Splitting of Islands-in-the-Sea Fibers (PA6/COPET) During Hydroentangling of Nonwovens

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ABSTRACT

This paper summarizes the investigations of hydroentangled islands-in-the-sea (PA6/COPET) fiber webs. An increase in water jet pressure improved the tensile strength and fiber splitting while elongation at break decreased. Scanning Electron Microscope (SEM) micrographs and ANOVA (MS Excel ™) were used for characterizing fiber splitting and data analyses respectively. It can be concluded that with a new innovation in spinnerette design and modification of co-polyester structure, PA6/COPET, fibers can be split in the hydroentanglement process without dissolution of the sea component.

INTRODUCTION

Islands-in-the-sea is one type of bicomponent fiber whereby many fibrils of one polymer are dispersed in the matrix of another polymer. The fibrils are known as islands and the matrix is the sea. The sea can be any number of dissolving polymers, including polystyrene, co-polyesters (COPET), polyvinyl alcohol (PVA), thermoplastic starches, each requiring its own particular solvent [1-3]. William Haile et. al.[4], in their patent discloses sea co-polyesters that can be solubilized and removed in PH 5-6 hot water at temperature of 60ºC - 82ºC. The sea polymer serves to maintain a barrier between the micro fiber components and to provide a carrier to make intermediate handling of these fibers possible. In 1970s, various bicomponent fibers began to be made in Asia, notably in Japan, where the islands-in-the-sea was first developed by Toray [5]. The islands-in-the-sea fibers have excellent tensile properties, and provided good feel, softness, bursting and tearing strength for the nonwoven fabrics.

In the development of islands-in-the-sea fibers, many attempts have been made to increase the number of islands in the cross-section area of the sea. For example in June of 1999, Hills Inc. developed a new method of pack construction that resulted in fibers with 240 islands-in-the-sea. Currently fibers with 16, 37, 64, 240, 600, 900, and 1120 islands-in-the-sea fibers are being produced [1, 6, 7]. Hills Inc. a manufacturer of equipment for producing ultra-fine bicomponent micro fibers is doing research aimed at producing fibers having up to 3000 islands-in-the-sea [6].

It is well known that a co-polyester sea cannot be split or dissolved in water during hydroentanglement, so these fibers have been used mainly in the needle punching process to make nonwoven fabrics and then later split the fibers in alkaline medium. However recently, techniques such as copolymerization and physical blending are being used to improve the physical properties of different copolymers. Co-polymers have become more and more important, since their characteristics can be more easily tailored to fit specific applications. Researchers [8] have found that the acceptable mechanical properties of co-polyesters can be obtained when the crystallinity and the aliphatic content ratio are appropriate, so the degradation occurs easier in amorphous regions. Liancai Wanga et. al. [9] made a study to investigate the behavior of aliphatic/aromatic co-polyesters. From their studies they concluded that the co-polyesters containing many cyclohexanedicarboxylic acid (CHDA) units were characterized by higher water uptake and faster degradation due to the increased amorphous phase within them. From those studies it seems that it is possible to change the structure of co-polyester (sea) so as to tailor the properties and benefits to the hydroentanglement process. On this basis, it appeared possible to split a modified islands-in-the-sea fiber (polyamide 6 (PA6)/co-polyester (COPET)) during hydroentangling, compared to the accepted norm that these fibers can be split only in alkaline aqueous baths or organic solvents.

