

Development of Superhydrophobic Polyester Fabrics Using Alkaline Hydrolysis and Coating with Fluorinated Polymers

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(Received September 16, 2015; Revised December 14, 2015; Accepted January 17, 2016)

Abstract: Alkaline hydrolysis is one of the most classic fiber finishing methods, however, its potential as tuning surface superhydrophobicity in mass scale has not been studied much. In this research, fine roughness was formed on the polyester fiber surfaces by alkaline hydrolysis at room temperature and fluorinated polymer mixtures were further coated. The developed superhydrophobic fabrics were evaluated in terms of structural changes, mechanical properties, surface hydrophobicity, and permeability for practical applications. As alkaline hydrolysis treatment time increased, surface roughness was increased as a lot of nano-craters were generated with the decrease of fabrics weight and tensile strength as well. As air pockets formed through nano-craters on the fiber surfaces, static contact angle increased, and shedding angle tended to decrease. In this study, the sample treated with alkaline hydrolysis for 20 minutes showed the highest static contact angle of $167.8 \pm 1.3^\circ$ and lowest shedding angle of $4.4 \pm 2.3^\circ$. Considering tensile strength loss, however, the 15-minute alkaline hydrolyzed fabrics which showed static contact angle of $162.2 \pm 2.7^\circ$ and shedding angle of $8.8 \pm 0.2^\circ$ was selected as the optimal condition for practical application. The newly developed superhydrophobic fabrics were found to have higher water vapor and air permeability than those of untreated samples. At the same time, fluoropolymer coating played a certain role for tensile strength and water vapor permeability demonstrating the importance of understanding and designing proper fluorinated-compound treatment processes.

Keywords: Superhydrophobic fabrics, Alkaline hydrolysis, Surface roughness, Polyester, Fluorinated polymer coating

Introduction

Superhydrophobic fabrics has been an area of intense interests for decades in both academia and industry thanks to their wide range of uses including electronic device, filter, and clothing due to its capabilities of preventing corrosion, water-repellency, and self-cleaning [1-3]. Superhydrophobicity is defined as the condition having a static contact angle of 150° or higher and a shedding angle of less than 10° , and is prepared by adequate control of surface roughness and surface energy [1,4]. A number of methods have been suggested to engineer superhydrophobic surfaces including electrospinning [5,6], plasma treatment [7,8], sol-gel process [5,9], etc.

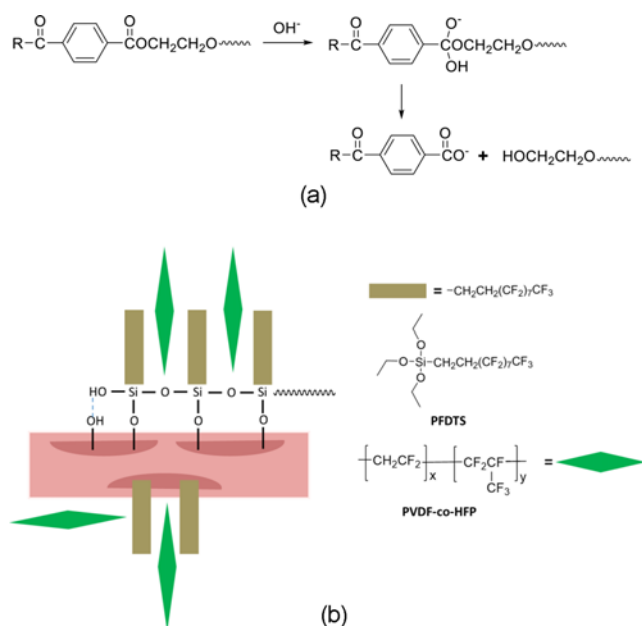
Nanoparticles synthesized through sol-gel process have been widely applied on fabric surfaces to impart fine surface roughness [9-12], but the concern is low affinity between fabric surfaces and nanoparticles which may lead to loss of functions [5,13] and potential health risk [14]. Although plasma induced etching and polymerization is an alternative method for nanoparticle incorporation, plasma treatment requires special equipment [7,8,15]. Thus, these two very popular methods in superhydrophobic surface design has limitations in mass production and surface coating durability.

