

EFFECT OF AIR QUENCHING ON CHARACTERISTICS OF THERMOPLASTIC POLYURETHANE MELTBLOWN NONWOVEN

ERİYİK ÜFLEME YÖNTEMİYLE ÜRETİLEN TERMOPLASTİK POLİÜRETAN DOKUSUZ YÜZEYLERİN ÖZELLİKLERİNE İKİNCİL SOĞUK HAVANIN ETKİSİ

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ABSTRACT

Nonwoven products have a wide usage area in many sectors and industries. Meltblowing (MB) is a process presenting versatile and cost effectiveness in the production of nonwovens from polymers. Characteristics of meltblown nonwovens have depended on highly some process variables and polymer used. Although polypropylene generally is preferred in meltblowing process due to its low cost, thermoplastics polyurethane (TPU) elastomers have superior properties. Very limited information has been reported about the characteristics of meltblown TPU nonwovens. In addition to process variables, quenching significantly affects meltblown nonwovens. In this study, the effect of die-collector-distance (DCD), air pressure and air quenching on meltblown TPU nonwoven was investigated. It was observed that air quenching reduces air permeability and increases hydrostatic heads that means the increment in resistance to leakage. Also air quenching increases tear strength of nonwoven. High orientation formed during the production with the assist of air quenching also plays an important role in fiber crystallization.

Keywords: Thermoplastics polyurethane, Nonwoven, Air quenching, Meltblowing, Process variables.

ÖZET

Dokusuz yüzey ürünler pek çok sektörlerde ve endüstrilerde geniş kullanım alanına sahiptir. Eriyik üfleme polimerlerden dokusuz yüzeylerin üretiminde çeşitlilik ve maliyet etkinliği sunan bir üretim şeklidir. Eriyik üfleme dokusuz yüzeylerin karakteristikleri büyük oranda kullanılan polimere ve bazı üretim değişkenlerine bağlıdır. Her ne kadar polipropilen ucuz maliyetinden dolayı eriyik üfleme yönteminde genellikle tercih edilse de termoplastik poliüretan elastomerler (TPU) daha üstün özelliklere sahiptir. Eriyik üfleme TPU'nun karakteristikleri hakkında sınırlı bilgi iletilmiştir. Üretim değişkenlerine ek olarak ikincil hava soğutma eriyik üfleme dokusuz yüzeylerin özelliklerini önemli derecede etkiler. Bu çalışmada, eriyik üfleme TPU dokusuz yüzeylere kalıp-toplayıcı mesafesi (DCD), hava basıncı ve ikincil soğuk havanın etkisi araştırıldı. İkincil soğuk havanın hava geçirgenliği azalttığı ve sıvı sızıntısına karşı direnci ifade eden sıvı geçirgenliği basıncını azalttığı gözlemlenmiştir. Ayrıca, ikincil hava soğutma dokusuz yüzeyin yırtılma dayanımını artırır. Ayrıca, ikincil hava soğutmanın yardımıyla üretim sırasında oluşan yüksek oranda lif yönlenmesi lifin kristalizasyonunda önemli bir rol oynamaktadır.

Anahtar kelimeler: Termoplastik poliüretan, Dokusuz yüzey, Hava soğutma, Eriyik üfleme, Üretim değişkenleri

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1. INTRODUCTION

The most important advantage of the MB process is that fundamentally all thermoplastics polymers can be processed by MB technology (1,2). Recently, elastomers including thermoplastic polyurethane (TPU) have been a focus of MB

research because of their unique properties such as high elasticity in all directions, good shore hardness for a given modulus, high abrasion and chemical resistance, excellent mechanical and elastic properties, low stress relaxation and resistance to long-term cyclic flex failure. Furthermore,

TPUs have blood/tissue compatibility, structural versatility and hydrophilic compatibility (3,4). The TPU is one of the thermoplastic elastomers produced from molten state by conventional process. The TPU has rubber like properties upon solidification. TPU has been made from soft and hard segments which have not miscible in each other resulting in a phase separated structure. Its stretch and annealing properties depends on degree of crystallinity in hard segment (5).

Meltblowing process is a single step process that converts polymeric raw materials into the web structure, a fibre network. Schematic view of meltblowing process is presented in Fig. 1. Meltblowing process includes five constituents that are the extruder, metering pump, die assembly, web formation and winding. The polymer in the form of beads, pellets, chips or granules is fed into the extruders from the hopper. In extruder, polymer is heated and then melted until desired temperature for application or viscosity is reached. Several additives can be added into the polymer in this stage in order to increase or modify the properties according to the aims planned.

The extruder consists of three zones which are the feed zone, the transition zone and the metering zone as seen from Fig. 1. In the feed zone, polymer is subjected to pre-heating. After pre-heating, softened or melted polymer is transferred into transition zone. In transition zone, polymer melted and homogenized is compressed. And then polymer is transferred into the metering zone. Metering pump, known also as a gear pump, provides for pressure required in the extruder in order to transfer the polymer melted uniformly and consistently into the die assembly in ways such as viscosity, pressure and temperature. Metering pump has two interlocking wheels rotating opposite ways according to each other. The melted polymer is obtained from the extruder and transferred by gear tooth and then discharged to the die assembly system. Die assembly is the most important stage of the meltblowing system and responsibly for the quality of fibres produced. As shown in Fig. 2, die assembly has three components which are the polymer feed distribution plate, the die nosepiece and the air manifolds. In these three components, the temperature is hold above

215°C in order to keep uniformity and quality of web structure. But, this temperature may change according to polymers type used. The feed distribution plates are responsible for having polymer flow across the plate. Keeping the plate at a consistent and suitable temperature is highly important in order to have polymer flowing and prevent it from changing its properties. Feed distribution shape is also crucial since it affects distribution of polymer.

