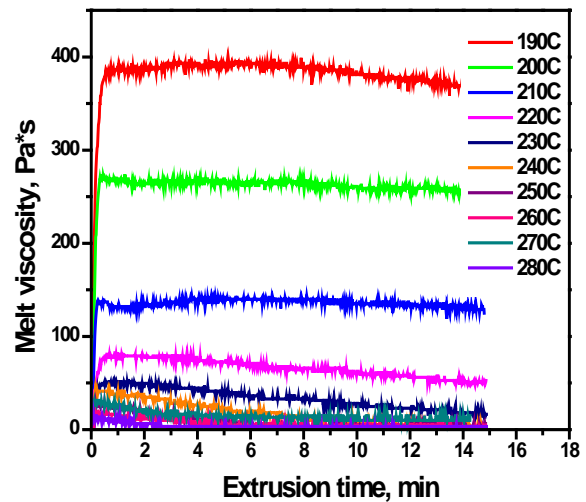


Fig.5. Melt viscosity of AQ65 vs extrusion time

It is clear that the melting viscosity of AQ 55 is higher than that of AQ 65. Also, AQ 65 shows more stable viscosity than AQ 55. The softening (melting) of AQ 65 starts at temperatures approximately 190⁰C while the melting of AQ 55 starts around 220⁰C.

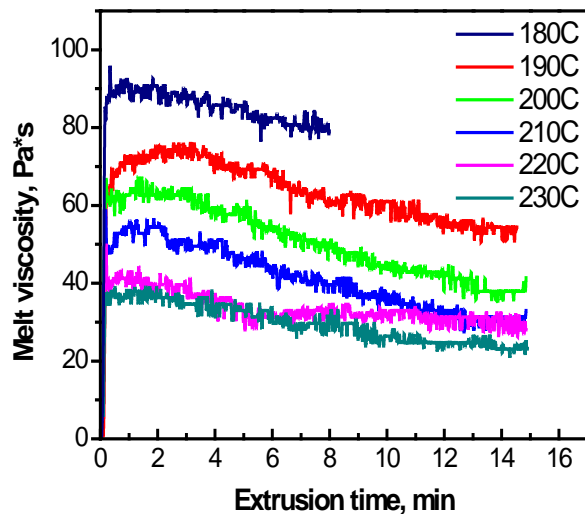


Fig. 6. Melt viscosity of PP vs extrusion time

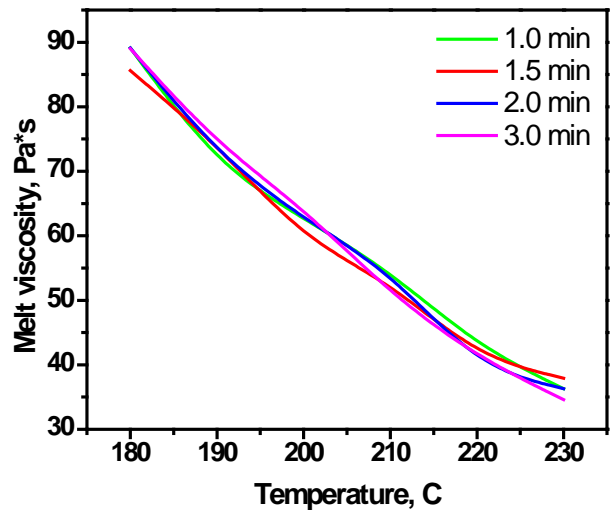


Fig.7. Melt viscosity of PP vs temperature

It can be seen that PP (Sonoco) starts to melt around 180⁰C and has almost linear dependence on temperature (correlation coefficient 0.99).

Comparative characteristics of PP, AQ55 and AQ65 melting viscosity are depicted in Fig.8.