On the other hand, alkaline hydrolysis, a treatment to give physical and chemical changes through hydrolysis of

polyester (polyethylene terephthalate; PET) fabrics under alkaline condition, is a simple method with versatile applications in fabric industry in which surface treatment in large scale can be conducted [16-18]. The mechanism of alkaline hydrolysis have been extensively studied to find that strong base causes cleavage of ester linkages on the fiber surfaces leading to the formation of nano-craters with extra functional groups such as hydroxyl and carboxylase groups [17,19]. Alkaline hydrolysed polyesters successfully possessed enhanced wettability [16], dyeability, silk-like lustre and handle [18]. Despite these advantages, studies on the development of superhydrophobic fabrics via alkaline hydrolysis are currently lacking.

Mazrouei-Sebdani and Khoddami [13] probably first noticed the applicability of alkaline hydrolysis for superhydrophobic fabric manufacturing. They extensively studied the surface treatment recipe including the combination of alkaline hydrolysis/nanoparticle/fluorochemical to successfully develop durable superhydrophobic fabrics. A specific commercial water repellent agent, Rucostar EEE[®] which consists of dendrimers and fluoropolymer chains, was used in the research. Later several studies [12,20] utilized alkaline hydrolysis to develop superhydrophobic fabrics but did not pay much attention to critical fabric properties such as tensile properties, water vapor and air permeability. Some researchers also demonstrated that alkaline hydrolysis is a promising surface etching method not only for polyester fabrics but also for other materials such as poly(butylene

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Scheme 1. Proposed mechanisms of superhydrophobic surface treatments; (a) alkaline hydrolysis of PET which causes nano-craters and hydrophilic groups on the fiber surfaces [18,19] and (b) fluoropolymer mixture coating on alkaline hydrolyzed PET fiber surfaces; PFDTs molecules can be reacted to form covalent bonds or hydrogen bonds with -OH groups on the alkaline hydrolyzed PET fiber surfaces. PFDTs is known to crosslink with each other as well (top); it is also possible for PFDTs molecules diffused into non-crystalline regions inside of PET fibers (bottom); PVDF-co-HFP molecules were expected to be attracted closely to alkyl-fluoro groups of PFDTs as well as fiber surfaces.

terephthalate) melt blown fibers [21] and poly(vinylidene fluoride) films [22].

Based upon literature review, we found that there still is a research gap in applying alkaline hydrolysis as a simple mass production method for the development of superhydrophobic polyester fabrics. In this study, nano-scale roughness was imparted on the drawn textured yarn (DTY) polyester fabrics which have inherent micro-level roughness through alkaline hydrolysis. In our previous research [23], the combination of textured fibers and water repelling agents was shown to be an effective strategy in enhancing surface hydrophobicity. Perfluorodecyltriethoxysilane (PFDTs) was chosen as water repelling agent because of its strong anchoring

properties with functional groups on alkaline hydrolyzed PET surfaces and non-crystalline region of PET fibers [20]. Poly(vinylidene fluoride-co-hexafluoropropylene) (PVDF-co-HFP) was applied together with PFDTs expecting to have high intermolecular interactions with PFDTs molecules bound to PET fiber surfaces to prepare more stable fluoropolymer coatings (Scheme 1(b)). Alkaline hydrolyzed and fluoropolymer mixture coated PET fabrics were evaluated in terms of physical properties, surface hydrophobicity, and permeability to understand its applicability as practical fabrics.

Experimental

Materials

100 % PET fabrics purchased from Dong Jin Textile Co. Ltd. (Korea) were used as samples. Fabric characteristics are described in Table 1. For the scouring process, 60 % sodium dodecylbenzenesulfonate (Daejung chemicals & metals Co. LTD), sodium carbonate (Junsei chemical Co. Ltd.), and Triton X-100 (Laboratory grade, Sigma Aldrich) were used. Sodium hydroxide purchased from Junsei chemical Co. Ltd. was used in alkaline hydrolysis process. For hydrophobization, N,N-dimethylformamide (DMF, extra pure, Daejung chemicals & metals Co. Ltd.), poly(vinylidene fluoride-co-hexafluoropropylene) (PVDF-co-HFP), and 97 % 1H, 1H, 2H, 2H-perfluorodecyltriethoxysilane (PFDTs) (Sigma Aldrich) were used.