The die nosepiece is an important component of the die assembly and highly responsible for diameter, quality and uniformity of web structures. In this respect, the design and production of the die tip is very crucial and requires precise measurement. The air manifold, also known as air knives, is responsible for supplying high velocity air which is called as primary air and assists in drawing the polymer to form microfibers. The manifold is located on the sides of the die nosepiece and hits the polymer with hot, high velocity air when it reaches the exit of die tip. Air is hotter than polymer so as to keep the polymer in liquid form. At this stage, formation of web structure starts. Turbulent air fractures polymer stream and produces microfibers beginning to twist. Secondary air, also known as surrounding air, cools microfibers falling toward the moving collection drum. Fibres are still solidified. In this respect, fibres are drawn to diameter desired while still in semi-molten form. Fibres reach to collection screen in this stage without any fibre attenuation and rapidly quench producing low crystallinity. Lower crystallinity presents lower fibre strength. If cooler air is used, the polymer will not be sticky upon contact with the collection belt and will not self-bond. Thus, web structure will need to be post-bonded before wind-up. Uniformity is important in web characteristics. Uniformity of web is affected by uniformity of fibre distribution in air stream. Poor die design or non-uniform ambient air negatively affects the air stream and fibre distribution resulting in a non-uniform web structure. The final stage of meltblowing process is winding and finishing. Fibres which are still hot when laid down, produce a bonded web ready for wind-up. Finishing and bonding can be done both before and after wind-up according to requirements. Some properties such as smooth and pattern of surface, strength, abrasion resistance, density and thickness of web change by depending on finishing.

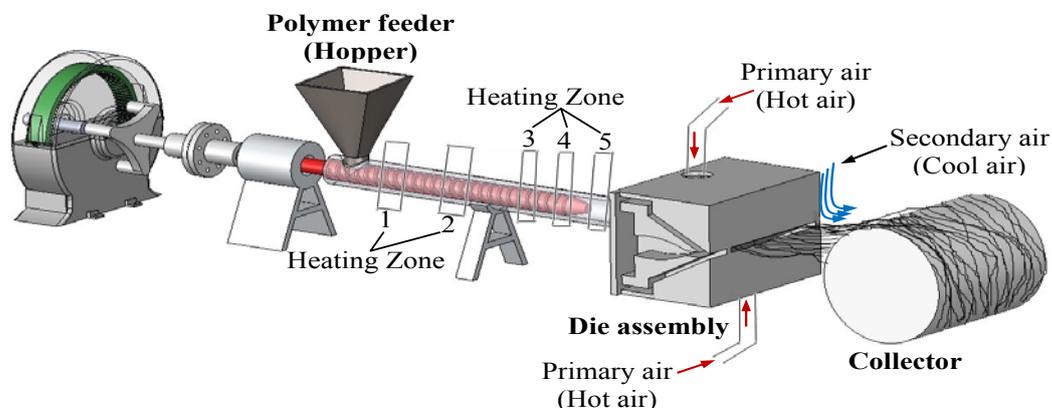


Fig. 1. Process of meltblowing technique.

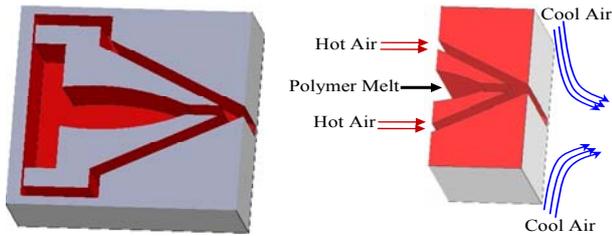


Fig. 2. Die assembly.

TPU is melt processed into single fibers, yarns, spunbond and meltblown into nonwovens. The main advantage of the MB process is its ability to handle a wide variety of fiber forming polymers and blends of polymers. Very little information about the polyurethane meltblown nonwoven has been reported. Very limited information concerning meltblowing and crystallization kinetics of TPU has been published.

The meltblowing process is a complex one that involves turbulence, which is poorly understood by the scientist even today. Isolation of experimental factors is difficult because of highly variable interaction. The multi-filament environments and factors such as humidity of the processing room and quench air temperature wildly change boundary conditions (6). In this respect, the aim of this study is to clarify the effect of air quenching on characteristics of meltblown thermoplastic polyurethane nonwoven. Bresee and Qureshi (7) studied the effect of water quenching on properties of polypropylene meltblown nonwoven. They found that the influence of quenching on web structure depends on greatly on the processing conditions used. Lee and Wadsworth (8) investigated fiber and web formation of meltblown TPU. They observed the effect of meltblowing process variables such as DCD and air velocity on some characteristics of TPU nonwoven. They concluded that TPU polymer has more complexity at fiber and web formation. So further studied is needed. As understood clearly from this literature survey, studies done are very limited. This paper is the first paper investigating the effect of air quenching on characteristics of meltblown thermoplastic polyurethane nonwoven in detail.

2. EXPERIMENTAL STUDIES

In this study, the influence of some variables on characteristics of thermoplastic polyurethane (TPU) meltblown nonwovens was investigated. The TPU was used for meltblowing process to produce web structure. The TPU was selected due to its superior properties such as elasticity, transparency, resistance to oil and grease, chemical resistance, abrasion, mechanical properties like shear strength etc. TPU can provide a considerable number of physical property combinations making it an extremely flexible material adaptable to dozens of uses. The TPU was obtained commercially from ExxonMobil Chemical Company, Baytown, TX.

Main production settings such as extruder temperature, connector temperature, die temperature, screw pressure, screw speed, extruder current, and collector speed are given in

Table 1. Main production settings.

Production setting	Zone	Value
Extruder Temperature (°C)	1	215
	2	226
	3	235
Connector Temperature (°C)		232
Die Temperature (°C)		220
Screw Pressure (Psi)		203
Screw Speed (Hz/rpm)		6.4 / 19
Extruder Current (amps)		4.3
Air Temperature (°F)		550
Collector Speed (m/min)		225 / 8.36
Die hole diameter (inches)		0.018

Meltblown nonwoven was produced by TPU polymer which has 210 °C, 10 kg melt flow index (MFI) conditions and 35 g/10min MFI. Production variables presented below dominate many variables of melt blown process which affect the properties of nonwoven.

- Quenching
- Die-collector-distance (DCD)
- Air pressure

Sample codes and production factors of the TPU meltblowing process are presented in Table 2.

