

## Facile fabrication of polyester filament fabric with highly and durable hydrophilic surface by microwave-assisted glycolysis

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**ABSTRACT:** Poly(ethylene terephthalate) (PET) fabric with highly and durable hydrophilic surface was fabricated using microwave-assisted glycolysis. Sodium hydroxide (NaOH) as a catalyst was proven to be suitable for PET glycolysis under assistance of microwave. The modified PET fabric (0.5% NaOH, irradiation 120 s) showed high surface hydrophilicity with a contact angle of 17.4° and a wicking length of 19.36 mm. The exposure of the carboxyl- and hydroxyl-end groups on the surface of PET and the introduction of etches were confirmed by Methylene Blue staining and field emission scanning electron microscopy (FESEM), respectively. Although the strength of PET fabric decreased after modification, it was still high enough for textile applications. The thermal properties of the modified PET fabrics were well maintained. The high hydrophilicity and its original properties of PET could be controlled by changing the irradiation time from 60 s to 120 s and adjusting the content of sodium hydroxide from 0.2% to 0.5%. These results suggest microwave-assisted glycolysis with sodium hydroxide is an effective method for PET hydrophilic finishing. © 2016 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2016**, *133*, 44069.

**KEYWORDS:** fibers; hydrophilic polymers; polyesters; structure-property relations; textiles

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### INTRODUCTION

The consumption of poly(ethylene terephthalate) (PET) fiber is greater than any other man-made fiber in textile industry. PET as an important garment material is widely used in clothing because of its uniquely promising properties, such as good mechanical properties, excellent wrinkle resistance and recovery, high thermal stability, and wash-and-wear performance.<sup>1,2</sup> However, the poor hydrophilicity of PET fabric results in low wear comfort, such as sultriness, electrostatic charge, and easily contaminated.<sup>3,4</sup> The poor hydrophilicity becomes the bottleneck and limits the applications in garment industry because of its compact structure and shortage of strong hydrophilic groups. Towards this direction, growing attention has been paid to enhance the hydrophilicity of PET fabrics. Techniques currently used for the hydrophilic finishing mainly include surface oxidation, hydrolysis using strong acid or alkali,<sup>5–7</sup> aminolysis,<sup>8,9</sup> alcoholysis,<sup>10</sup> enzymatic treatment,<sup>11</sup> plasma treatment,<sup>12</sup> and coating of hydrophilic finishing agent.<sup>1,12</sup> The hydrophilic mechanism of these methods is based on the introduction of hydrophilic groups (—COOH, —OH, —NH<sub>2</sub>, —SO<sub>3</sub>H, etc.) to the PET macromolecules and the exposure of pits or etches on

the surface of the PET fibers.<sup>13</sup> However, these techniques have several drawbacks that include high chemical cost and energy requirements, generation of undesired ionic groups, yellowing effect, generation of secondary toxic slurries, uncontrolled process, time-consuming, considerable strength loss, and substantial initial capital investment.<sup>8,13–15</sup> Therefore, there is a need to develop effective and facile process for improvement of hydrophilicity of PET fabrics.

Microwave assisted glycolysis has been investigated as a high-efficiency method for recycling of PET bottle waste. The results showed heating under microwave can substantially reduce the time of depolymerization, and the purity of glycolyzed products were much higher than that of traditional methods.<sup>16–18</sup> Since microwave irradiation method has rapid heating with high specificity and this ability makes it suitable for the surface modification of PET fabrics. Up to now, only a few reports on modification the surface of the PET fabric with microwave irradiation were presented. Sulfonating a PET fabric by dilute sulfuric acid and microwave irradiation was found to produce a super-hydrophilic PET fabric.<sup>13</sup> Hydrophilic PET surface was also successfully obtained by applying deep eutectic solvent

under 60 s microwave irradiation with 0.5% NaOH as reported by J. Cho.<sup>14</sup> Furthermore, alkali concentration and the irradiation time were main factors in determining the treatment efficacy.<sup>15</sup> However, the mechanism of hydrophilic modification with microwave irradiation was neither systematic nor deep enough; furthermore, it was really difficult to keep the balance between the hydrophilicity and the original properties of PET fabrics.

Towards this direction, the objective of the current study was therefore to understand the mechanism of hydrophilic modification with microwave irradiation and to investigate the feasibility of making the hydrophilicity controllable. The process parameters such as catalyst variety and dosage, irradiation time were optimized. In addition, the hydrophilicity of modified PET fabrics was examined by contact angle, wicking length, and Methylene Blue staining. Three dimension (3D) laser microscope and field emission scanning electron microscope (FESEM), Fourier transform infrared (FTIR) spectrometer, and thermogravimetric analysis (TGA) were utilized to characterize the morphology, structure, and thermal properties.

## EXPERIMENTAL

### Materials

Polyester filament fabric obtained from the market (plain weave; weight 160 g m<sup>-2</sup>; thickness 270 μm, China) was used as the substrate. Ethylene glycol (C<sub>2</sub>H<sub>6</sub>O<sub>2</sub>, EG), sodium hydroxide (NaOH), lithium hydroxide monohydrate (LiOH), zinc sulfate heptahydrate (ZnSO<sub>4</sub>), sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>), acetic acid (CH<sub>3</sub>COOH), and methylene blue (C<sub>16</sub>H<sub>18</sub>N<sub>3</sub>ClS) were obtained from Sinopharm Chemical Reagent Co., Ltd. (China). All chemicals were of analytical reagent grade and used as received.

### Microwave-Assisted Glycolysis for PET

For the hydrophilic finishing, MKX-J1-3 microwave irradiation synthesizing equipment (Qingdao Maiwei microwave chemical equipment Co. Ltd., China) was employed for the glycolysis. 10 cm × 30 cm PET fabrics were immersed in a bath containing EG with various catalysts (addition was 0.1 – 1% by weight of the fabrics), and the bath ratio was carefully dominated at 1:25. The irradiation time was between 0 s and 180 s. In order to keep constant of bath ratio and concentration of catalysts, a reflux cooler system was installed. After glycolysis process, 1% aqueous acetic acid solution was used to neutralize the alkali, and then rinsed with deionized water for two minutes followed by atmospheric drying. These modified PET fabrics and pristine PET fabrics were stored in a standard environment (21 ± 1 °C, Relative humidity 65 ± 2%) for further use. The weight loss of PET was calculated according to the equation below.

$$WL(\%) = \frac{(W_o - W_t)}{W_o} \times 100 \quad (1)$$

where *WL* is weight loss of PET; *W<sub>o</sub>* is the initial weight of PET fabric (g); and *W<sub>t</sub>* is the weight of PET fabric after the treatment with microwave-assisted glycolysis (g). All experiments were carried out for three times and only mean values were presented.