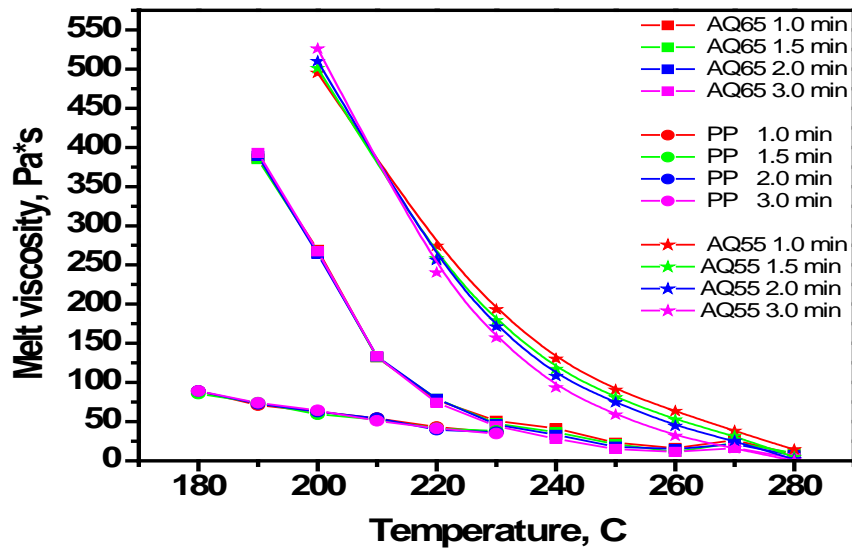


Fig.8. Melting viscosity vs temperature.

It has been revealed that PP and AQ 65 have comparable viscosity in the range of temperatures 220-230⁰C while PP and AQ 55 have matching viscosity around 240-250⁰C or even higher. It can be assumed that such difference in melting viscosity of PP and AQ55 will cause difficulties in spinning both of these polymers together. Problems can appear first in quenching zone and then in drawing zone. Since the melting point of PP is in the range 165-175⁰C, to crystallize it supposed to be cooled down by more than 80⁰C. On the other hand to solidify AQ 55 has to be cooled down by 30-40⁰C. Because of such different solidification conditions it can be possible that at the same cooling conditions AQ 55 will solidify while PP will be still in semi-liquid highly viscous state. Because of this, PP stays tacky and can cause roping. Finally, incomplete solidification makes attenuation almost impossible. Such a hypothesis was actually confirmed experimentally when we were trying to spin PP with AQ55. On the other hand, the spinning of AQ65 with PP did not cause any problems and the cross-sections of free fall fibers 36 and 108 I/S PP/AQ65 can be seen in Fig. 9, 10.

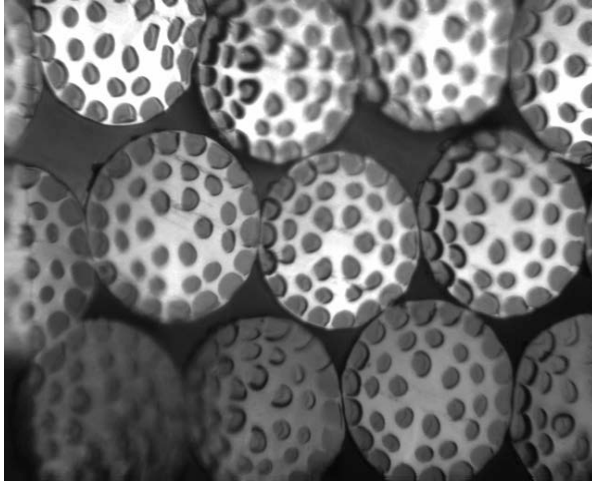


Fig. 9 Cross-sections 36 I/S 25/75 PP/AQ65 Fig. 10. Cross-sections 108 I/S 50/50 PP/AQ65

But, removing AQ65 from the obtained fabric is almost impossible task. Even treating such samples in Jet dyeing machine at temperature 120⁰C for 120 min did not cause removing of this entire polymer. Only very small portion of AQ65 was removed while the fabric lost its properties and aesthetic significantly.