Superhydrophobic Surface Rendering Processes

PET fabrics cut into 2.5 cm×5 cm were scoured in solution containing 60 % sodium dodecylbenzenesulfonate (5 g/l) and sodium carbonate (5 g/l) at 90 °C for 1 h followed by rinsing and drying. Alkaline hydrolysis and fluoropolymer coating were conducted based on previous research [12,20] with some minor modifications. The fabrics were immersed in alkaline hydrolysis solution containing 38 % NaOH (aq.) for 5, 10, 15, or 20 min at 90 degree C. Specimens were rinsed with distilled water until reaching pH 7 and then air dried. 1 g PVDF-co-HFP and 0.5 ml PFDTs were added into 50 ml DMF stirred for 1 h at 110 °C to prepare fluoropolymer mixtures for fabric coating. The alkaline hydrolyzed specimens were dip-coated in the prepared fluoropolymer mixtures for 3 minutes at room temperature and further cured at 135 °C for 10 min. Figure 1 summarizes the entire experimental procedures. Sample codes were

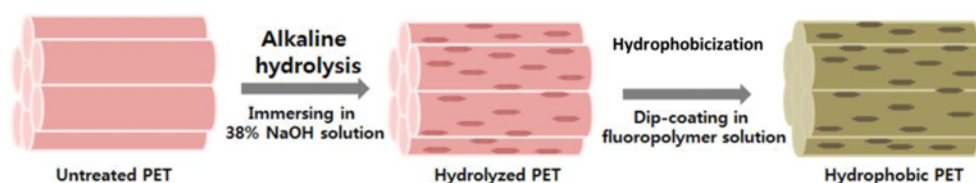


Figure 1. Scheme for experimental procedures.

Table 1. Characteristics of the specimens

Yarn count (in inch×inch)	75 denier yarn (warp) of 72 filaments+ 150 denier yarn (weft) of 144 filament DTY (drawn textured yarn)
Specific weight (g/m ²)	108.0
Thickness (mm)	0.21

named according to the alkaline hydrolysis conditions as described in Table 2.

Characterization

Physical Properties and Chemical Compositions

The surface morphology and diameter of fibers were observed using field-emission scanning electron microscope (FE-SEM, JSM-7600F, JEOL, Germany), and the chemical compositions of the sample surface before and after hydrophobization were compared through energy dispersive X-ray analysis, (EDX, Aztec, Oxford instruments, UK). Fabric thickness was measured with a thickness gauge (ID-C112, Mitutoyo Corp, Japan), and the average value was calculated from 3 measurements. The ASTM D 5035 strip method was used to measure tensile strength. Samples were cut into 2 cm×15 cm, and measured in the warp direction before and after alkaline hydrolysis. The average value from 5 measurements were presented.

Surface Hydrophobicity

Surface hydrophobicity was evaluated by static contact angle and shedding angle by contact angle goniometer (Theta lite optical tensiometer, KSC Instruments, Finland). An average static contact angle was calculated by measuring 5 different sites on each sample. Minimum shedding angle was measured at the tilting angle where 12.5 μ l or 35 μ l water droplets were slid for at least 2 cm.

Water Vapor and Air Permeability

Untreated, F, 15E, and 15E+F specimens were conditioned in laboratory standard condition for 24 h before measuring water vapor transmission rates. Environment temperature and humidity were set as 40±2 °C and 50±5RH% according to ASTM E96-80. Air permeability was measured by permeating air pressure of 125 Pa through sample area of 38.3 cm² according to ASTM D 737 Frazier method in the vertical direction.

Results and Discussion

Physical Properties and Chemical Compositions

Surface morphology changes of PET fabrics by alkaline hydrolysis are shown in Figures 2 and 3. It was found that as alkaline hydrolysis treatment time was increased, surface etching became deeper creating more nano-craters which lead to increased surface roughness. The formation of nano-craters by alkaline hydrolysis is caused by the attack of hydroxide ion to ester bonds of PET (Scheme 1(a)) [18,19]. Unlike untreated samples in which fibers are closely packed, fibers became gradually thinner as alkaline hydrolysis time increased, resulting in more spaces between fibers. Figure 3 shows SEM images of alkaline hydrolyzed PET fibers after hydrophobization with fluoropolymers showing that surface morphology was not changed greatly by the subsequent coating process.