### Hydrophilicity Test

Contact angles were measured by sessile drop method using OCA20 contact angle goniometer (Data Physics Corporation, Germany). The water drop for the test was 2 μL.

The vertical wicking test was performed by M215 capillary effect machine (Qingdao shanfang instrument Co. Ltd., China) to evaluate the hydrophilicity of the treated PET fabric. At least three replications were made and only mean values were presented.

### Dye Affinity of Modified PET Fabrics

The presence of carboxylic acid groups within the modified PET fabrics was determined by cationic dye sorption process. The original and modified PET fabrics were dyed by Methylene Blue (0.1 g L<sup>-1</sup>) with 1 g L<sup>-1</sup> ammonia for 20 min at 60 °C using liquor ratio 1:50. They were rinsed afterwards with deionized water followed by atmospheric drying. Finally, the reflectance of the PET fabrics was measured by 7000A Colour-Eye (X-rite, USA) and the *K/S* value was calculated according to the Kubelka–Munk equation:

$$K/S = \frac{(1-R)^2}{2R} \quad (2)$$

where *K/S* is the degree of exhaustion of PET fabrics and *R* is the reflectance of PET fabrics at the wavelength of maximum absorption.

### Treatment Durability-Wash Fastness Test

The durability of the hydrophilicity of the modified PET fabrics was assessed using wash fastness test, which was carried out at 60 °C using pure water without soap or detergent. To test the wash fastness of the PET fabrics, a rectangular piece 3 cm × 3 cm of each treated sample was subjected to 20 successive washing cycles of 5 min each by soaking in 20 mL of pure water at room temperature. The contact angles of the pristine and modified PET fabrics were monitored before and after wash fastness tests.

### Characterization of the Modified PET Fabrics

Morphologies of original and modified PET fabrics were observed using 3D laser microscope (VK-X100, Keyence, Japan) and FESEM (JSM-2100F, JEOL Co., Japan).

The chemical structure of the modified PET was examined by Fourier Transform Infrared Spectroscopy Attenuated Total Reflectance (FTIR ATR) analysis using a FTIR spectrophotometer (NEXUS670, Thermo Nicolet Corporation, USA), equipped with a diamond of simple reflection with penetration depth ~ 2 μm. All spectra were recorded at absorption mode of 4 cm<sup>-1</sup> interval and in the range from 4,000 to 600 cm<sup>-1</sup>.

Thermogravimetric analysis (TGA) was performed in a TG209 T1 thermobalance from 30 to 600 °C with a heating rate of 15 °C min<sup>-1</sup> under nitrogen flow (10 mL min<sup>-1</sup>) for detecting degradation temperature at its maximum weight loss.

To evaluate the changes on strengths of the modified PET fabrics, the breaking strength of each PET fabric was measured using Instron 5582 (Canton, MA, USA). The grip separation speed and clipping distance were fixed at 50 mm min<sup>-1</sup> and 100 mm. Each sample was measured five times. All the

experiments were performed at room temperature (20°C) and relative humidity (RH) of 65%.

## RESULTS AND DISCUSSION

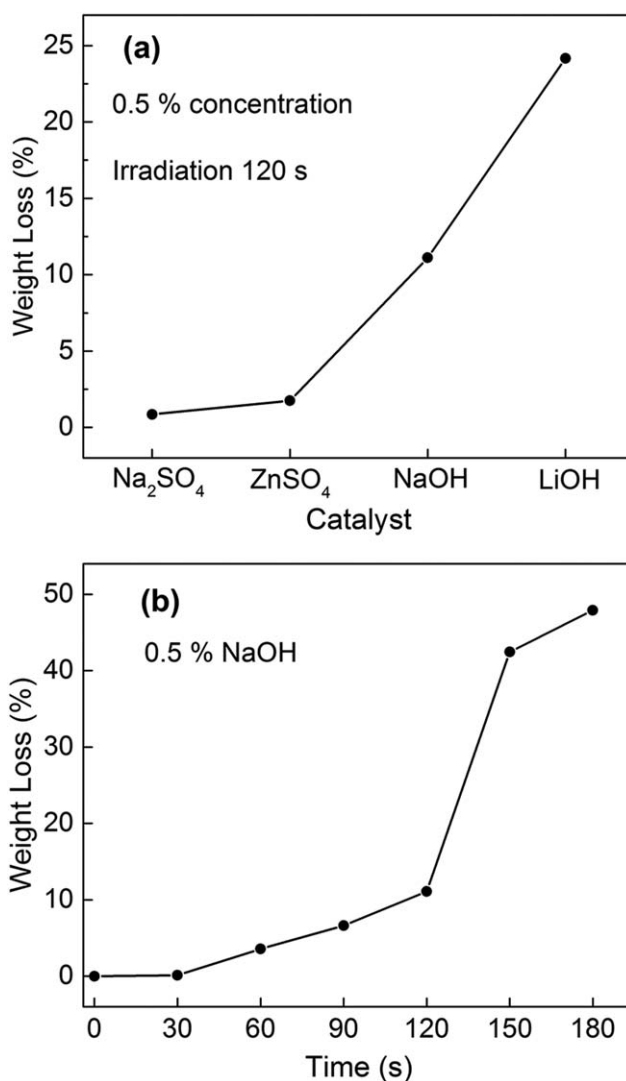
### Optimization of the Micro-Assisted Glycolysis Process

To render the modified PET with highly and durable hydrophilic surface for future textile applications, the process parameters such as catalyst variety and dosage, irradiation time were optimized. Weight loss was usually as an indication to evaluate the glycolysis degree of PET during the microwave assisted treatment process.<sup>15</sup>

Various catalysts, including heavy metallic salt, organic, and alkali metallic salt, have been attempted to glycolysis the PET for recycling process.<sup>18</sup> To obtain high-efficient and environmental protective catalyst, NaOH, LiOH, ZnSO<sub>4</sub>, and Na<sub>2</sub>SO<sub>4</sub> were selected as candidate for microwave assisted glycolysis PET fabrics. Under the selected experimental conditions (Irradiation 120 s, 0.5% catalyst), the effects of catalyst on weight loss of PET were shown in Figure 1(a). For both ZnSO<sub>4</sub> and Na<sub>2</sub>SO<sub>4</sub>, the weight loss of PET was less than 2%, indicating that the catalytic efficiency was very low with very few rupture of ester bond in PET molecules. As comparison, the weight loss of PET was exceeded 10%, when NaOH and LiOH were used as catalysts. Especially, the weight loss of PET reached 24% with LiOH as catalyst. However, it is difficult to control the glycolysis degree of PET, because the glycolysis with LiOH as catalyst was intensive. By comprehensive consideration, NaOH is the best catalyst, which is suitable to glycolysis for PET under microwave-assisted.