The hydroentanglement process has played an important role in nonwoven manufacturing for many years. Its main functions are: consolidation, fiber splitting, and pattern structuring, or fabric enhancement for woven fabrics. Many researchers have considered water jet pressure, nozzle geometry, water quality, fiber web substrates, impact force,

production speed, fiber types and web structure as some of the main parameters in hydroentanglement process [10, 11, 12, 13, 14]. Tensile strength is the main factor for characterizing hydroentanglement efficiency. Furthermore researchers [11, 15, 16] have demonstrated the effect of water jet pressure and specific energy on the tensile strength of hydroentangled nonwoven fabrics and concluded that there is an optimum pressure and specific energy to be used for obtaining maximum tensile strength of hydroentangled nonwoven fabrics. Recently P. Xiang et. al. [17] showed that the degree of fiber entanglement is linearly related to flow vorticity in the fiber web, which is induced by impinging water jets. However other researchers [1, 18, 19] have shown that the possible splitting rate of split fibers is a function of water jet pressure, impact force, nozzle diameter and polymer/co-polymer selection.

Michael Ellison et. al. [20] made a study using pulsed elliptical jets in hydroentanglement process using splittable fibers, and they concluded that pulsing an impinging jet flow through elliptical holes increases fiber splitting and bending and improved filtration efficiency in nonwovens. Christine Q. Sun et. al. [21] conducted a study on fiber splitting of side-by-side bicomponent meltblown filaments (PA/PP, PA/PE, PA/PET). They used water jet pressure levels of 80, 100, and 120 bars during the hydroentanglement process and concluded that bicomponent melt blown fibers were too weak to achieve overall fiber splitting by mechanical means because of very low molecular orientation.

Carlson et. al. [22] used splittable fibers to make hydroentangled nonwoven fabrics. They showed that using high pressure liquid streams on fiber webs formed from fiber components having relatively small deniers, caused a reduction of bending modulus. As a result, fiber entanglement of the web is enhanced so that the resultant fabric exhibits relatively high tensile strength.

Finally, Behnam Pourdeyhimi, et. al. [23] utilized hydro-energy (hydroentanglement process) for fibrillating a set of bicomponent fibers. From their invention, they discovered that islands-in-sea fibers can be made to split by hydroentangling without dissolution if the sea polymer is sufficient weak and particularly when the two components have little or no affinity for one another.

Even if many studies have been done in the area of hydroentanglement, very few studies have investigated the possibility of splitting islands-in-sea fibers without dissolution of the sea component. More work is still needed in this area. The aim of this study was to investigate the splitability and tensile properties of modified islands-in-the- sea fiber (PA6/COPET) during the hydroentanglement process. The results of this study can be beneficial for the acceleration of research work for co-polyester based islands-in-the-sea fibers, to explore many possible ways of utilizing these fibers in hydroentanglement applications without using chemical solvents.

EXPERIMENTAL

Materials

The carded webs of 100 grams per square meter (gsm) of modified islands-in-the-sea bicomponent fiber, (70%PA6/30%COPET) having linear density 4.6 dtex with 37 polyamide 6 (PA6) islands was used. The co-polyester (COPET) used has the glass transition temperature (Tg) in the range of 50ºC - 60ºC, melting point in the range of 242ºC -250ºC (peak melting point 246.051 ºC). The samples of carded webs were in the range of 250mm (width) × 770 mm (length). The carded webs were produced by longitudinal layering technique; therefore the properties of carded webs are anisotropic in nature because of the unidirectional arrangement of fibers. In order to investigate the fiber splitting behavior of these fibers without going through the carding process, fiber bundles of 2 grams each were used. The fibers were purchased from Shanghai Synthetic Fiber Research Institute and designed for use in hydroentangled applications.

Hydroentangled Fabrics Preparation

The Fleissner hydroentanglement machine at Donghua University was used for the manufacturing of hydroentangled nonwoven fabrics. Pressure levels of 60, 80, 100, 120, 150, and 200 bars were used. The fiber webs were hydroentangled on one side using two passes and then on the opposite side using the same passes. The processing speed was 2 m/min, jet density 16 jets/cm and nozzle orifice diameter 0.127 mm. The samples of fiber bundles of 2 grams each were gently opened by hand and hydroentangled with pressure levels of 60, 100, and 200 bars while other processing parameters were the same as that mentioned above. After consolidation the samples were air dried at room temperature.