Table 2. Sample codes and production factors.

Sample Code	Die Air Pressure (Psi)	DCD (cm)	Air Quenching
S1	10	15	Off
S2	10	15	On
S3	10	25	Off
S4	10	25	On
S5	10	40	Off
S6	10	40	On
S7	15	15	Off
S8	15	15	On
S9	15	25	Off
S10	15	25	On
S11	15	40	Off
S12	15	40	On
S13	20	15	Off
S14	20	15	On
S15	20	25	Off
S16	20	25	On
S17	20	40	Off
S18	20	40	On

The mass per unit area of nonwoven is expressed in grams per square meter. It was determined by cutting test pieces with 10 cm diameter from a nonwoven web and weighting them using the Mettler AE 240 tester. The basis weight of the fibre nonwoven media sample was calculated according to ASTM D 3776 in g/cm².

Air permeability of nonwoven is very crucial for applications. Air permeability is affected by the fibre diameter and the pore size of nonwoven media. Air permeability is the rate of

airflow passing perpendicularly through area of 38 cm² under 125 Pa pressure. Air permeability was done according to ASTM D 737-96. The average of 10 measurements of each sample was determined using TEXTEST FX3300 air permeability tester.

A nonwoven fabric specimen is mounted on the test head reservoir. The specimen was subjected to a standardized water pressure, which is increased at a constant rate until leakage appears on the specimens. Water pressure is measured as the hydrostatic head height reached at the first sign leakage in the three separated areas on the specimens. A higher value indicates greater resistance to water penetration. The average of five samples from each sample was calculated using TEXTES FX 3000 hydrostatic head tester. Hydrostatic head test was done according to AATCC 127.

Elmendorf tear strength tester was done according to ASTM D5734-95. Average of five measurements from each specimen was taken. And also SEM images of microstructure were analysis using Leo 1525.

3. RESULTS AND DISCUSSION

3.1. Basis weight

The basis weight of meltblown nonwoven varies by adjusting process variables. Basis weights of TPU nonwovens are presented in Fig. 3.

Basis weight of nonwovens produced decreases when both DCD and air pressure increases. The effect of DCD can be explained on the basis of drag forces near the collector

during fiber laydown. Drag forces are large at two basic locations of the meltblowing process near the die. Air speed is very fast and fiber speed is very slow so aerodynamic drag is large. Near the collector, air speed is still quite fast and fiber speed decreases to zero during laydown so aerodynamic drag increases significantly during laydown. Since fiber speed is reduced to zero for all processing conditions, the magnitude of the drag force experienced by fibers during laydown is determined primarily the speed of air by the speed of air in the laydown region of the collector. Since the collector is located further from the air source at larger DCD's, the speed of air arriving at the collector and the drag force acting on fibers during laydown decrease. Bhat and Malkan (9) reported that the basis weight typically range from 20 -200 g/m². Bresee and Qureshi (10) reported that improvement in basis weight uniformity is observed as DCD is reduced. And small changes within the DCD significantly affects basis weight uniformity of nonwoven since entanglement concentrates fiber mass within fiber bundles rather than distributing fibers more randomly over a larger web area. When air pressure is increased basis weight decreases. This can be attributed to fiber diameter decreasing. Air quenching has a significant effect on basis weight. Application of air quenching on TPU meltblown nonwoven presents a higher basis weight values at all DCDs and air pressures. Basis weight uniformity is a complex web structural feature that depends on single fiber diameter, fiber entanglement and the air quenching affect fiber motion. Air quenching provides extra cold air to cool fibers down quickly. Thus, getting fiber finer is not achieved resulting in a fiber with higher diameter and weight.

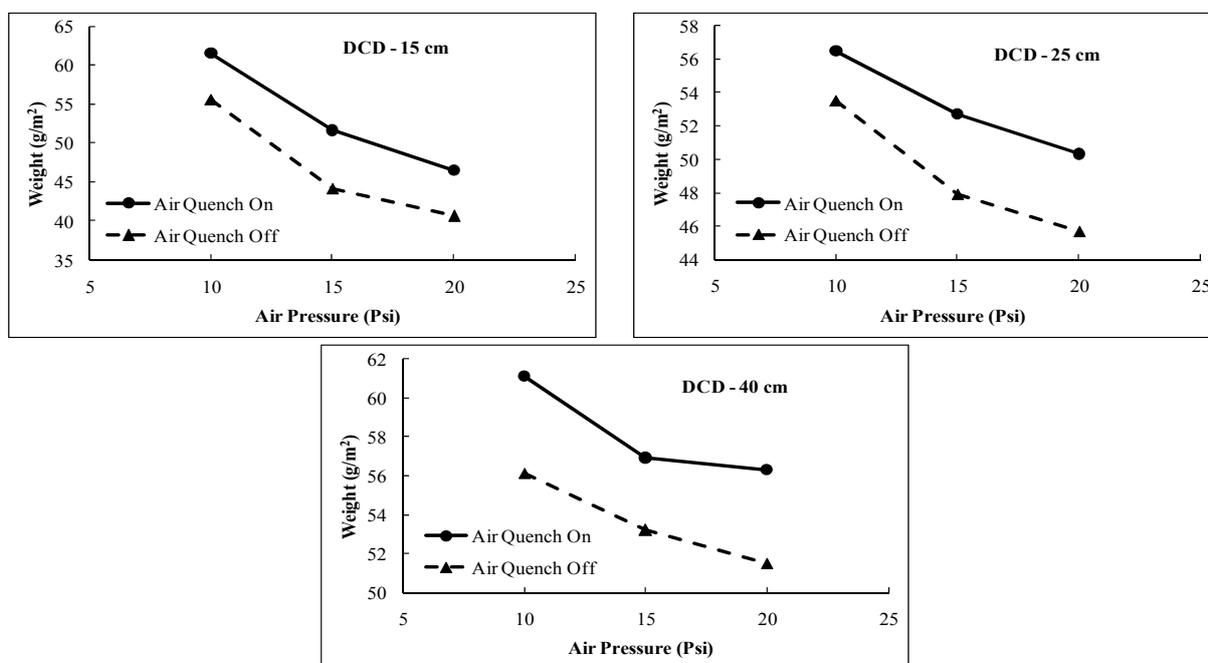


Fig. 3. Basis weight variations of TPU nonwovens.