The effect of irradiation time was investigated when PET fabric was treated with 0.5% NaOH, and the results were shown in Figure 1(b). There was almost no weight loss during the initial reaction stage (30 s). This was because that vast microwave energy was used to heat the EG solvent, moreover, the glycolysis for PET was difficult to proceed due to its compact structure and smooth surface as seen latter in the 3D laser microscope images. The weight loss of the glycolyzed PET obviously increased as the irradiation time prolonging. It even exceeded 40% after irradiation time of 150 s. However, the structure and properties of PET fabrics were damaged severely. The weight loss of PET was increased linearly as the irradiation time prolonged from 60 s to 120 s, therefore, the glycolysis degree or weight loss of PET could be controlled by changing the irradiation time between 60 s to 120 s.

In order to study the effect of NaOH dosage on the glycolysis modification of PET fabrics, the reactions were carried out with the range of NaOH from 0.1% to 1%. Laser microphotographs of PET fabrics modified with different amount of NaOH were shown in Figure 2. The pristine PET had uniform diameter and homogeneous structure with smooth surface as shown in Figure 2(a). Compared with pristine PET, 0.1% addition of NaOH could make its morphology change slightly [Figure 2(b)]. It indicated that the degree of glycolysis reaction on the surface of PET fabrics was neglectably small. With the amount of NaOH increased from 0.2% to 0.5% [see Figure 2(c–e)], the diameter of modified PET became small and the surface roughness

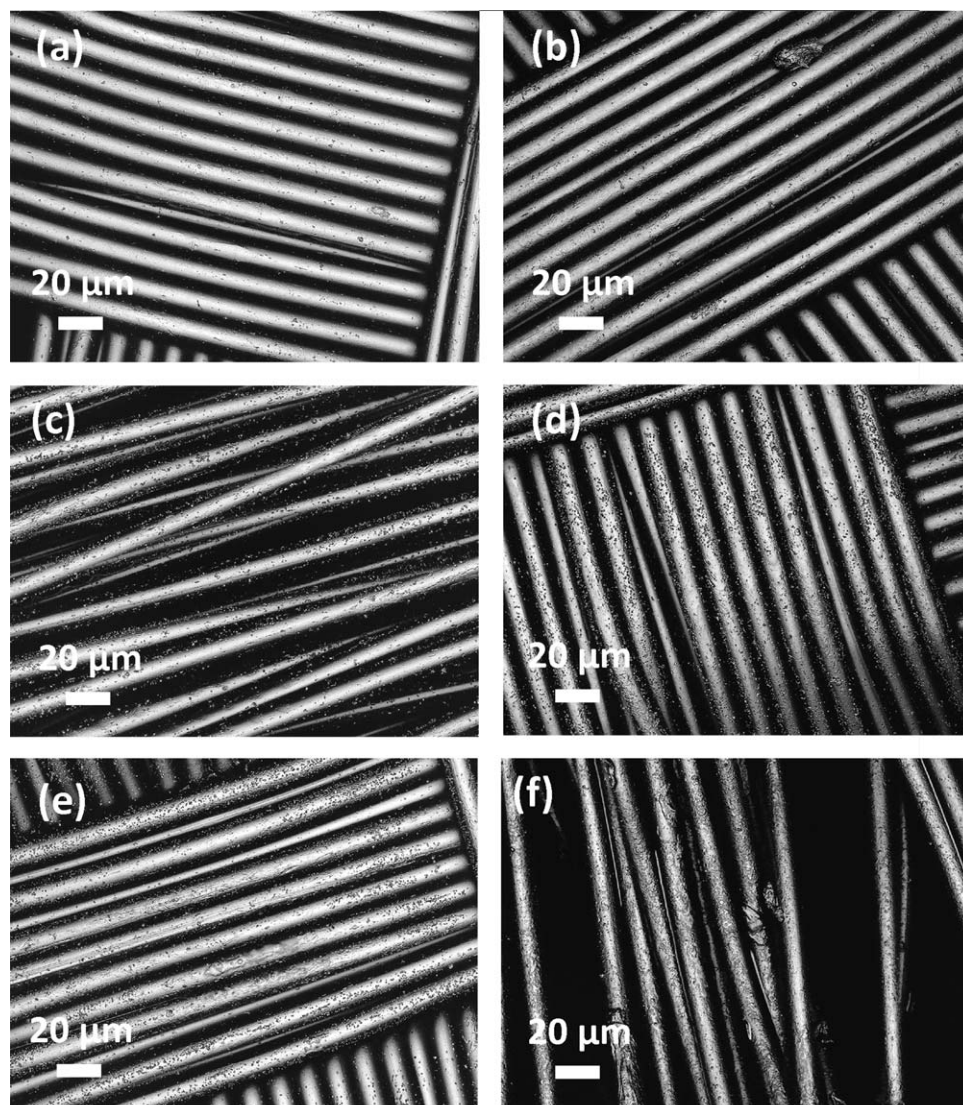


**Figure 1.** Effect of (a) catalyst and (b) irradiation time on weight loss of modified PET fabrics.

increased. However, the morphology of modified PET deteriorated seriously when the content of NaOH reached 1%, due to its severe surface cracks. The possible reason was that the main chains were over-reacted and seriously destroyed during the glycolysis process at 1% NaOH.