PHYSICAL MEASUREMENTS

Tensile Strength

Tensile strength was measured by Universal Tensile Testing Machine (WDW-20), made in China according to ASTM D 5035-95 (strip method) [24].

The dimensions of the samples were 50 mm (width) by 170 mm (length) in crosswise direction (CD) and in machine direction (MD). The gage length was 100 mm and the testing speed was 250 mm/min. Five samples in each direction were tested and the results are represented with the averages obtained from measurements in each case.

Fiber Splitting Evaluation

The hydroentangled nonwoven sample processed using 200 bars were marked with a straight line along their width on upper and bottom surface. On each line the 12 samples for SEM were taken from left to right side of the sample (see *Figure 1*). For other samples processed with 60, 80, 100, 120, and 150 bars and for samples of fiber bundles processed by 60, 100, and 200 bars, only 4 samples for SEM were taken randomly from upper surfaces of each sample. The reason for this was due to high costs in using Scanning Electron Microscope. The samples were coated with platinum and the microscopic structure was observed before and after hydroentanglement by Scanning Electron Microscope (SEM), JSM-5600LV made in Japan of resolution 3.5-4.5 nm and over a range of magnification from 18 to 300,000. Based on the SEM photos, the mean fiber diameters were calculated using 20 fibers seen on each of the SEM images of hydroentangled carded webs.

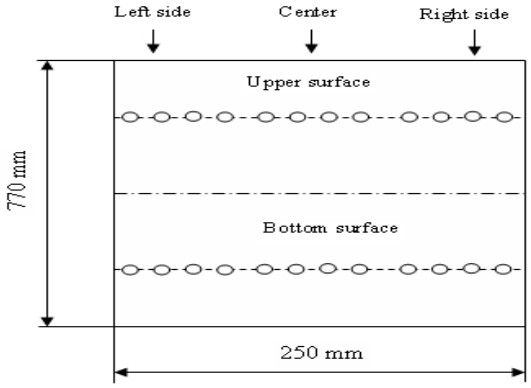


Figure 1. The sample arrangement for SEM photos for sample processed by pressure of 200 bars

For evaluating fiber splitting two methods were used. First method was used just for visualization on the SEM photos and investigating the splitting rate by considering partial, moderate or full splitting. The second method was used to quantify the percentage of split fibers only for hydroentangled samples processed by pressure of 200 bars. On each SEM photos the splitting percentage was investigated at the center line of each SEM photos from left to right side for all 12 photos from each surface. The amount of split fibers and non-split fibers were counted in each case. The percentage of non-split fibers was calculated using the following relationship:

% of non-split fibers=

$$\frac{37 \times \text{Non} - \text{splitfibers}}{\text{Splitfibers} + (37 \times \text{Non} - \text{splitfibers})} 100\%$$

Then the percentage of split fibers was determined by:

Percentage of split fibers

$$= 100\% \{1 - (\frac{37 \times \text{Non} - \text{splitfibers}}{\text{Splitfibers} + (37 \times \text{Non} - \text{splitfibers})})\}$$

where 37 is the number of islands

The percentage of split fibers for each SEM photos was calculated and the range was given. For the case where non-split fibers were not observed the splitting percentage was considered as 100%. This method has the disadvantages of being time consuming and not analyzing very clearly the entire layer between upper and bottom surface of nonwoven fabrics; however, it can give a rough estimation of split fibers.

RESULTS AND DISCUSSION

Table I shows the average values of tensile strength and elongation at break with their coefficient of variations of hydroentangled PA6/COPET fabrics. From *Table I* it can be noticed that the ratio of tensile strength (MD/CD) for all fabrics are in the range of 2.97-3.79. The variation of tensile strength in machine direction and crosswise directions demonstrates the anisotropic nature of carded webs.