So as to analyze the change in chemical composition of the fabrics surface after hydrophobization, EDX was employed. The results are given in Figures 4 and 5. For the untreated and alkaline hydrolyzed samples, only C and O which are the main ingredients of the PET fabrics appeared. In contrast, hydrophobicized samples showed F-peak, which indicates successful fluorinated polymer coating. Chemical

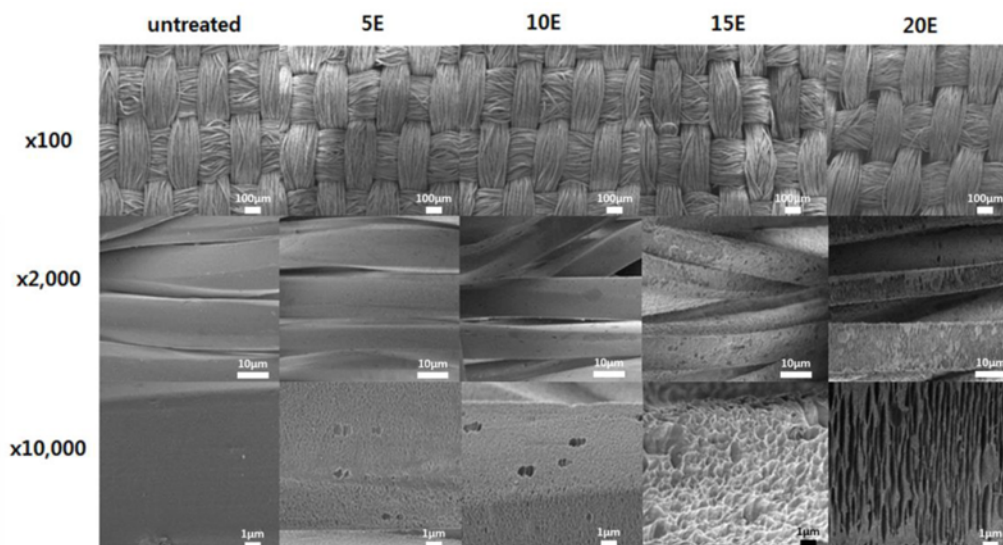


Figure 2. SEM images of alkaline hydrolyzed PET with different treatment time.

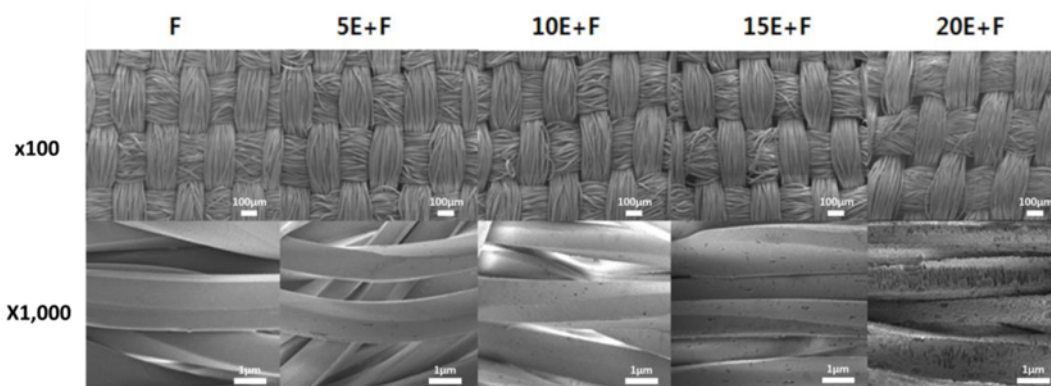


Figure 3. SEM images of alkaline hydrolyzed PET after hydrophobization treatment.