### Hydrophilicity Analyses of Modified PET

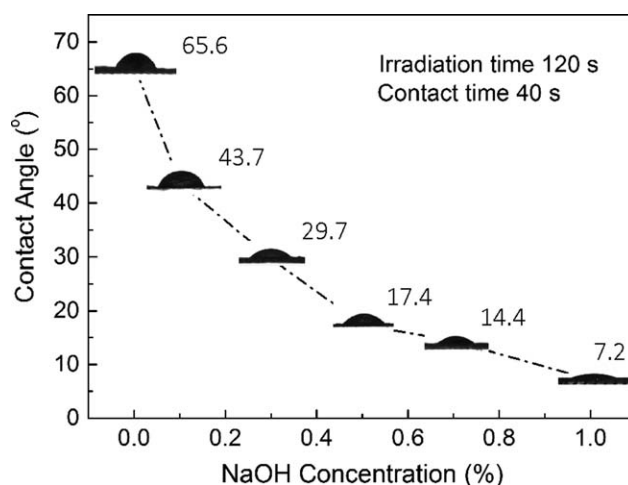
After studying the optimal conditions for the glycolysis process, the hydrophilicity of modified PET fabrics was measured and analyzed. Contact angle were usually used as an indication to evaluate the surface hydrophilicity of products during the pre-treatment process of dyeing industry.<sup>19</sup> Therefore, the contact angles of modified PET (contact time 40 s, irradiation 120 s) were measured and compared in Figure 3. The untreated PET fabric had a low wettability with the contact angle of 65.6°, which was much less than that reported (92.6°) by J Cho.<sup>14</sup> Comparing with the pristine PET, the contact angles of modified PET fabrics were dramatically reduced, indicating marked improvement in the hydrophilicity. Furthermore, the contact angle consistently decreased with the increase in NaOH



**Figure 2.** Three dimension laser microphotographs of modified PET with different NaOH concentration (a) 0%, (b) 0.1%, (c) 0.2%, (d) 0.4%, (e) 0.5%, and (f) 1%.

concentration. It dramatically decreased to  $43.7^\circ$  with 0.1% NaOH, exhibiting better surface hydrophilicity. The contact angle decreased gradually with the amount of NaOH increasing from 0.3% to 0.7%. The contact angle even reached less than  $15^\circ$  with 0.7% NaOH. The linear variation contact angles (NaOH concentration 0.3% to 0.7%) revealed that the hydrophilicity of modified PET fabric was able to be adjusted by changing the content of NaOH with optimal process. The reasons for the improvement of hydrophilicity of PET fabrics will be investigated by Methylene Blue staining and FESEM. At the treatment condition with higher NaOH dosage (over 1%), the contact angle reduced almost to zero, but some holes appeared on the fabric because of gravely degradation the PET molecules.

In addition, the existence time of the droplet on the surface of PET was remarkably shortened as shown is Figure 4. The contact angle dropped from at  $37^\circ$  (20 s) to  $17.4^\circ$  (40 s), and the contact angle could not be observed after 60 s (see Figure 4).



**Figure 3.** Contact angles of modified PET fabrics.

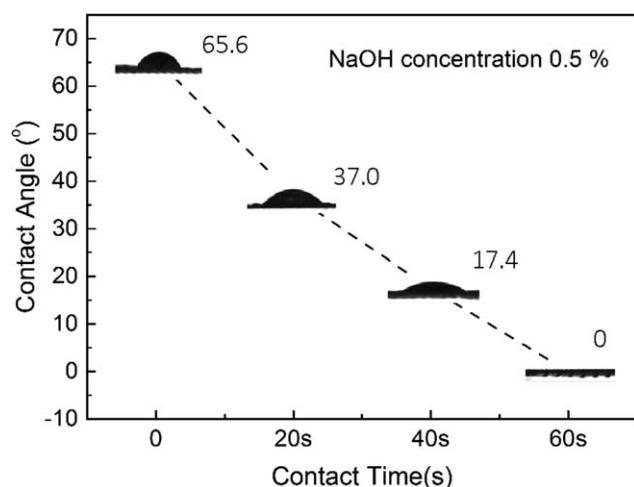


Figure 4. The relationship between contact angle and contact time.

To further confirm the highly hydrophilicity of modified PET fabrics, the wicking length was measured and presented in Figure 5. The vertical wicking length at the equilibrium state was increased with the increasing of NaOH concentration. As shown in Figure 5, the glycolized PET fabric using 0.5% NaOH as catalyst had a wicking length of 19.36 mm, which was much higher than that of pristine PET fabric (12.11 mm). These results implied the highly hydrophilicity of modified PET fabrics, which were consistent with the results of the contact angle. We could conclude that the polyester filament fabric with highly hydrophilic surface can be easily fabricated by microwave-assisted glycolysis. The reasons for the improvement of hydrophilicity of PET fabrics will be seen latter in the Methylene Blue staining (the exposure of the carboxyl- and hydroxyl-end groups) and FESEM images (etches on the surface of the PET fibers). According to the report by J Cho, the wicking length was mainly depended on the surface hydrophilicity (the number of  $-OH$  and  $-COOH$ ) and pore size.<sup>14</sup>

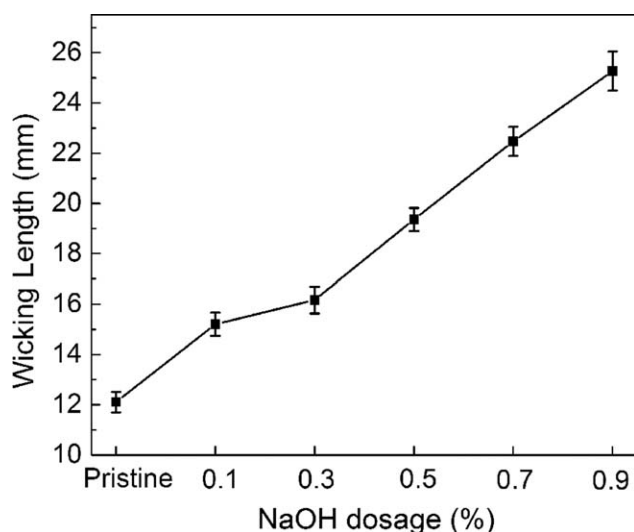


Figure 5. The relationship between the NaOH concentration and the wicking length.