3.2. Air permeability

Air permeability variations depending on the DCD, air pressure and air quenching is presented in Fig. 4. Air permeability increases when DCD is increased. The relationship between air permeability and DCD at different air pressures is shown in Fig. 5. As seen Fig. 5, the relation is at a high degree ($R^2=0.93$). It is well known that when the DCD is increased the fiber thickness increases resulting in a higher pore size. A web structure with high pore size has higher air permeability. But, air permeability decreases when air pressure is increased. Lower air permeability is obtained at higher air pressures. The reason behind it can be that when air pressure is increased the velocity of air will be higher resulting in increasing of cooling rate of fiber. The increased cooling rate causes a quicker solidification of fiber, and no more reduction of fiber diameter. At higher air

pressures, more uniform web structure is obtained. Lower air permeability means higher filtration efficiency due to capturing particles in the air more effectively. High air permeability is equal to a good barrier property. Lee and Wadsworth (11) reported that an increase in the DCD increases air permeability.

Air quenching gives slightly higher air permeability values. This can be attributed to the extra cooling effect resulting in increasing solidification of fibers. Air pressure and velocity, DCD and quenching have major influence on how much solidification and crystallization occurs before the fiber comes into contact with other fibers already on the collector belt. Amount of crystallinity depends on polymer type and time allowed for the sample to cool. Air quenching allowed the sample in a shorter time resulting in lower crystallinity degree.

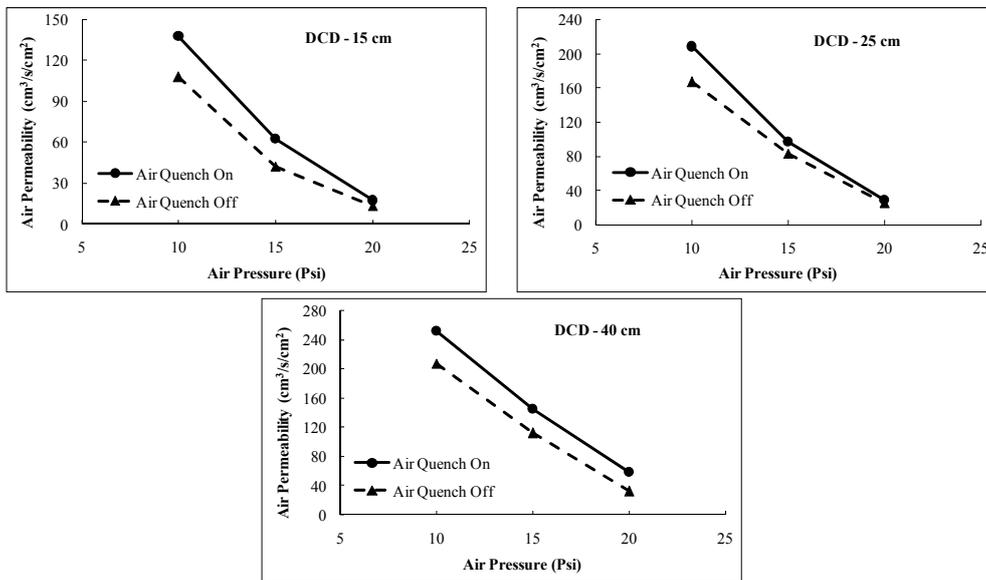


Fig. 4. Air permeability variations of TPU nonwovens.

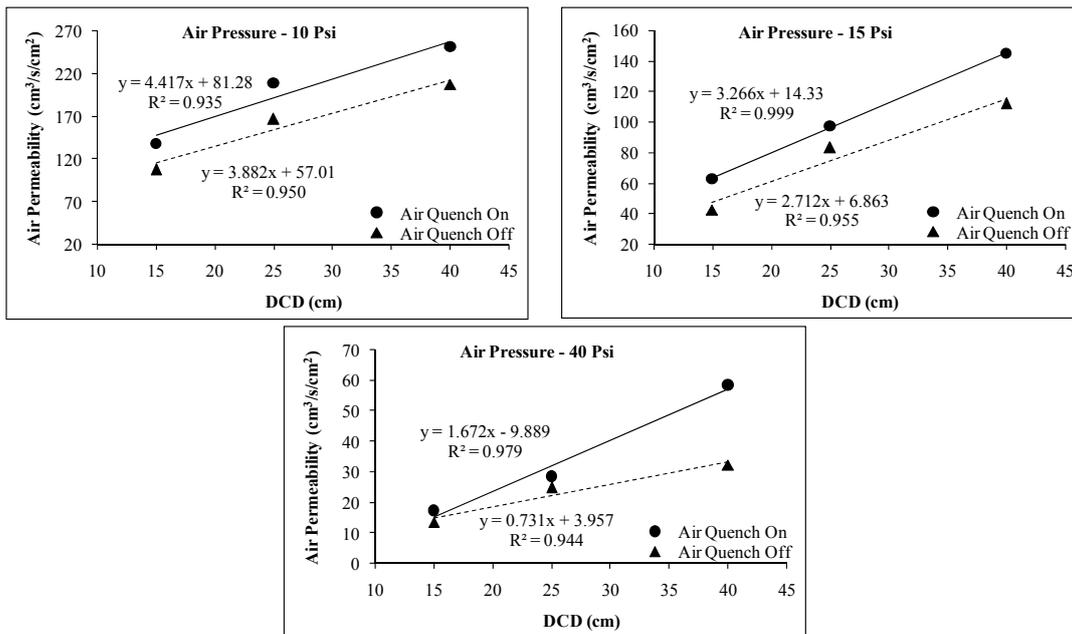


Fig. 5. Relationship between air permeability and DCD at different air pressures.

3.3. Hydrostatic Head

Hydrostatic head is a measure of liquid resistance of a fabric before three drops of water appear on the surface of the nonwoven. A fabric with higher hydrostatic head reading indicates it has a greater barrier to liquid penetration.