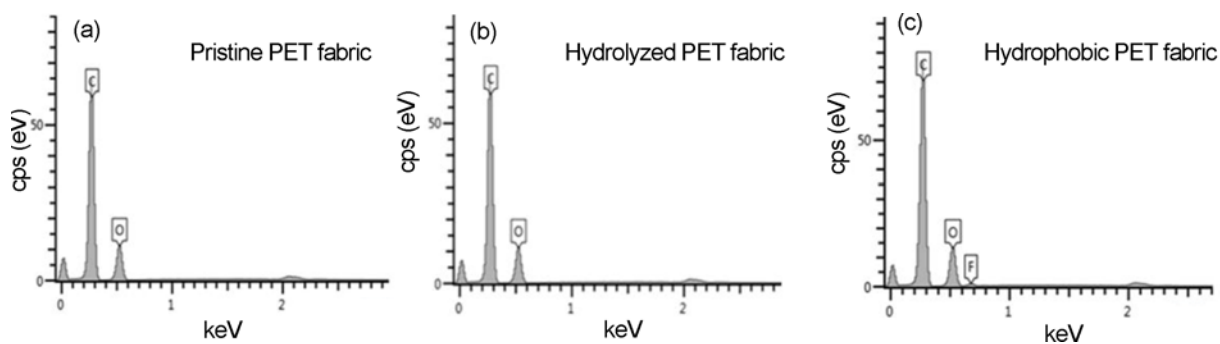


Figure 4. EDX analysis for the (a) untreated PET fabric, (b) 15 min hydrolyzed PET fabric before, and (c) after hydrophobization.

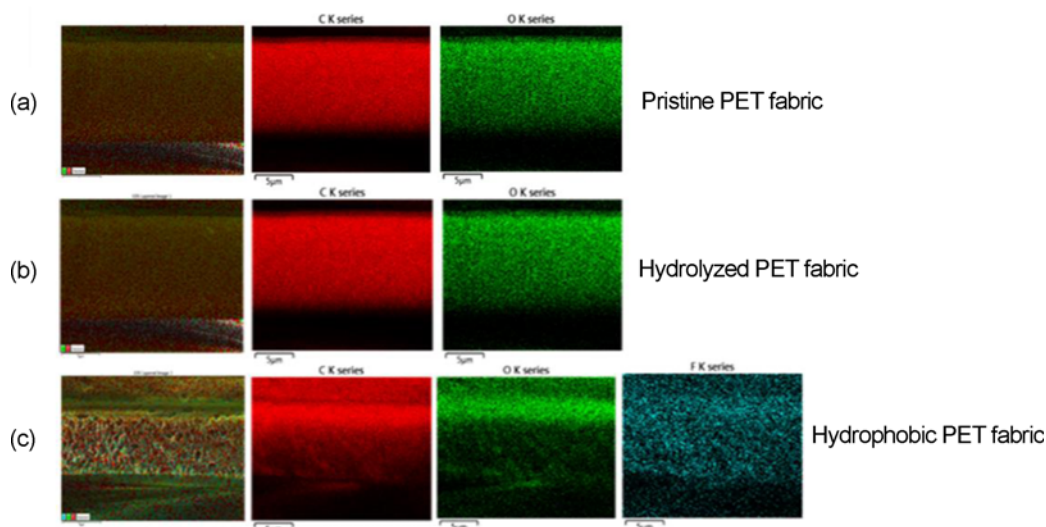


Figure 5. EDX elemental mapping for the (a) untreated PET fabric, (b) 15 min hydrolyzed PET fabric before, and (c) after hydrophobization.

mapping through EDX (Figure 5) showed uniform distribution of C and F atoms on the fiber surfaces but O atoms were somewhat covered. This indicates uniform and successful fluoropolymer coating through interaction with -OH groups on the PET surfaces.

Thickness, weight of fabrics and diameter of fiber were

altered as a function of alkaline hydrolysis time. When comparing 20E with untreated fabrics, fabric weights decreased by 66.7 %, thickness decreased by 32.8 %, and diameter decreased by 4 μm (Figures 6 and 7). The weight reduction as well as nano-craters formation on the PET surface is attributed to chain cleavages as OH^- attacks ester-

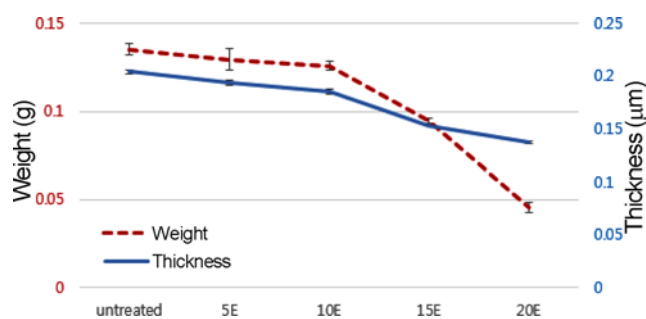


Figure 6. Weight and thickness changes for alkaline hydrolyzed PET.