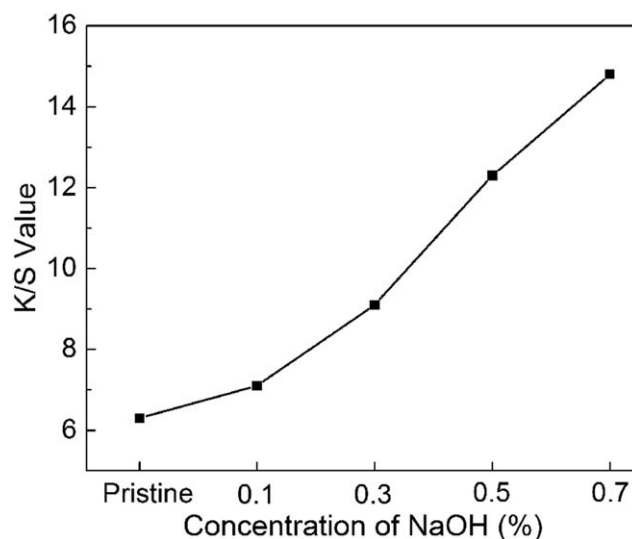


Figure 6. The relationship between the NaOH concentration and the  $K/S$  value.

To understand how hydrophilic end groups and pores size support the highly and adjustable hydrophilicity of modified PET fabrics, Methylene Blue was selected as a model cationic dyes and the  $K/S$  value was used to indicate the presence of

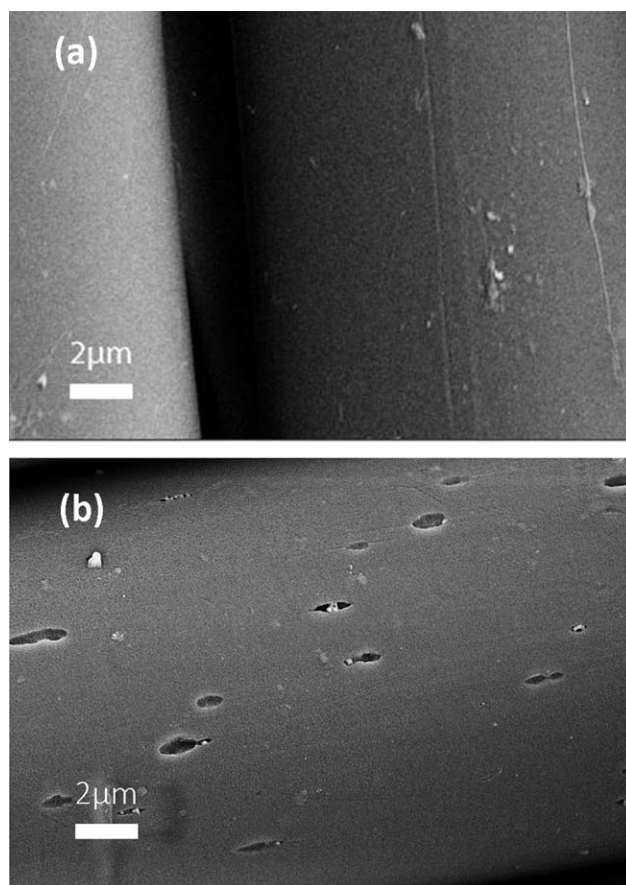
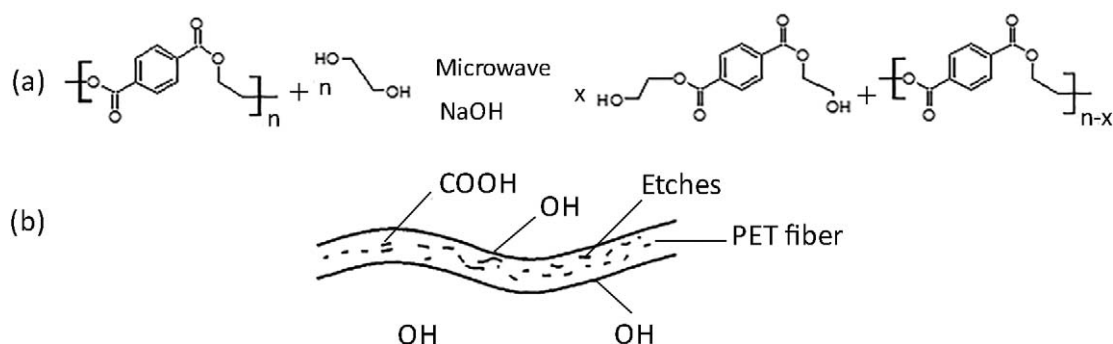


Figure 7. Surface SEM depictions of (a) untreated and (b) glycolized PET at optimal conditions PET fibers (NaOH concentration 0.5%, irradiation 120 s).



**Figure 8.** Schematic illustration of (a) glycolysis of PET with EG and (b) highly hydrophilic surface of modified PET fabrics.

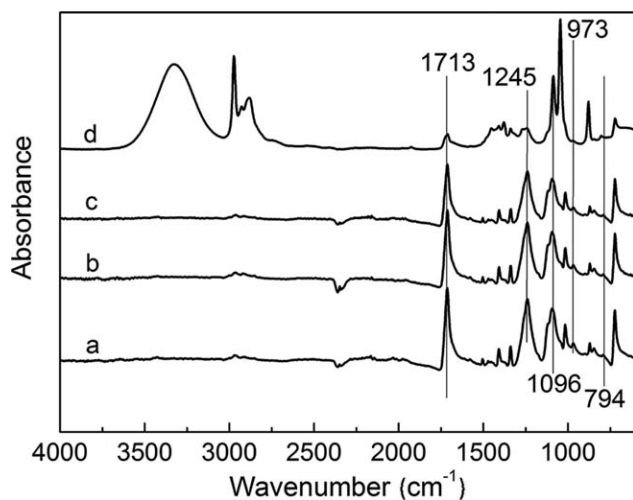
hydrophilic end groups (The larger the  $K/S$  value, the deeper the color and the higher adsorptive capacity of dye<sup>20,21</sup>). In Figure 6, the  $K/S$  values of the dyed modified fabrics were presented in comparison to the pristine fabric. There was a significant color variation after hydrophilic modification, indicating hydrophilic end groups ( $-\text{COOH}$  and  $-\text{OH}$ ) were successfully introduced into the surface of PET fabrics. The  $K/S$  value of the pristine PET fabric was 6.31, and that of the modified PET fabrics were much higher. The  $K/S$  value reached 12.37 at 0.5% NaOH. Furthermore, the  $K/S$  value clearly increased as the NaOH concentration increased. These reported values indicated increased dye adsorption by the glycolyzed PET fabrics, further proven the existence of hydrophilic end groups.