Table I: Tensile strength and elongation at break of PA6/COPET fabrics

Pg		Tensile Strength (N/5 cm)		Elongation at break (mm)		MD/ CD
bars		MD	CD	MD	CD	ratio
60	AV	245.26	73.96	76.94	136.10	3.33
	CV (%)	16.24	8.93	6.16	6.97	
80	AV	273.46	75.22	62.05	90.18	3.64
	CV (%)	6.46	10.91	7.14	12.83	
100	AV	283.3	76.17	59.17	87.40	3.79
	CV (%)	13.07	12.83	11.10	10.46	
120	AV	289.42	88.06	57.89	75.94	3.29
	CV (%)	11.24	7.3	5.5	8.98	
150	AV	290.06	91.01	54.79	70.04	3.18
	CV (%)	15.11	17.36	4.55	13.73	
200	AV	321.96	108.31	54.22	62.57	2.97
	CV (%)	14.15	14.94	8.7	5.62	

Note: AV is average, CV is coefficient of variation and MD/CD is the strength ratio of machine direction and crosswise direction

The coefficient of variation (CV) for tensile strength and elongation at break of PA6/COPET fabrics (in *Table I*) are in the range of 6.46-17.36% and 4.55-13.75% respectively. Probably the reason for higher coefficient of variation is due to non-uniformity of fiber splitting within fiber webs and non-uniformity of fiber web thickness.

TENSILE PROPERTIES

The tensile properties were evaluated using analysis of variance (ANOVA) in MS Excel ™ to investigate the effect of water jet pressure on tensile strength and elongation at break. The means comparison of tensile strength and elongation at break depending on pressure levels of 60, 80, 100, 120, 150, and 200 bars was conducted. *Table II* shows ANOVA summary of evaluation of mean tensile strength and elongation at break depending on pressure levels. From *Table II*, ANOVA analysis shows that there is statistical significant difference with pressure levels treatment on fabric tensile strength and elongation at break. The analysis show that water jet pressure plays important role in hydroentanglement process, and this fact is also supported by *Figure 2* and *Figure 3*.

It can be seen clearly from *Figure 2* and *Figure 3* that with increases of water jet pressure, the tensile strength at break increased while the elongation at break decreased. Generally in a hydroentanglement process, water jets strike the fiber web, displacing, twisting, and knotting individual fibers. As the process continues the water jets strike the screen mesh, and are deflected, forming turbulent effects within fiber webs which causes the formation of many entangled fibers. From previous researchers [11, 15, 16], it has shown that increasing water jet pressure to the optimum value improves the tensile strength. On the other hand, fiber splitting may also improve the tensile strength [22, 23]. Based on these studies, we can conclude that a combination of raising water jet pressure (see *Figure 2*) and fiber splitting (see *Figure 4*) may be the reason for the enhanced fiber entanglements, resulting in an increase of tensile strength.

It is expected that tensile strength in the machine direction (MD) is higher than crosswise direction (CD), because of the anisotropic nature of carded webs. *Figure 2* indicates that, as water jet pressure increases, the tensile strength increased. That no reduction of tensile strength is observed indicates that assuming no fabric damage, the optimum pressure may still be higher than the 200 bars being used in this study.

Figure 3 shows the expected decrease in elongation with increased water jet pressure, as well as the expected higher elongation in the direction crosswise of the fiber orientation created by carding.

Table II: ANOVA summary of tensile strength and elongation at break depending on pressure levels

ANOVA	FOR TENSILE STRENGTH MD					
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	15670.58	5	3134.116	4.570815	0.004537	2.620654
Within Groups	16456.32	24	685.6798			
Total	32126.89	29				
ANOVA	FOR TENSILE STRENGTH CD					
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	4414.875	5	882.975	3.095376	0.02697	2.620654
Within Groups	6846.147	24	285.2561			
Total	11261.02	29				
ANOVA	FOR ELONGATION MD					
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	1763.227	5	352.6454	17.65282	2.38E-07	2.620654
Within Groups	479.4413	24	19.97672			
Total	2242.668	29				
ANOVA	FOR ELONGATION CD					
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	17138.97	5	3427.794	45.78828	1.68E-11	2.620654
Within Groups	1796.684	24	74.86182			
Total	18935.65	29				