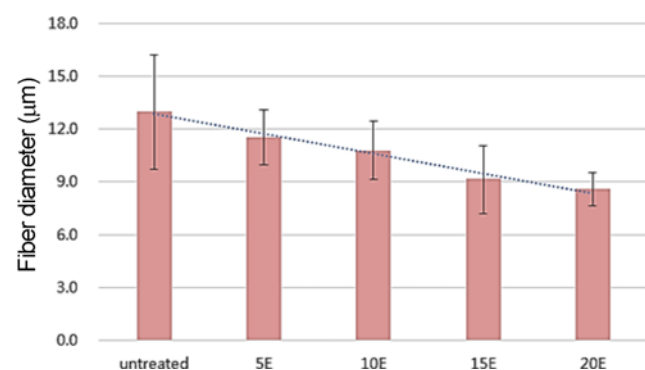


Figure 7. Fiber diameter for alkaline hydrolyzed PET under different treatment time.

Table 2. Specimens of various treatments

Code	Treatment
Untreated	Untreated PET
F	hydrophobization by coating with fluorinated polymers
5E	5 min alkaline hydrolysis
10E	10 min alkaline hydrolysis
15E	15 min alkaline hydrolysis
20E	20 min alkaline hydrolysis
5E+F	5 min alkaline hydrolysis+hydrophobization by coating with fluorinated polymers
10E+F	10 min alkaline hydrolysis+hydrophobization by coating with fluorinated polymers
15E+F	15 min alkaline hydrolysis+hydrophobization by coating with fluorinated polymers
20E+F	20 min alkaline hydrolysis+hydrophobization by coating with fluorinated polymers

bonded carbonyl carbon moieties [18,19]. As Figures 6 and 7 shows, fabric thickness and fiber diameter decreased linearly as alkaline hydrolysis time increased, but fabric weight decreased dramatically after certain treatment time (10 min). This is because hydrophilic groups are rapidly introduced onto the fiber surfaces but the etching reaction is relatively slow at the initial stage of alkaline hydrolysis. As

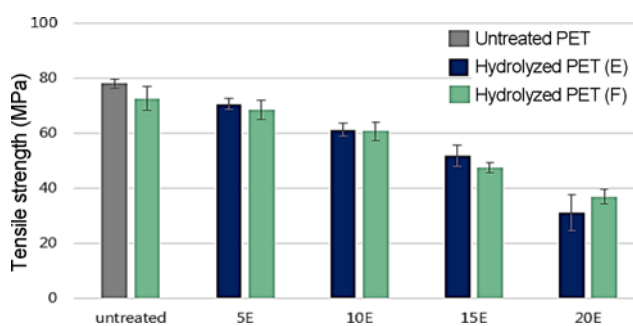


Figure 8. Tensile strength results of alkaline hydrolyzed PET before and after hydrophobization with different treatment time.

the reaction progresses, the surface area gets larger by the formation of nano-craters to be exposed to more hydroxide ions. It is thought that the weight loss rate is increased as more alkaline solution diffuses inside the fiber [19,24].

Similar to other physical properties, tensile strength was reduced as alkaline hydrolysis treatment time increased (Figure 8). Nano-craters formed on the fiber surfaces and the reduction of fiber diameter seemed to affect tensile strength since tensile strength is considerably affected by the weak points [25]. In this study, 20E samples which were exposed to alkaline solution for the longest time, showed the lowest tensile strength, a decrease of 60 % compared to untreated samples. Although hydrophobization did not affect tensile strength greatly for other samples, 20E+F had much higher tensile strength compared to 20E. This may be associated with trapping of PFDTs within alkaline hydrolyzed PET fibers as shown in Scheme 1(b) [20]. PFDTs molecules could be located in larger non-crystalline region of 20E+F samples than other samples to hold PET molecules more tightly and improve tensile strength. It should also be noted that PET has generally very good mechanical properties comparing to other synthetic or natural fibers [25]. Minor deterioration in mechanical properties after alkaline hydrolysis is expected to be acceptable for conventional fiber usage.