As shown in the FESEM images of pristine PET fabric and glycolyzed PET fabric (0.5% NaOH, irradiation 120 s), respectively (Figure 7), the hydrophilic modification is visualized by the presence of small etches or grooves on the surface of the treated PET compared to the pristine one. These significant changes were caused by glycolysis, as mentioned terephthalic acid (TPA) equivalents released in the reaction supernatant during the glycolysis process. Furthermore, the number and size of etches and grooves could be controlled by the glycolysis process as mentioned above. This etches or grooves on the surface of PET

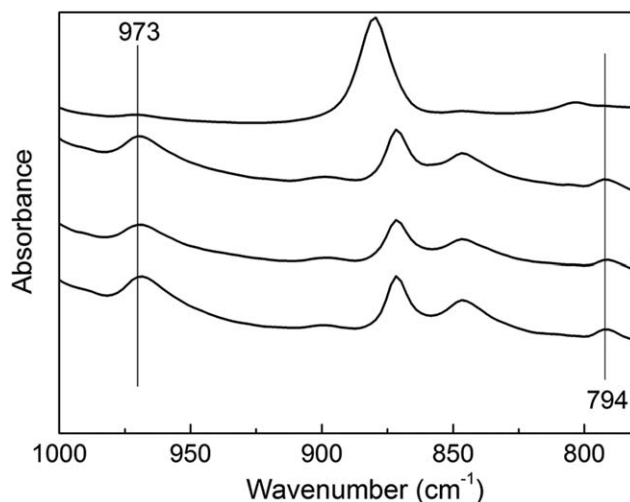
fabrics could increase the capillary pressure, and further lead to the highly hydrophilic surface.

The mechanism of highly hydrophilic PET fabrics by microwave-assisted glycolysis could be proposed as the interaction of the rough surface (etches on the surface of PET) and the exposure of the carboxyl- and hydroxyl-end groups on the surface of PET (see Figure 8). The glycolysis of the PET fabric leads to the exposure of the carboxyl- and hydroxyl-end groups on the surface of PET, while terephthalic acid (TPA) equivalents, were released in the reaction supernatant [see Figure 8(a)]. This method result in the introduction of hydrophilic end groups and etches to the PET macromolecules to make the fabric hydrophilic.

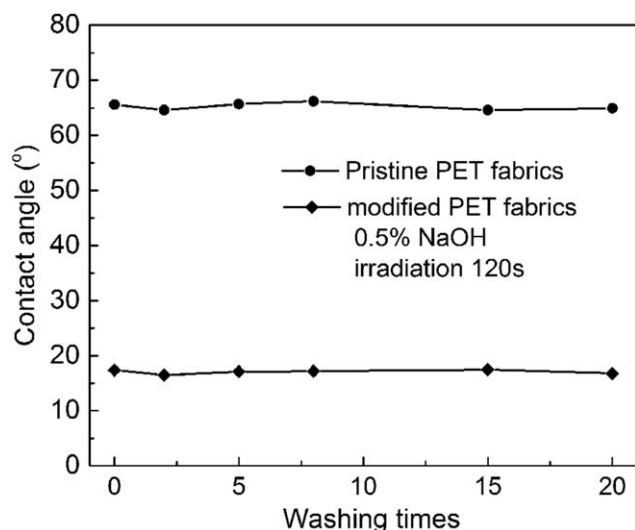
Aiming at the spectroscopic identification of the changes on the glycolyzed PET surface, ATR-FTIR was carried out, as shown in Figure 9. The pristine and modified PET all had the same main absorption peaks. A strong peak appeared at  $1713\text{ cm}^{-1}$  was assigned to the  $-\text{C}=\text{O}$  stretching of ester bond in PET. The characteristic peaks at  $1245\text{ cm}^{-1}$  and  $1096\text{ cm}^{-1}$  were attributed to the stretching of  $-\text{C}-\text{O}-$ .<sup>13</sup> In comparison of pristine PET fabric, the intensity of these three peaks weakened gradually with the increasing of NaOH concentration. These results



**Figure 9.** FTIR spectra of PET fabrics treated with different concentration of NaOH (a) 0%, (b) 0.1%, (c) 0.5%, and (d) 1%.

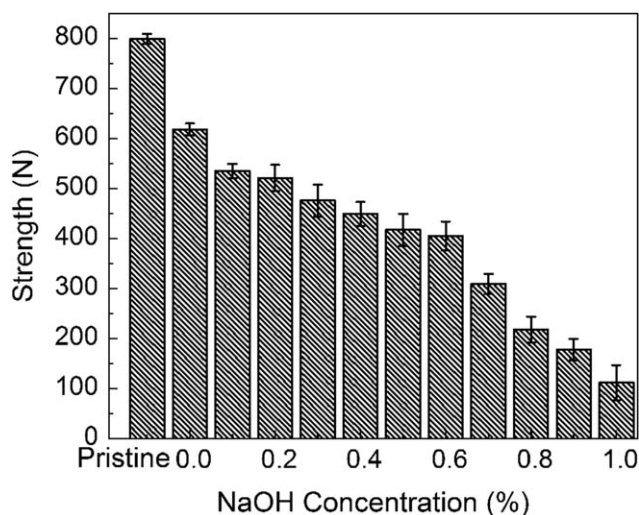


**Figure 10.** The varieties of the main characteristic peaks of PET fabrics treated with different concentration of NaOH (a) 0%, (b) 0.1%, (c) 0.5%, and (d) 1%.