Note: Groups in this case are tensile strengths and elongation at break in different pressure levels of 60, 80, 100, 120, 150, and 200 bars

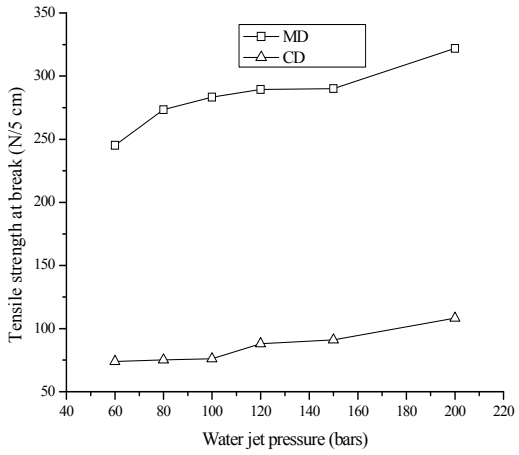


Figure 2. The effect of water jet pressure on tensile strength of bicomponent fabric

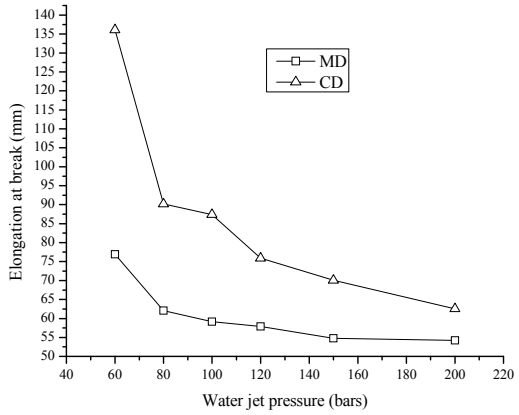


Figure 3. The effect of water jet pressure on elongation at break of bicomponent fabrics

FIBER SPLITTING

Figure 4 shows SEM photo of PA6/COPET carded fiber web before hydroentanglement. *Figure 4* shows that there were cracks along PA6/COPET fibers after carding process, probably resulting from the weak interfacial bonding between polyamide and co-polyester as well as friction between fibers and carding wires.

Figure 5 shows the photomicrograph of fiber bundles hydroentangled with water jets pressure of 60, 100, and 200 bars, and indicates that the fibers in the fiber bundles were partially split.