Hydrostatic head variations depending on the DCD, air pressure and air quenching is presented in Fig. 6. As seen from Fig. 6, hydrostatic head increases at both air pressure and DCD is increased. This means that resistance to

leakage increases. The relationship between DCD and hydrostatic head at all air pressures studied is shown in Fig. 7. A high correlation between them is seen.

When air permeability and hydrostatic head are considered together, it is obviously seen that air permeability decreases while hydrostatic head increases. The relation between hydrostatic head and air pressure is given in Fig. 8. As seen from Fig. 8, the coefficient of determination showing this relation is over $R^2=0.85$. This is highly rational that lower air permeability means a lower pore in the web structure.

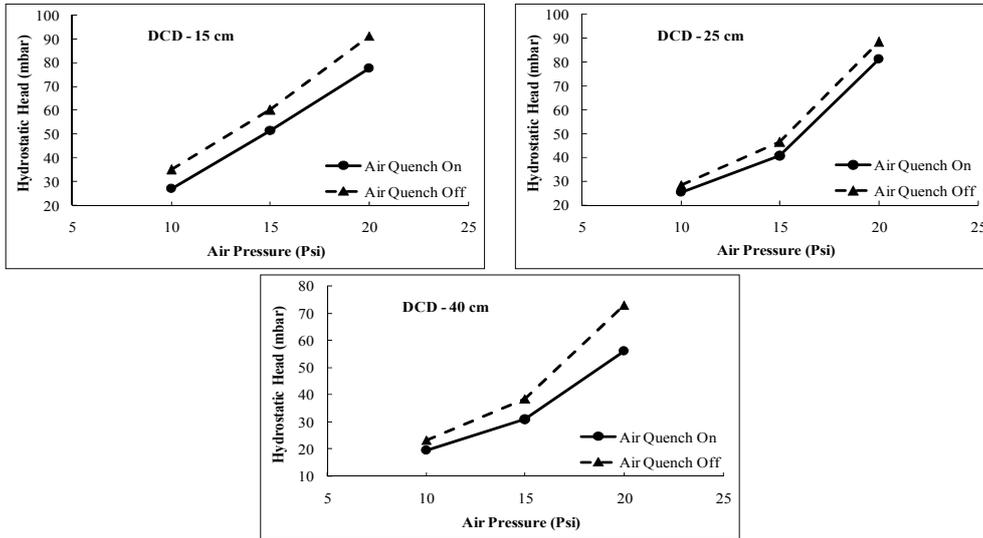


Fig. 6. Relationship between hydrostatic head and DCD at different air pressures.

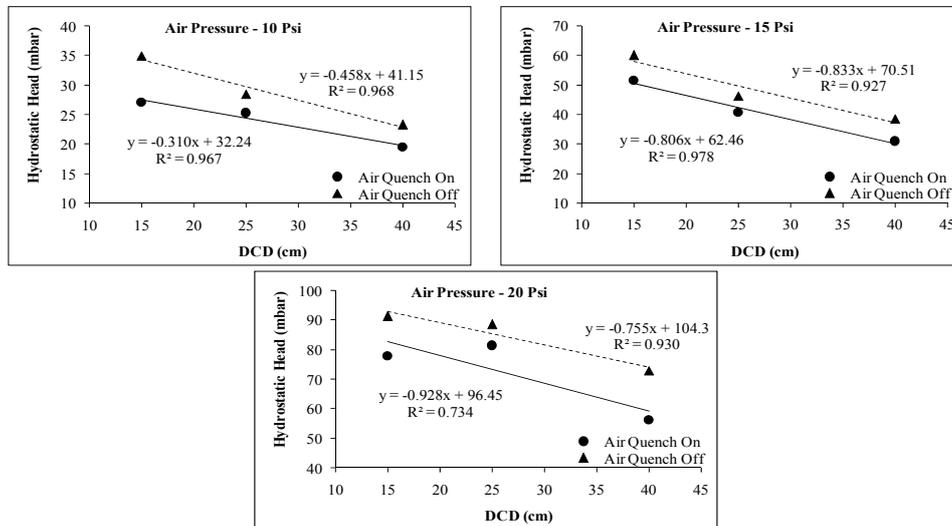


Fig. 7. Relationship between hydrostatic head and DCD at different air pressures.

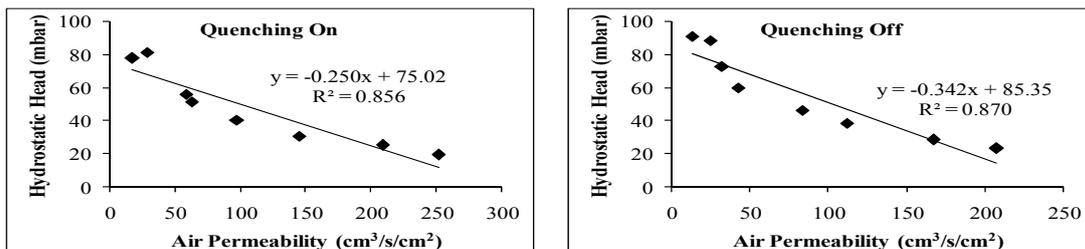


Fig. 8. The relation between hydrostatic head and air permeability.

Through a web with lower pore, a higher hydrostatic head is required. This means that meltblown TPU nonwoven produced has a good leakage resistance. Hydrostatic head decreases when DCD is increase at constant air pressure. The highest hydrostatic head was obtained at 15 cm DCD. Contrary to the effect of DCD, air pressure is increased up to 20 Psi highest hydrostatic head was obtained. Further studies should be done to see the different DCD and air pressure on hydrostatic head of meltblown TPU nonwoven. Air quenching has an effect on hydrostatic head of meltblown TPU nonwoven studied. Air quenching increases hydrostatic head when compared to those of samples which were not subjected to air quenching. This can be attributed to the nonwoven with a higher basis weight obtained by air quenching.