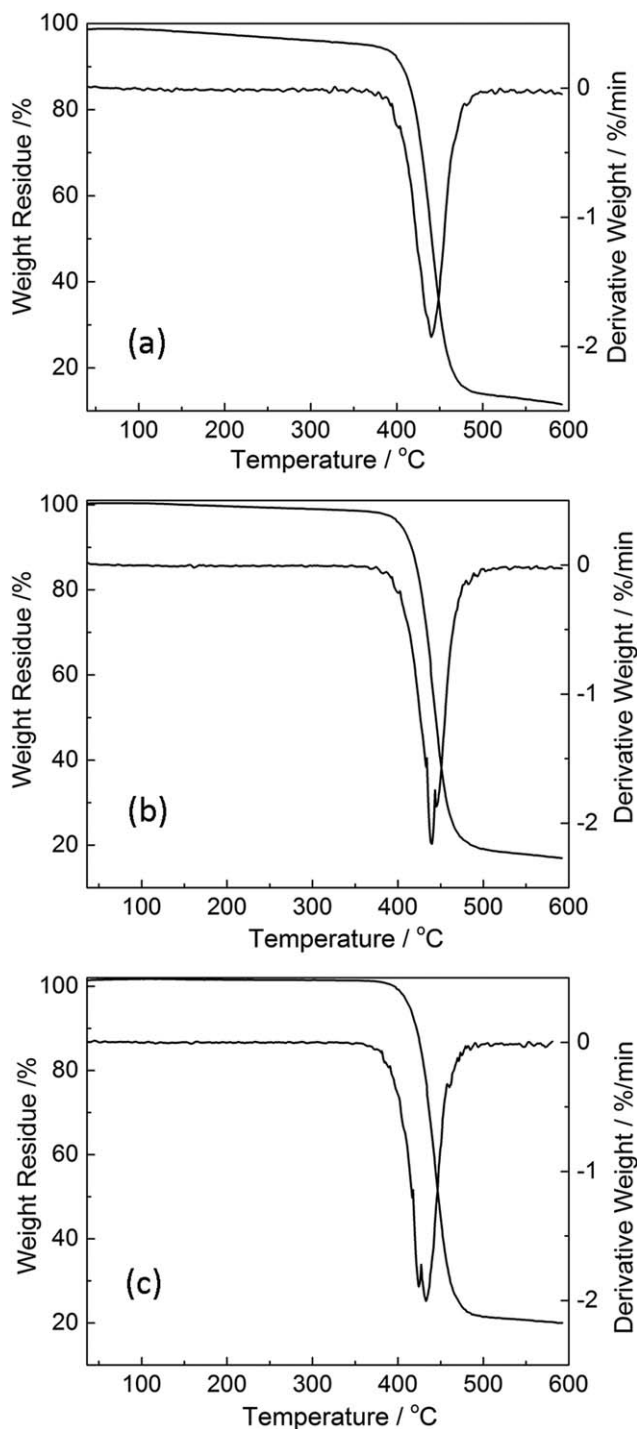
**Figure 11.** Variation in contact angle with increasing washing cycles of pristine and modified PET fabrics.

indicated that the higher the concentration of NaOH, the larger the number of ruptured ester bond of PET and more hydroxyl and carboxyl end groups were obtained. However, no peaks at  $3700\text{--}3300\text{ cm}^{-1}$ , which attributed to  $\text{--OH}$  and  $\text{--COOH}$ , were observed when the concentration of NaOH increased from 0.1 to 0.5%, confirming that glycolysis degree of PET was low under the selected experimental conditions. This was probably related to the relative lower ratio of depolymerized ester bond comparing to that of ester bond in PET fabric, further lead to the characteristic peaks of  $\text{--OH}$  and  $\text{--COOH}$  cannot be shown in FTIR spectra. Furthermore, the level of free  $\text{--COOH}$  and  $\text{--OH}$  groups was rather small. However, a strong characteristic peak at  $3431\text{ cm}^{-1}$  were observed at 1% concentration of NaOH, proved the formation of the large number of  $\text{--OH}$  and  $\text{--COOH}$  groups. However, the structure and textile properties were damaged as mentioned above.



**Figure 12.** Effect of NaOH concentration on strength of modified PET fabrics.

In addition, the varieties of the main characteristic peaks at  $973$  and  $794\text{ cm}^{-1}$  were presented in Figure 10. The peaks at  $973\text{ cm}^{-1}$  and  $795\text{ cm}^{-1}$  were assigned to the Trans isomer of ethylene glycol linkage and the internal thickness band. The ration between  $973$  and  $794\text{ cm}^{-1}$  was used to determine effect of drawing on crystallinity.<sup>14</sup> It was noted that this ratio was lower in the modified PET than that of original PET, which indicated the decreasing of the crystallinity and orientation



**Figure 13.** TG curves of PET fabrics treated by microwave irradiation fabrics with different concentration of NaOH (a) 0%, (b) 0.5%, and (c) 1%.

**Table I.** TG Characteristic Parameters of PET Fabrics

NaOH concentration (%)	Initial temperature (°C)	Peak temperature (°C)	Final temperature (°C)	Residue (%)
0	359.5	442.0	501.4	11.51
0.5	369.9	439.5	537.5	16.94
1	359.5	434.4	506.5	19.99

degree on the surface of the modified PET. The mechanism might be proposed as both disruption of chain extension and transformation of trans-configuration of EG to gauche configuration after glycolysis. This fact was proved by Jae Cho.<sup>14</sup>

To evaluate the hydrophilicity change and durability of the modified PET fabrics in continuous recycling, the wash fastness test was performed for several washing cycles. Contact angle was measured after each washing cycle. Figure 11 showed the change of the hydrophilicity of modified PET fabrics (0.5% NaOH, irradiation time 120 s) during repeating recycling. As a result, the number of washing cycles did not affect the water contact angle during the whole process. The contact angle of the selected sample was steadily maintained around 15° (The contact angle of the pristine PET after washing kept at over 60°). Therefore, the microwave-assisted glycolysis process is very suitable method for hydrophilic finishing of PET and has an economically great benefit for practical use.

Herein, we draw the conclusion that the microwave-assisted glycolysis for PET fabric modification were successful, the glycolyzed PET fabrics results in the exposure of the carboxyl- and hydroxyl-end groups on the surface of PET and introduction grooves to make the fabric hydrophilic. Furthermore, the durable hydrophilicity of the modified PET fabric could be controlled by the degree of glycolysis through changing the concentration of NaOH and irradiation time.

#### Mechanical and Thermal Properties of Treated PET

The mechanical properties such as tensile strength of PET fabrics were an important index to define the applications.<sup>2</sup> Therefore, the glycolysis process should not cause excessive descent that would render the final product unsuitable to use. The breaking strength of the original and modified PET fabrics was evaluated, and the results were presented in Figure 12. The breaking strength of the pristine PET fabric reached 799.4 N. In comparison of pristine PET fabric, the breaking strength decreased sharply step-by-step as the concentration of NaOH increased. The breaking strength of the PET fabrics modified by using NaOH as catalyst at a concentration from 0.1% to 0.6% was over 400 N, which was high enough for it to be used as textile materials.<sup>13,22,23</sup> The reason was that the glycolysis for PET was occurred on the surface of PET fibers, and the main chains of PET molecules were well maintained. These results were consistent with ATR-FTIR results. However, the breaking strength decreased more than 50% when the NaOH concentration was too high (0.7%). The significant strength loss might be caused by the reduction in degree of polymerization of PET polymers, the glycolysis, and hydrolysis further occurred through etches and grooves, resulting in the rupture of main

chain. From the commercialization point of view, it was important that PET have excellent wearability properties, therefore, the largest dosage of NaOH should be under 0.6%.