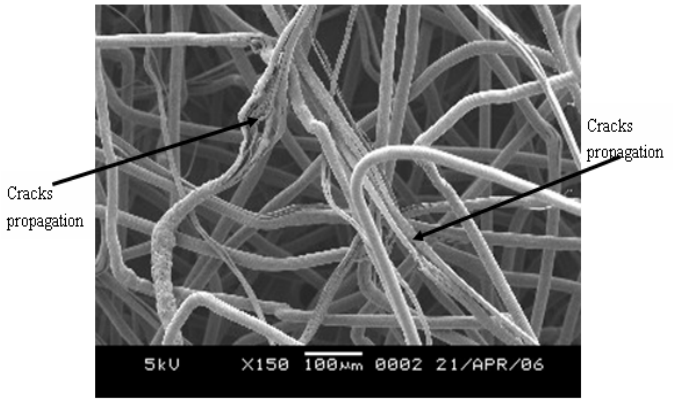


Figure 4. SEM photos of PA6/COPET carded fiber web before hydroentanglement

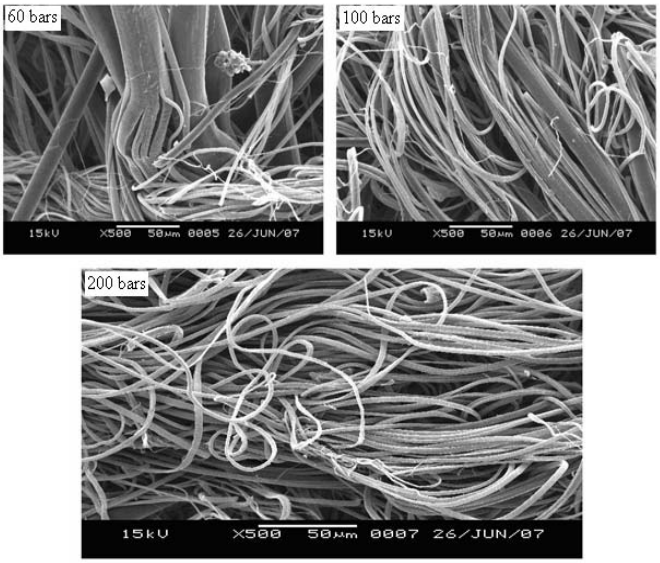


Figure 5. SEM photos of fiber bundles of PA6/COPET after hydroentanglement

As water jet pressure increased to 200 bars, the fiber splitting was fairly good as can be seen in *Figure 5* even without carding.

Figure 6 shows the photomicrograph of PA6/COPET carded fiber webs hydroentangled using pressure levels of 60, 80, 100, 120, 150 and 200 bars. In *Figure 6* it can be seen that as the water jet pressure increased, the entanglement of fiber was improved and the fibers diameters were reduced in many fibers. In the case of fiber diameter, it was observed from *Figure 4* that the average fiber diameter of PA6/COPET before hydroentanglement was 24.42µm with CV of 6.62% and after

hydroentanglement based on *Figure 6* at 200 bars was 4.71μm with CV of 12.66 %. The small fiber diameters observed reflects the splitting of PA6/COPET fiber. It is well known that the conventional co-polyesters in islands-in-the-sea fibers do not dissolve or split in a water jet process. But the very interesting results from this study open new possibilities, suggesting more research is needed to better understand the splitting mechanism of these fibers. This may lead to both improved combinations and improved processing of these fibers, allowing producers to move away from chemical solvents for splitting purposes. By comparison of *Figure 5* and *Figure 6*, it can be noted that the splitting of islands-in-the-sea fibers of hydroentangled carded webs was better, probably because the crack propagation on fibers or pre-damaging of fibers during carding makes them more susceptible to later splitting (see *Figure 4*). Possible reasons for the enhanced splitting behavior include:

1. The modification made in the co-polyester structure, such that the amorphous regions were increased with respect to the crystalline regions – producing lots of cracking during hydroentanglement (see *Figure 6* SEM photos of PA6/COPET nonwoven fabrics after hydroentanglement).

2. The special spinneret used to manufacture these fibers, regarding the arrangement of islands polyamide 6 (PA6) in the sea (co-polyester). In the non-splittable fibers, the surface bonding area of co-polyester to the polyamide is very large; therefore, the interfacial bonding becomes very strong and as a result the fibers cannot be split easily.