Surface Hydrophobicity

Static contact angles measured on hydrophobized samples following alkaline hydrolysis are shown in Figure 9. Untreated samples had static contact angles of $119.7 \pm 3.7^\circ$. The static contact angle of F was $131.3 \pm 0.3^\circ$, which was increased by about 10° compared to the untreated condition. On the other hand, when water is dropped on an alkaline hydrolyzed samples, the surface of the samples were completely wet. This is because the number of hydrophilic groups and surface roughness have increased after alkaline hydrolysis [26,27]. According to Wenzel's theory [28], increases in surface roughness of hydrophilic surface promotes surface wetting further. It was also found that the longer the alkaline hydrolysis treatment time, the more quickly water drops were absorbed onto the surface.

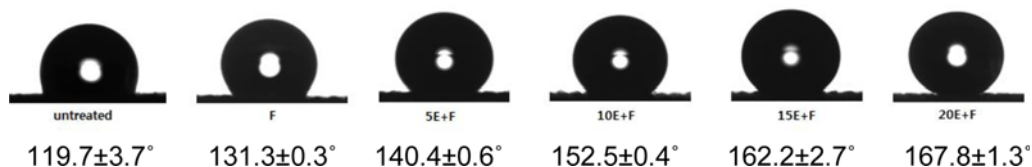


Figure 9. Images of contact angle for different hydrophobic PET fabrics.

Table 3. Shedding angle values for hydrophobic samples

Code	SHA-S (°)	SHA-L (°)
Untreated	> 90	> 90
F	> 90	> 90
5E+F	20.0±0.5	16.2±0.6
10E+F	13.0±1.0	12.8±0.3
15E+F	8.8±0.2	5.1±0.5
20E+F	4.4±2.3	2.3±1.0

Hydrophobicized samples, 5E+F, 10E+F, 15E+F, and 20E+F, showed static contact angles of $140.4\pm 0.6^\circ$, $152.5\pm 0.4^\circ$, $162.2\pm 2.7^\circ$, and $167.8\pm 1.3^\circ$ respectively; longer alkaline hydrolysis treatment time along with fluoropolymer coating was therefore associated with larger increases in static contact angles. This was because as hydrolysis treatment time got longer, more nano-craters were formed which increased surface roughness when surface energy of PET fibers are very low due to fluoropolymer coatings. This resulted from the reduction of contact area between PET sample surfaces and water droplets by the introduction of air pockets within nano-craters which are well understood from Cassie-Baxter's theory [29].

Shedding angles are given in Table 3. Shedding angles of the untreated and F sample were larger than 90° . Since the fiber surfaces of untreated and F sample were relatively smoother than alkaline hydrolyzed specimens, water droplets may adhere to the gaps between fibers promoted by wicking phenomenon [9]. On the other hand, 5E+F, 10E+F, 15E+F, and 20E+F samples on which nano-craters formed showed roll-off when tilted to a certain level because water droplet and sample surfaces created Cassie-Baxter state [4,5,9,29,30]. When larger droplets were placed on the specimens, lower shedding angles were measured which is reasonable because of gravity. Park *et al.* [9] noticed the discrepancy of shedding angle results as experimental condition changes and suggested the necessity of standardized characterization methods for superhydrophobic surfaces. In this study, 15E+F and 20E+F were found to be superhydrophobic surfaces but considering tensile properties, 15E+F is suggested as the optimal condition for practical applications.