Our attempt to spin AQ 55 with PP with the same number of islands and the same cross-sectional area as depicted above failed. PP in the outer layer of fiber did not solidify properly. Consequently, the fibers were tacky and formed ropes yielding poor fabric formation. In order to solve this problem, we increased the quenching zone length by 6 inches but the result was still negative. Finally, we found a temporary solution of existing problem by changing spinpack and cross section of fibers. New cross section of PP/AQ55 fibers depicted in Fig.11.



Fig.11. Cross-sections 25/75 PP/AQ55

Fig.12. Optical microscopic image of 18 I/S 25/75 PP/AQ55

New cross section has outer layer that contains AQ55 sea polymer instead of PP as islands and AQ 55 as sea. This change helped to isolate PP and fibers could be actually spun. But because of this the number of islands was reduced twice.

To remove AQ55 polymer 18 I/S PP/AQ 55 drawn as well as freefall fibers were washed. In contrast to AQ65, AQ55 was dispersed readily. The optical microscopic pictures of fibers before and after washing are depicted in Fig.12, 13 and 14.



Fig. 13. 18I/S 25/75 PP/AQ55, drawn

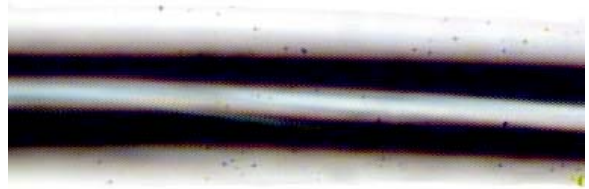


Fig 14. 18I/S 25/75 PP/AQ55, freefall

By examining Fig.12 to Fig.14, it can be seen even visually that there is some reduction in fiber size due to decreasing of AQ55 polymer content. The results of actual fiber diameter measuring can be seen from Table1.

Table1. The fiber diameter of washed and unwashed samples.

	Unwashed		Washed	
	Diameter, μm	Denier	Diameter, μm	Denier
25/75 PP/AQ, drawn	23.3	3.9	9.43	0.64
75/25 PP/AQ, drawn	27.6	4.6	14.76	1.3
25/75 PP/AQ, freefall	138.71	138.6	115.69	96.45
50/50 PP/AQ, freefall	189.52	238.5	170.34	192.7
75/25 PP/AQ, freefall	142.8	123.9	138.8	116

It can be seen that all fiber diameters were reduced after fibers were washed but the most significant reduction of diameter have drawn fibers.

The tensile properties of 18 I/S PP/AQ55 washed as well as unwashed fibers were tested and the results are depicted in Fig. 15-18.

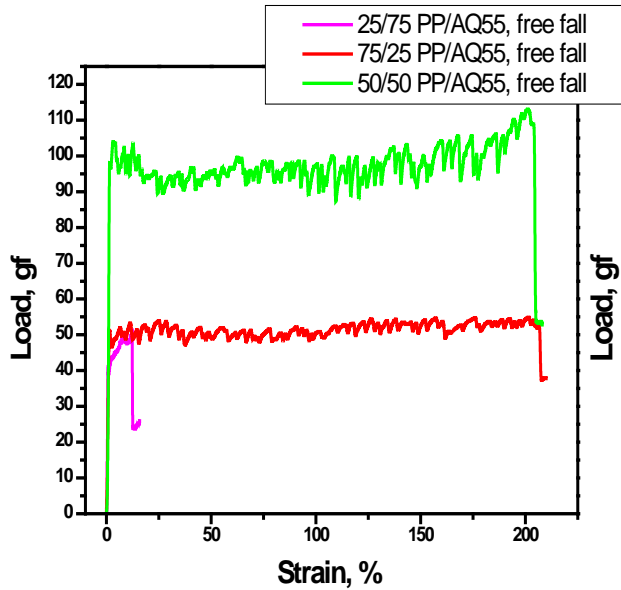


Fig.15. Load vs strain, unwashed fibers.