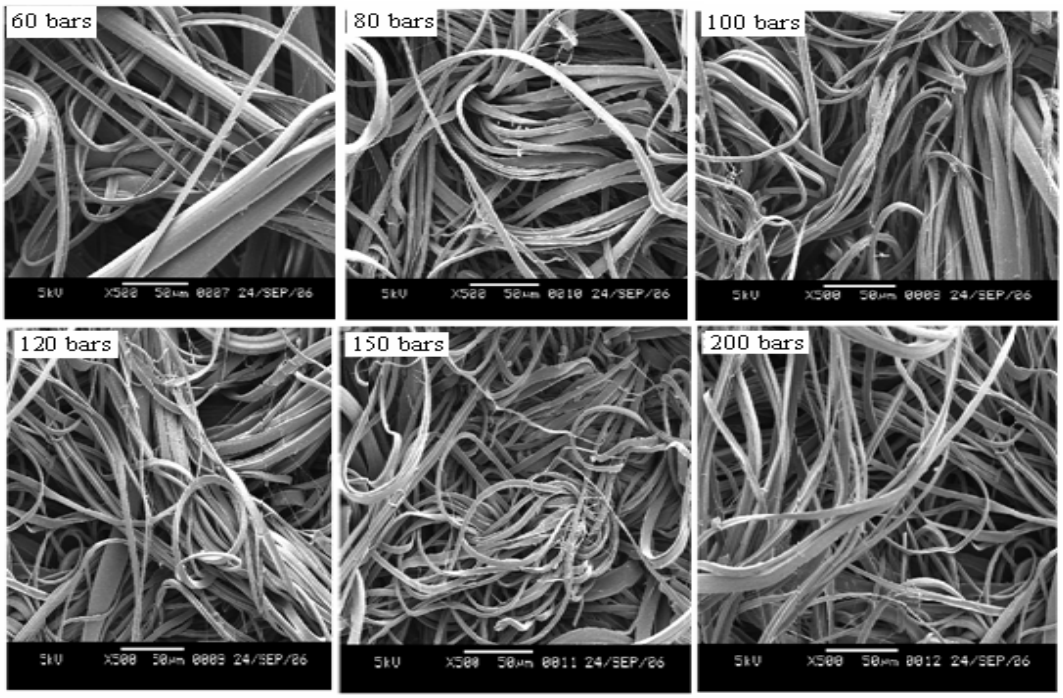
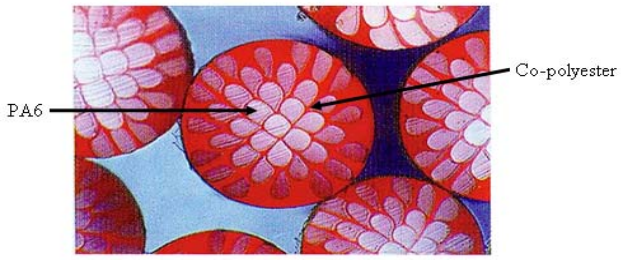


Figure 6. SEM photos of PA6/COPET nonwoven fabrics after hydroentanglement

The structures of the traditional islands-in-the-sea fiber can be seen elsewhere in the literature [3, 5]. For the case of these modified islands-in-the-sea fibers, the walls of the sea between the islands appear to be very thin and perhaps weak, while the islands solidifying and crystallizing within the matrix during processing become stronger. Therefore with implementation of high mechanical forces on these modified island-in-the-sea fibers, the sea components damaged easily and remain in contact with islands. Furthermore it can be seen in *Figure 6* that the fibers split shows small dots or attachments which may be the presence of co-polyester particles on the surface of split fibers. This reflects the remains of co-polyester on the island (PA6) surfaces since it does not dissolve in water.

Figure 7 shows the structure of modified PA6/COPET fiber.



Source: Brochure of Shanghai Synthetic Fiber Research Institute

Figure 7: Cross-section of 37 islands based (PA6/COPET)

Figure 8 shows the cross-section of islands-in-the-sea (PA6/COPET) bundles hydroentangled with pressure of 200 bars while *Figure 9* shows the enlargement of one fiber within the cross-section of fiber bundles processed by 200 bars. *Figure 10* shows the cross-section of islands-in-the-sea (PA6/COPET) hydroentangled carded fiber webs treated with pressure level of 200 bars.