3.4. Tear strength

Tear strength of the produced nonwoven in machine and cross direction is given in Fig. 9 and Fig. 10. Concentration and orientation of fibers in the stress area affects the tear strength. As seen from Fig. 9, when DCD is increased tear strength increases. Tear strength is 2.6 N at quenching on and 2.1 N at quenching off when the air pressure is 10 Psi and DCD is 15 cm. Tear strength is 6.1 N at quenching on and 5.9 N at quenching off when the air pressure is 10 Psi and DCD is 25 cm. Tear strength is 11 N at quenching on and 9.3 N at quenching off when the air pressure is 10 Psi and DCD is 40 cm. The same behavior is observed when air pressure is 15 Psi, but at a lower values of tear strength. When the air pressure is increased to 20 Psi, tear strength of nonwoven produced is highly reduced. As clearly understood here, DCD and air pressure have a significant effect on tear strength. The reason behind the increment of tear strength depending on the increment of DCD is that

finer fibers may be obtained, and higher finer fiber concentrations in the stress area results in the increment of tear strength.

Air pressure applied has a decreasing effect on tear strength. The increment in air pressure may negatively affect the bond strength of fiber to fiber. Air quenching also has an effect on tear strength of meltblown TPU nonwoven studied. Nonwoven presents very slightly higher value. This can be attributed to that air quenching reduces the fly formation and polymer degradation. The same behavior is observed in cross direction (Fig. 10). When tear strengths at machine direction and cross direction is compared, it is observed that there is no significant change. Their strengths are almost in the same range. So, the changes can be negligible. This shows that a uniform web structure was obtained in both directions.

As seen from Fig. 11, when the basis weight of nonwoven produced increases tear strength in both directions increases. This can be attributed to that fiber concentration increases when basis weight increases. The coefficient of determination showing this relation is over $R^2=0.82$.

3.5. Scanning Electron Microscope (SEM) and Differential Scanning Calorimetry (DSC) analysis

In Fig. 12, SEM views of nonwovens taken at constant air pressure, 15 Psi, and different DCDs are presented. As seen clearly from Fig. 12, the DCD is increased from 15 cm to 25 cm fiber diameter increases. But, when the DCD is increased from 25 cm to 40 cm the change of fiber diameter is not significant. Application of air quenching results in the increment fiber diameter when compared to fiber diameter which is not subjected to air quenching.

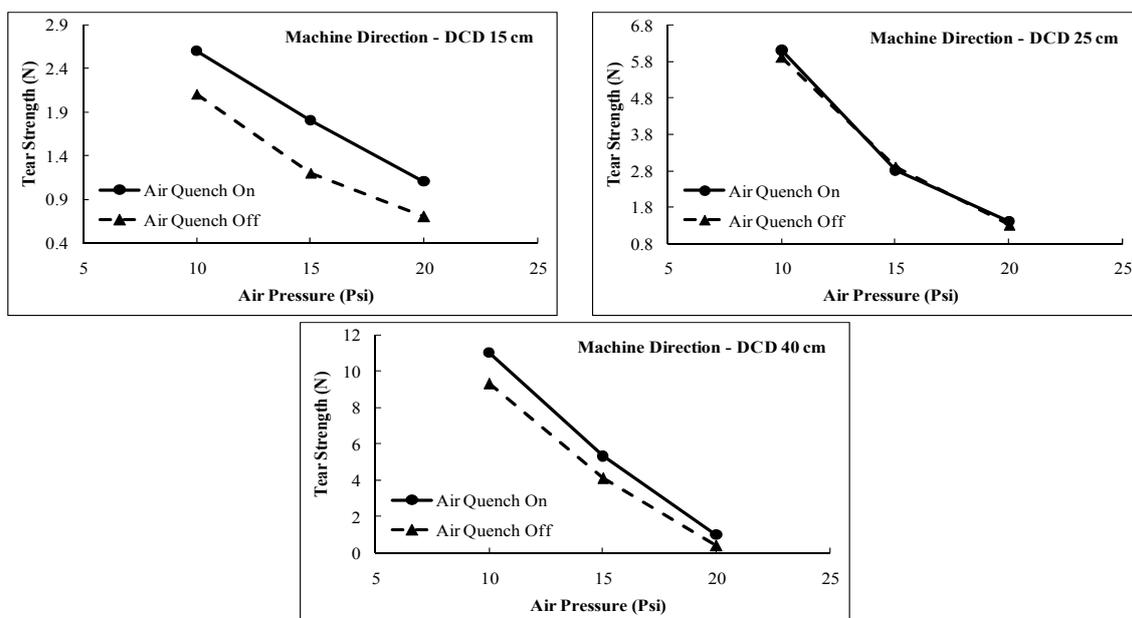


Fig. 9. Relationship between tear strength at machine direction and DCD at different air pressures.

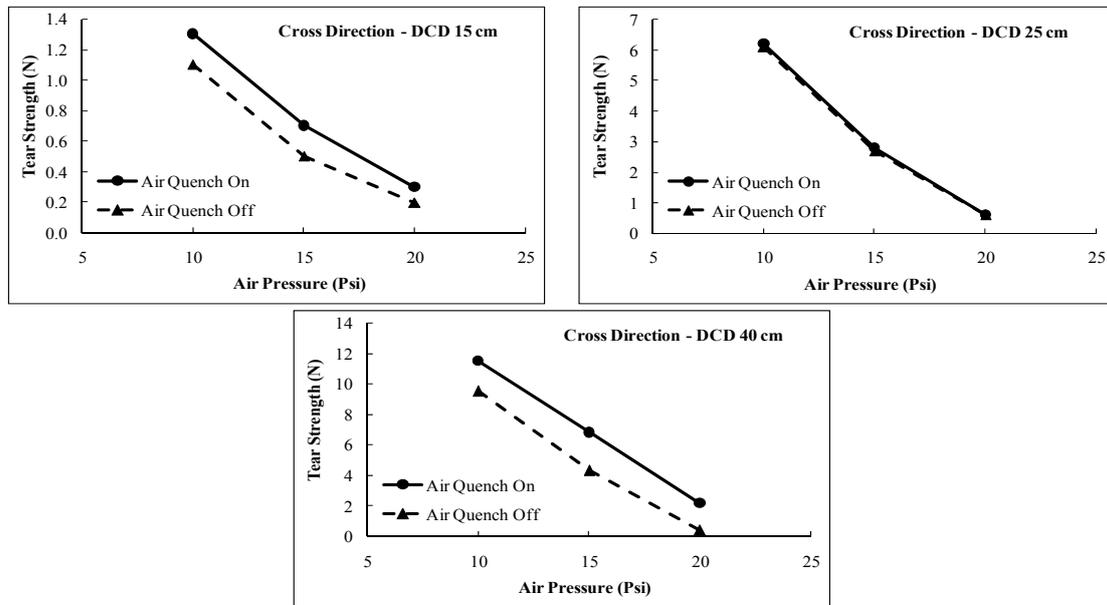


Fig. 10. Relationship between tear strength at cross direction and DCD at different air pressures.