To find any change of the thermal properties of the modified PET fabrics. Thermogravimetric (TG) analyses were carried out. There were no obvious hydration stage ranging from 70 to 150 °C because of the PET fabrics were stored in desiccator (Figure 13). From TG analysis, the weight residue of the pristine PET fabric was 11.5% at 600 °C, whereas the modified PET fabric was about 17% (Table I). The increased weight residue might be caused by the EG participation in the glycolysis process. It can be discerned that the glycolysis under the selected experimental conditions had little effects on the thermal properties of the PET fabrics.

Furthermore, the differential thermogravimetric analysis (DTA) curves were obtained by differentiating TG curves shown in Figure 13. The pristine PET fabrics had a sharp thermal decomposition peak at about 442 °C (see Figure 13(a) and Table I). In contrast, the maximum weight loss of modified PET was shifted to 440 °C with the concentration of NaOH at 0.5% and at 435 °C for 1% NaOH (sees Figure 13(b) and Table I). Moreover, several shoulder peaks were observed around the main decomposition peak, indicating the degree of polymerization changed, due to the glycolysis process. As we can conclude that the thermal properties of the modified PET fabrics under the selected experimental conditions were well maintained, nevertheless small variations exist and are indicative of the microwave-assisted modification.

#### CONCLUSIONS

In the present investigation, the polyester filament fabrics with highly and durable hydrophilic surface were fabricated by microwave-assisted glycolysis techniques. The process parameters such as catalyst variety, dosage, and irradiation time were optimized by using weight loss were as an indication. The results showed that sodium hydroxide was suitable to glycolysis for PET under microwave-assistance. The modified PET fabric (0.5% NaOH, irradiation time 120 s) showed highly surface hydrophilicity with a contact angle of 17.4° and a wicking length of 19.36 mm. The exposure of the carboxyl- and hydroxyl-end groups on the surface of PET and the introduction of etches were confirmed by Methylene Blue staining and FESEM, respectively. The strength of modified PET was decreased. However, the thermal properties of the modified PET fabrics under the selected experimental conditions were well maintained. The highly hydrophilicity and its original properties of PET could be controlled by changing the irradiation time



from 60 s to 120 s and adjusting the content of sodium hydroxide from 0.2% to 0.5%. These results suggest microwave-assisted glycolysis is an effective method for polyester hydrophilic finishing.

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#### REFERENCES

1. Zaman, M.; Liu, H. B.; Xiao, H. N.; Chibante, F.; Ni, Y. H. *Carbohydr. Polym.* **2013**, *91*, 560.
2. Kordoghli, B.; Khiari, R.; Dhaouadi, H.; Belgacem, M. N.; Mhenni, M. F.; Sakli, F. *Colloid Surf. A* **2014**, *441*, 606.
3. Lee, S. H.; Song, W. S. *Fiber. Polym* **2010**, *11*, 54.
4. Zhao, M. L.; Li, F. X.; Yu, J. Y.; Wang, X. L. *J. Appl. Polym. Sci.* **2014**, *131*, 39823.
5. Alicia, A.; Martínez, L.; Becerra, L.; Arieta-araunabeña, M.; Arnaiz, S.; Asueta, A.; Robertson, I. *J. Mater. Cycles. Waste* **2014**, *16*, 201.
6. Mancini, S. D.; Nogueira, A. R.; Rangel, E. C.; Cruz, N. C. *J. App. Polym. Sci.* **2013**, *127*, 1898.
7. Donellia, I.; Freddia, G.; Nierstraszb, V. A.; Taddeic, P. *Polym. Degrad. Stabil.* **2010**, *95*, 1542.
8. Zhu, Y.; Mao, Z. W.; Gao, C. Y. *RSC. Adv.* **2013**, *3*, 2509.
9. More, A. P.; Kute, R. A.; Mhaske, S. T. *Iran. Polym. J.* **2014**, *23*, 59.
10. Liu, F.; Chen, J. Y.; Li, Z. L.; Ni, P.; Ji, Y. M.; Meng, Q. Y. *J. Anal. Appl. Pyrol.* **2013**, *99*, 16.
11. Kanelli, M.; Vasilakos, S.; Nikolaivits, E.; Ladas, S.; Christakopoulos, P.; Topakas, E. *Process Biochem.* **2015**, *50*, 1885.
12. Lv, J. C.; Zhou, Q. Q.; Zhi, T.; Gao, D. W.; Wang, C. X. *J. Clean. Prod.* **2016**, *118*, 187.
13. Xu, F.; Zhang, G. X.; Zhang, F. X.; Zhang, Y. S. *Appl. Surf. Sci.* **2015**, *15*, 437.
14. Cho, J. Y.; Choi, H. M.; Oh, K. W. *Text. Res. J.* **2015**, *86*, 1318.
15. Cho, J. Y.; Hong, C. J.; Choi, H. M. *Ind. Eng. Chem. Res.* **2013**, *52*, 2309.
16. Chaudhary, S.; Surekha, P.; Kumar, D.; Rajagopal, C.; Roy, P. K. *J. Appl. Polym. Sci.* **2013**, *129*, 2779.
17. Pingale, N. D.; Shukla, S. R. *Eur. Polym. J.* **2008**, *44*, 4151.
18. Siddiqui, M. N.; Redhwi, H. H.; Achilias, D. S. *J. Anal. Appl. Pyrol.* **2012**, *98*, 214.
19. Xue, C. H.; Ji, P. T.; Zhang, P.; Li, Y. R.; Jia, S. T. *Appl. Surf. Sci.* **2013**, *284*, 464.
20. Akgun, M.; Becerir, B.; Alpay, H. R. *Fiber. Polym.* **2014**, *15*, 126.
21. Xie, K. L.; Gao, A. Q.; Li, M.; Wang, X. *Carbohydr. Polym.* **2014**, *101*, 666.
22. Palaskar, S. S.; Desai, A. N.; Shukla, S. R. *J. Text. I.* **2016**, *107*, 405.
23. Hong, K. H. *Cellulose* **2015**, *22*, 2129.