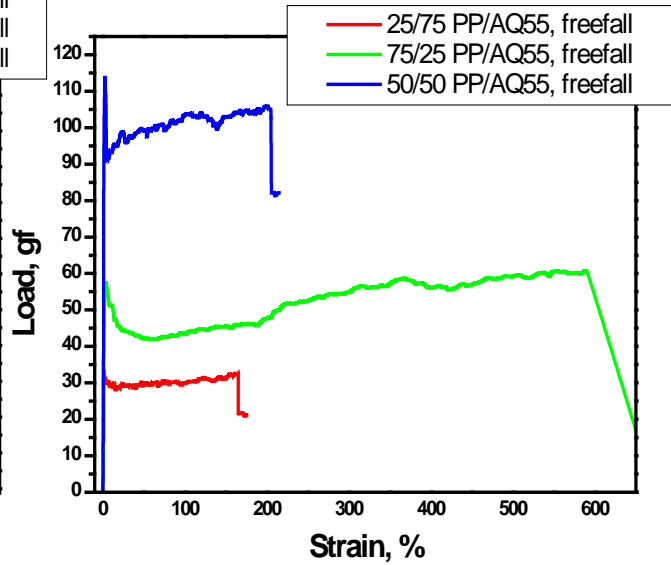


Fig.16. Load vs strain, washed fibers

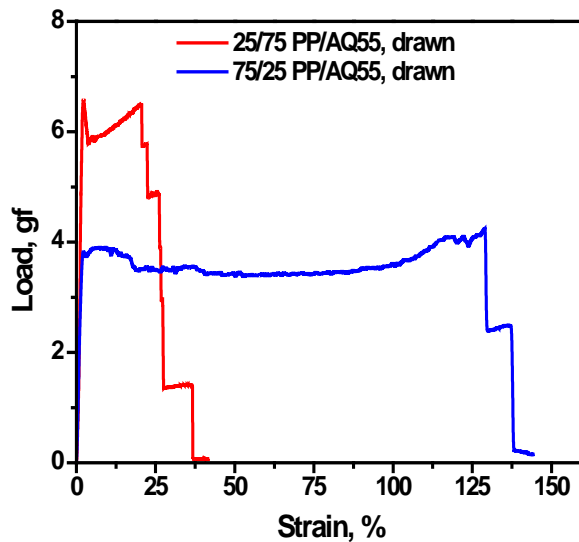


Fig.17. Load vs. strain, unwashed fibers

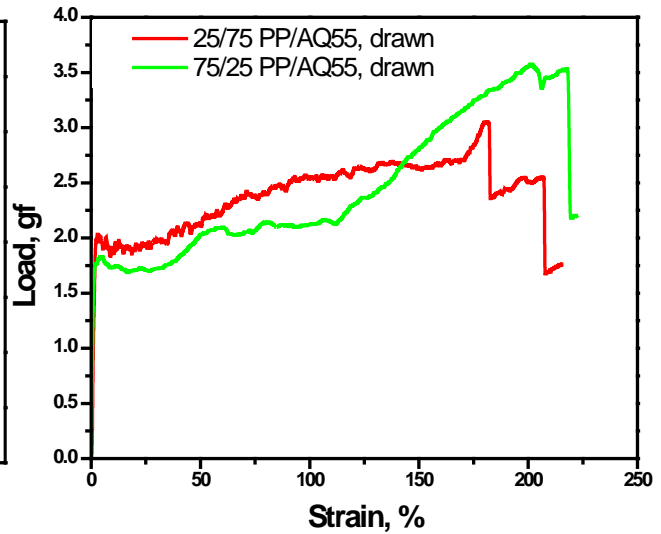


Fig.18. Load vs. strain, washed fibers

It was revealed that after complete or partial removal of AQ55 from fibers, the elongation at break was increased significantly. At the same time breaking force was decreased while tenacity increased. Such significant increase in elongation at break also supports our hypothesis about the problem of PP crystallization. In presence of AQ55, fibers are stiffer, they show higher resistance to deformation than pure PP fibers or fibers with smaller amount of AQ55. Because of this, load applied to these fibers is higher and elongation-at-break is lower compare to load and elongation-at-break of washed fibers. Also, because of different solidification conditions of

AQ55 and PP, AQ 55 fails earlier than PP. This cause the stress concentration on the interface if PP-AQ55 and as a result premature failure of a whole fiber. In addition, since PP did not crystallize properly, its chains are not ordered properly as well. PP responds to applied stress elastically and plastically. During plastic deformation polymeric chains are changing their orientation up to some point after which applied stress cause fiber failure. Such chain reorientation allows PP to be drawn to the high extent.