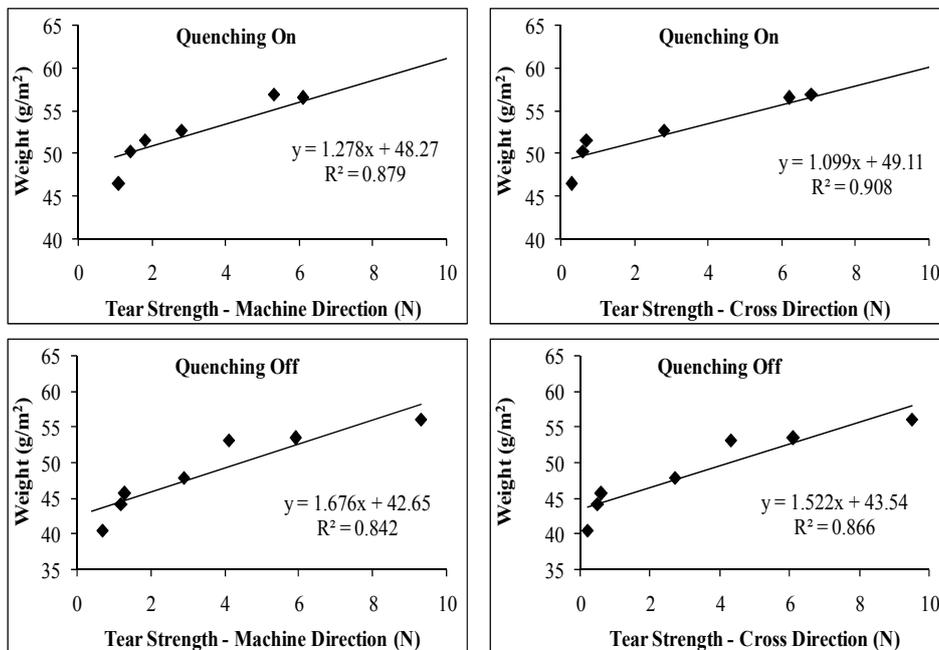


Fig. 11. The relation between tear strengths and basis weight.

The effect of air quenching presents an extra cold air to cool fiber left the die down until the fiber reaches to the collector or belt. After the melt polymer leaves from the die, the fibers occur. Fibers cool down fast resulting in quick fiber formation and preventing the fiber from elongation.

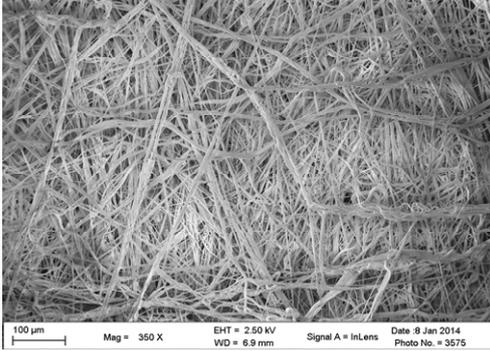
It is also seen that a uniform web structure of TPU meltblown nonwoven was achieved. Air quenching increases fiber diameter at constant DCD and different air pressures. A good fiber orientation and distribution were achieved in this study as seen from Fig. 12.

DSC is widely used to investigate the morphological behavior of phase separated block or segmented copolymers. Thermoplastic polyurethanes usually display distinct transitions as T_g and T_m for the hard and soft

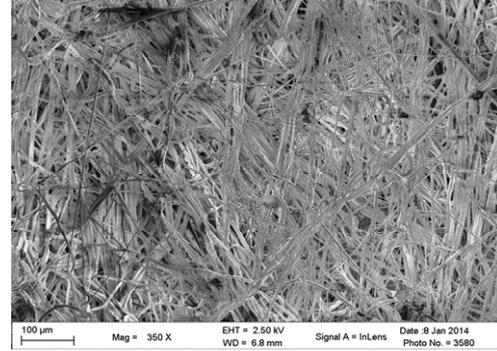
segments. Because of their chemical structure and segmented lengths, they show a T_g at above room temperature and a T_m at temperatures above 150 °C (12). We have investigated two type TPU samples which are normal TPU (n-TPU) sample and exposed to the cold air sample (c-TPU). Fig. 13 shows the DSC curves for n-TPU and c-TPU. While n-TPU shows two distinct melting endothermic (T_m) peaks at 207 °C and 225 °C, c-TPU shows three T_m peaks at 207 °C, 225 °C and 240 °C. c-TPU shows the second peak of intensity that was more pronounced and also third peak compared to n-TPU. This third endotherm peak is due to the melting of microcrystalline hard segment domains. c-TPU may have been properly rearranged its structure because of applying cold air.