Water Vapor and Air Permeability

Untreated and F and 15E and 15E+F which was chosen as the optimal condition for superhydrophobic surface rendering were evaluated for water vapor and air permeability. Water

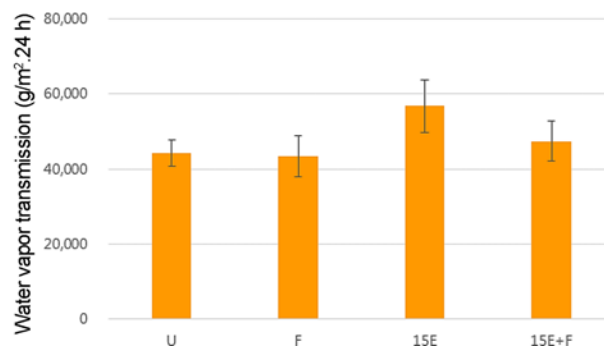


Figure 10. Water vapor transmission rates of specimens.

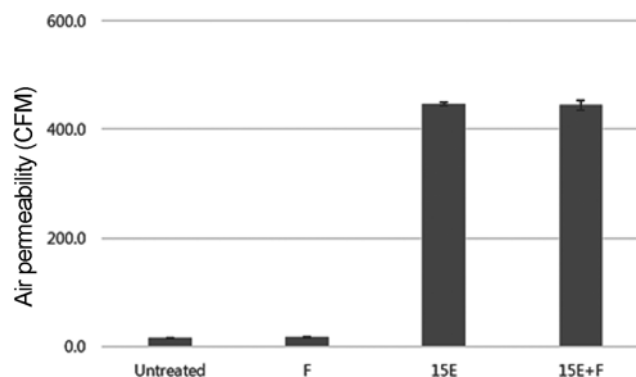


Figure 11. Air permeability results of specimens.

vapor permeability and air permeability results are shown in Figures 10 and 11, respectively. Alkaline hydrolyzed samples showed an increase in water vapor and air permeability as expected. Since fiber diameter and thickness diminished and hydrophilic groups were introduced after alkaline hydrolysis, it is natural for 15E and 15E+F samples to have higher water vapor and air permeability than U and F. Interestingly, no significant changes in water vapor and air permeability were noticed between U and F suggesting porosity should remain intact after hydrophobization. On the other hand, 15E+F samples had similar air permeability with that of 15E but had much smaller water vapor transmission than 15E did. This result may be stem from the trapping of PFDTS in non-crystalline region of alkaline hydrolyzed PET fibers since PFDTS molecules take up non-crystalline region in which water vapor molecules can be dissolved, and hydrophilic surface groups to which water molecules can be attached. It is well known that both air between fibers and water dissolution inside of fiber are crucial factor for water vapor

permeability [31,32]. Our results are in line with previous research [5,33] in that air permeability is mostly related to fabric porosity but water vapor permeability is influenced by porosity as well as fiber chemistry.

Conclusion

In this study, superhydrophobic fabrics were developed via conventional alkaline hydrolysis and subsequent surface coating with fluoropolymers. Physical changes, surface hydrophobicity, and permeability of the novel fabrics were evaluated. As alkaline hydrolysis treatment time increased, more nano-craters were formed on the fiber surface, increasing surface roughness and surface hydrophobicity measured by static contact angles and shedding angles. Physical properties such as fabrics weight, thickness, fiber diameter, and tensile strength were reduced upon alkaline hydrolysis treatment time increase. The 15-minute alkaline hydrolysis and fluoropolymer coating (15E+F) resulting in a contact angle of $162.2 \pm 2.7^\circ$ and a shedding angle of $8.8 \pm 0.2^\circ$, and tensile strength comparable to that of cotton fiber was considered as optimum condition. For 15E+F samples both water vapor and air permeability were increased compared to those of unhydrolyzed samples indicating good potential of developed fabrics for conventional use. Interestingly, fluoropolymer coating appeared to have a certain effects on tensile strength and water vapor transmission rates. Proper utilization of fluoropolymer coating mechanism is thought to be crucial for achieving the desired textile properties. This study has significance in the development of superhydrophobic fabrics for mass production in a relatively simple way, particularly using alkaline hydrolysis which is widely used for polyester fabrics with better handle.

Acknowledgment

This work was supported by the National Research Foundation of Korea (NRF) grant funded by the Korea government (MSIP) (No.2015R1A2A2A03002760) and BK21 Plus of the National Research Foundation of Korea (NRF) Grant funded by the Korean Government (2011-0014765).

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