The results of tensile testing washed and unwashed 18 I/S PP/AQ55 are also summarized in Table 2,3,4 and 5.

Table 2. Tensile properties of 18 I/S PP/AQ55 freefall fibers.

	Unwashed			Washed		
	25/75 PP/AQ	50/50 PP/AQ	75/25 PP/AQ	25/75 PP/AQ	50/50 PP/AQ	75/25 PP/AQ
Breaking force, gf	50.2	113	53.5	33.2	106.6	61
Elongation at break, %	11.8	201	207	164	205	590
Tenacity, gf/den	0.36	0.47	0.43	0.35	0.55	0.53

Table 3. Tensile properties of 18 I/S PP/AQ55 drawn fibers.

	Unwashed		Washed	
	25/75 PP/AQ	75/25 PP/AQ	25/75 PP/AQ	75/25 PP/AQ
Breaking force, gf	6.52	4.26	3.08	3.58
Elongation at break, %	20.56	129.2	180	201
Tenacity, gf/den	1.67	0.92	4.8	2.75

Table 4. Tensile properties of 18 I/S PP/AQ55 freefall fibers.

	Unwashed			Washed		
	25/75 PP/AQ	50/50 PP/AQ	75/25 PP/AQ	25/75 PP/AQ	50/50 PP/AQ	75/25 PP/AQ
Initial modulus, gf/den	52.9	47	37	34	44	36.5
Secant modulus, gf/den	3.5	4.12	4.10	3.05	4.9	4.4

Table 5. Tensile properties of 18 I/S PP/AQ55 drawn fibers.

	Unwashed		Washed	
	25/75 PP/AQ	75/25 PP/AQ	25/75 PP/AQ	75/25 PP/AQ
Initial modulus, gf/den	130.22	52	167.6	131
Chord modulus, gf/den	15.38	8.3	29.22	13.3

DISCUSSION:

From the conducted research was determined:

To obtain the finest fiber diameter from 36 I/S 50/50 PP/ Exceval fabric Jet dyeing machine can be used at temperature 110⁰C and 30 min.

Use the AQ polymers over Exceval polymer is more preferable, because Exceval polymer has unstable viscosity at higher temperatures and could not be spun with PET or Nylon while AQ could be spun with these polymers. Also, to bond the web containing Exceval polymer hydroentanglement could not be used, because web is swelling under water jets and destroying. AQ polymers do not have such problems with hydroentangling.

On the other hand the use of AQ polymers also has some problems:

- hard to remove AQ 65 polymer from PP/AQ 65 spunbond samples;

- significantly different melting viscosity of PP and AQ 55 at lower range of temperatures;
- problem of solidification of PP during spinning PP/AQ 55 fiber;
- problem of drawing PP/AQ55 fibers.

Nevertheless, the usage of AQ55 over AQ65 is more preferable since it dissolves easily and the problems of spinning PP together with AQ 55 could be overcome.

FUTURE WORK

- ◆ To look more closely into the problem of solidification PP/AQ 55 fibers we have to develop spunbond samples made up of:
 - 100% AQ55;
 - 1 island PP, AQ55 sea (basically sheath-core cross-section);
 - 1 island AQ55, PP sea (basically sheath-core cross-section).
- ◆ Try to develop the computational model for the solidification and attenuation processes based on PP and AQ 55 polymers by first using the simplest cross section structure (sheath-core) in order to look into interaction of this two polymers
- ◆ In case of successfully solving the problem of spinning PP and AQ 55 we have to continue to develop PP/AQ 55 spunbond samples
 - 36, 108, 216, 360
 - 25/75, 50/50, 75/25
- ◆ Define the best known method for AQ 55 polymer removal.
- ◆ Perform the fiber and fabric characterization.

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