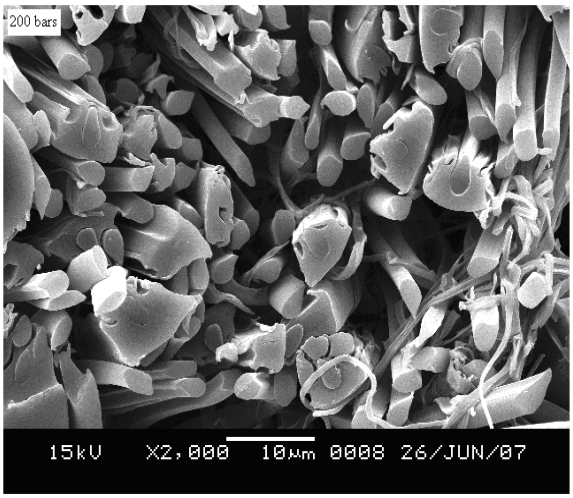


Figure 8. SEM photo of fiber bundles of PA6/COPET fiber web cross-section after hydroentanglement

From *Figure 8* and *Figure 9* it can be seen clearly that the fiber was only partially split. This may be due to large thickness of fiber bundles, showing entire fibers were not impacted sufficiently with high jet forces to make them split easily. *Figure 10* illustrates that other PA6/COPET fibers were split and furthermore it is indicated that some of fibers were fully split while others partially split. Based on *Figure 6* and *Figure 10* we can conclude that the splitting efficiency was moderate. According to the method used for quantifying the percentage of fiber split in hydroentangled fabrics processed by using 200 bars, the average percentage of fiber splitting was in the range of 60-80% (considered only in *Figure 6* of carded hydroentangled fabrics treated with 200 bars).

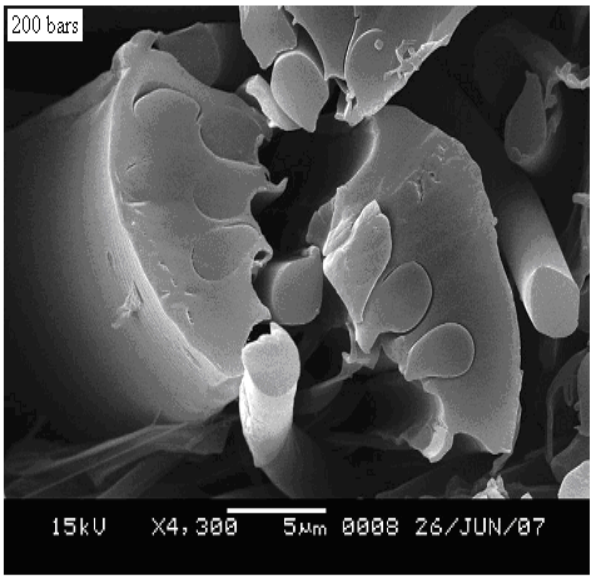


Figure 9. SEM photo of cross-section area of PA6/COPET fiber within fiber bundle processed by pressure of 200 bars

Figure 10. SEM photo of PA6/COPET fiber web cross-section after hydroentanglement

CONCLUSION

This study evaluated the splitting behavior and tensile properties of a modified islands-in-the-sea fiber (PA6/COPET) in a hydroentangled nonwoven fabric. The fabric tensile strength and fiber splitting efficiency increased with higher water jet pressure. The ANOVA analysis shows that water jet pressure contributes significantly to the resultant tensile properties of the fabric. From the trend of the tensile strength, it can be concluded that the optimum water jet pressure for consolidation of these fibers can be higher than 200 bars. This as well as developing more easily splittable modifications at somewhat lower pressures should be investigated. The splitting of modified PA6/COPET fibers in the hydroentanglement process occurred without dissolution of sea components and is probably due to the weak and thinner walls of sea component between the islands. These preliminary results should encourage the modification of other co-polyester based fibers for use in the hydroentanglement process. Such bicomponent possibilities offer many advantages through the use of hydroentangling to produce microfibers without the use of chemical solvents, thus reducing processing and environmental costs.

ACKNOWLEDGEMENT

The authors will like to acknowledge the financial support received from Tanzania Industrial Research and Development Organization (TIRDO) and China Scholarship Council (CSC).

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