Die-to-distance (DCD) 15cm

Quenching Air OFF

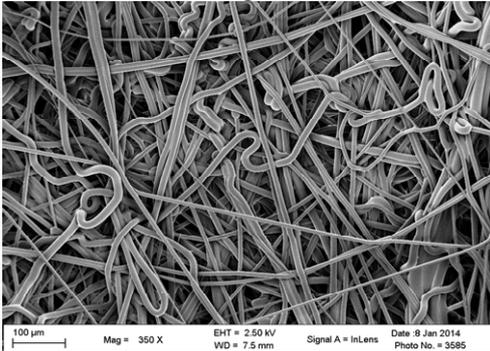


Quenching Air ON

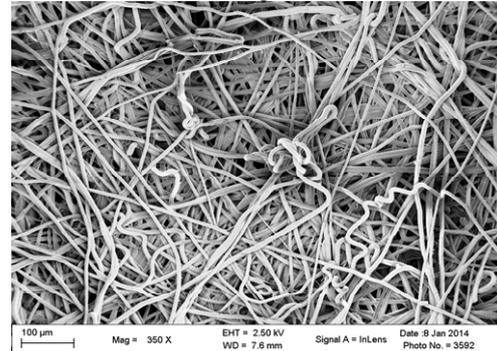


Die-to-distance (DCD) 25cm

Quenching Air OFF

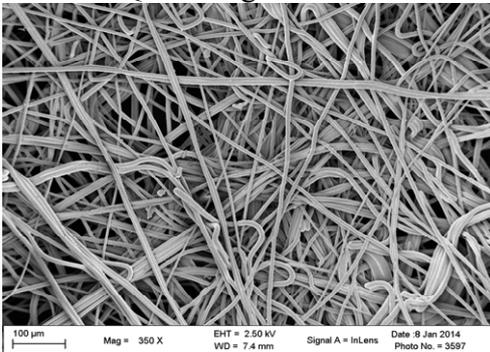


Quenching Air ON



Die-to-distance (DCD) 40cm

Quenching Air OFF



Quenching Air ON

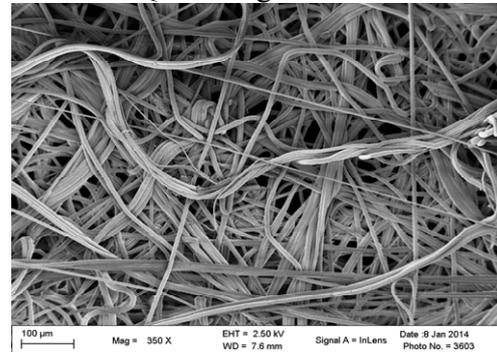


Fig. 12. SEM views of the polyurethane meltblown nonwoven produced at 15 Psi die air pressure and at different DCD (350x magnification).

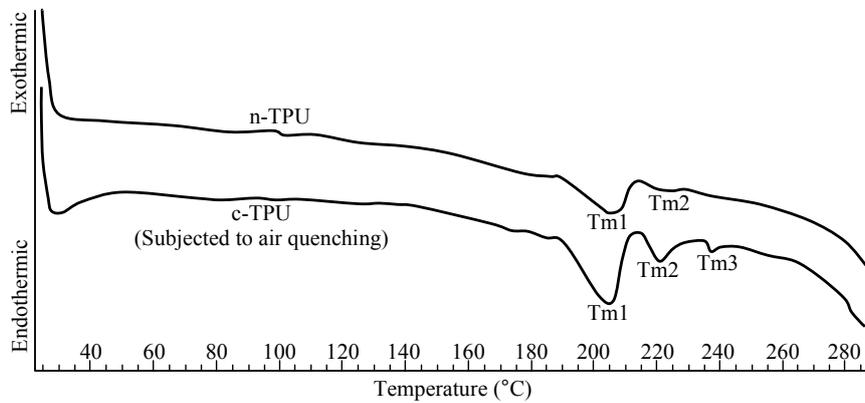


Fig. 13. Morphological behavior of TPU.

4. CONCLUSIONS

Nonwoven samples with thermoplastic polyurethane were produced by using meltblowing. Effects of process variables such as DCD and air pressure and air quenching were investigated on the web properties of nonwoven produced. It was observed that process variables and quenching have a significant effect on the web structure of TPU nonwoven. The key findings are presented as below;

The increments in both air pressures applied and DCD result in a reduction trend of basis weight of TPU nonwoven. The effect of air quenching dominates the basis weight at all DCDs and air pressures. Air quenching presents a nonwoven with a higher basis weight. Basis weights of nonwovens subjected to air quenching vary between 46.6 and 61.5 g/m² while those which were not subjected to air quenching vary 40.6 and 56.1 g/m². Air quenching providing cold air allows fibers to get a thick structure due to quick solidification of melt TPU.

Air permeability of TPU nonwoven increases at DCD increasing while it decreases at air pressure increasing. The increment in DCD produces fibers with a higher thickness resulting in a high pore size. The bigger pore size increases air permeability of nonwoven. The relation between air permeability and DCD is at high correlation, about R²=0.93 and higher. Lower air permeability is produced with the decreasing air pressure applied. Higher air pressure increases the cooling rate of fiber, consequently lower pore size. Air quenching increased air permeability of nonwoven due to its extra cooling effect. Air permeability of nonwovens subjected to air quenching vary between 17.3 and 251.3 cm³/s/cm² while those which were not subjected to air quenching vary 13.4 and 207.2 g/m². The increment in both air pressure and DCD increased hydrostatic head of

nonwoven. There is a close relationship between air permeability and hydrostatic head of nonwoven. Higher air permeability means a lower hydrostatic head. The correlation between this relation is R²= 0.85. For filtration application, lower air permeability and higher hydrostatic head are desired. Mechanical strength of TPU nonwoven is not affected significantly from the directions, machine and cross direction. This is a result of a uniform fiber orientation in the web structure. Tear strength increases with the increment in DCD while tear strength decreases with the increment in air pressure applied. Air pressure may decrease the bond of fiber to fiber. Application of air quenching reduces fly formation. Tear strength of nonwoven subjected to air quenching vary between 1 and 11 N at machine direction while between 0.3 N and 11.5 N at cross direction. Tear strength of nonwoven not subjected to air quenching vary between 0.4 and 9.3 N at machine direction while between 0.2 N and 9.5 N at cross direction. Also basis weight is an important parameter on the tear strength of nonwoven. The increment in weight results in the increment of tear strength at both directions. SEM views show that a uniform fiber distribution and orientation were obtained in the production of meltblown TPU nonwovens. High orientation formed during the production with assist of cold air cooling may play an important role in fiber crystallization.

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