

Hydrophilic Property of Polyester Fabric Coated with Polyethylene Glycolated Bisphenol A

Suthasinee Ploymalee,¹ Sireerat Charuchinda,^{1,2} Kawee Srikulkit^{1,2}

¹Department of Materials Science, Faculty of Science, Chulalongkorn University, Bangkok 10330, Thailand

²National Center of Excellence for Petroleum, Petrochemicals and Advanced Materials, Chulalongkorn University, Bangkok 10330, Thailand

Received 29 March 2009; accepted 28 September 2009

DOI 10.1002/app.31530

Published online 1 December 2009 in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: Polyethylene glycolated bisphenol A (PEGBPA), a hydrophilic–hydrophobic compound, was synthesized and applied onto polyester fabric using a pad-dry-cure method to impart a durable hydrophilic property. After coating and heat fixation, the treated fabrics were evaluated for wettability by measurement of the net moisture regain and wicking distance, and their surfaces were characterized by SEM and ATR/FTIR spectroscopy. The durability of the treated fabrics were tested by ten standard repeat washings and samples were characterized for their wettability after each wash. The surface properties of the fabric changed from hydrophobic to hydrophilic after heat treatment with the coating agent PEGBPA. Using the degree of increased moisture regain and wicking distance of the treated fabrics as a guide,

optimal treatment was attained by coating with 10–20 g/L PEGBPA followed by 160–180°C heat treatment for 3 min. Wash fastness evaluations, coupled with SEM and ATR/FTIR analyses, showed that PEGBPA exhibited good adhesion onto the PET surface and was capable of withstanding repeated washings. It was concluded that the coating adhesion solely depended on physical aspects, such as hydrophobic–hydrophobic interactions between the bisphenol A segment of PEGBPA and the PET aromatic segment. © 2009 Wiley Periodicals, Inc. *J Appl Polym Sci* 116: 473–478, 2010

Key words: polyester; hydrophilic; hydrophilic–hydrophobic compound; polyethylene glycolated bisphenol A

INTRODUCTION

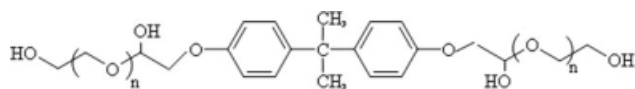
Thermoplastic synthetic fibers, such as typically polyesters and especially polyethylene terephthalate (PET), are one of the most important ‘man-made’ synthetic fibers as they have excellent mechanical properties, and chemical and heat resistance. However, due to the hydrophobic nature of polyester, compared to cotton, they lack the properties associated with hydrophilic fibers, which are desirable for producing clothes, such as antistatic, sweat-absorbing, and soil-releasing properties.

Polyester fabrics used in direct contact with or near to the skin as an innerwear, intermediate wear, or sportswear are thus inferior to natural fibers in the feeling of comfort as perspiration from the skin is not efficiently wicked away making the wearer feel stuffy and sticky. Moreover, polyesters are more electrostatic than natural fibers, as it is characteristic of fibers that have low moisture absorbency, which is undesirable as it causes clothes to cling to each other. To eliminate these disadvantages, static build-up on polyester fabrics is controlled by surface med-

ication with cationic surfactants.^{1–3} However, most of these compounds are not durable requiring repeated applications with concomitant economic and environmental costs and concerns.

Research into improving the surface properties of polyester based materials, such as the production of multicomponent fibers or sheath/core fibers, has consequently attracted much attention.^{4–8} In these cases, the outer sheath layer is comprised of a hydrophilic–hydrophobic copolymer, which is more hydrophilic in nature to absorb moisture and dissipate the static charge, whereas, the inner core layer has only hydrophobic properties and retains, for example, the crease resistance properties. However, the production of these kinds of multicomponent or composite fibers requires sophisticated technology. Finally, the method of grafting hydrophilic or hydrophobic copolymers onto the surface of polyester, for example, the grafting of acrylic acid, poly(acrylic acid) or ethyl acrylate onto polyester using benzoyl peroxide initiator,⁹ is not economic sound. Improved grafting copolymerization efficiency may be achieved by pretreatment of the polyester before grafting. Studies of the plasma treatment¹⁰ or swelling assisted treatment¹¹ of polyester have shown better wettability and dissipability of static charge. However, the disadvantages of graft copolymerization by some techniques are that it requires a lengthy treatment time and so is not

Correspondence to: K. Srikulkit (kawee@sc.chula.ac.th).



Scheme 1 The structure of polyethylene glycolated bisphenol A (PEG segment with M_w of 1000)

commercially viable for applying to polyester fabrics for the textile industry.

An alternative approach to improve the PET hydrophilicity is by hydrolysis of PET using alkali, such as sodium hydroxide,¹² or enzymes, such as lipase,¹³ and coating with hydrophilic polymers. The utilization of natural polymers such as chitosan for improving the antistatic and hydrophilic properties of polyester has been reported.¹⁴ As the chitosan has free amine groups, it is possible to crosslink them with the hydroxyl group-functionalized polyester using a catalyst and diacids to produce an antistatic finish. Such crosslinked chitosan–polyester derivatives have been shown to have a lower electrostatic voltage than untreated polyesters, which is attributable to the hydrophilic contribution of chitosan. In another case, the synthetic hydrophilic copolyester, polyoxyethylene diester, was applied to PET fabrics to impart a durable hydrophilic coating to polyester and polyolefin fabrics.¹⁵

Here, in this report, a hydrophilic–hydrophobic copolymer (polyethylene glycolated bisphenol A (PEGBPA)), was synthesized. Then, the coating of the hydrophilic–hydrophobic copolymer onto PET fabrics was undertaken to render a hydrophilic polyester fiber surface. It was anticipated that the aromatic segment would be capable of firmly adhering to the polyester surface by hydrophobic interactions, hence allowing the durable adhesion of hydrophilic segment on the fiber surface. The scope of this investigation was focused on the effect of application factors affecting the adhesion capability of the modifier onto the polyester surface, and finally the evaluation of the surface properties of the modified polyester, such as moisture regain, wettability, and adhesion ability of the modified PET.

EXPERIMENTAL

Bisphenol A and poly(ethylene glycol) with M_w of 1000 were purchased from Fluka (Switzerland). Epichlorohydrin (industrial grade) was kindly provided by Thai Organic Industry Co., (Thailand). 100% woven PET fabric without heat-set treatment was purchased from a local textile mill.

Synthesis of polyethylene glycolated bisphenol A

Epichlorohydrin (92.5 g, 1 mol) was mixed with bisphenol A (69 g, 0.5 mol) in a round bottom flask

equipped with dropping funnel containing 100 mL (~ 1 mol) of 37 wt % HCl solution. On the course of 2 h, HCl solution was slowly added into the epichlorohydrin/bisphenol A mixture under continuous stirring. The acid-catalyzed ringopening of epichlorohydrin with bisphenol A proceeded immediately, yielding an intermediate, 3-chloro-2-hydroxypropyl-bisphenol A. After the completion of HCl addition, 3-chloro-2-hydroxypropylbisphenol A was isolated as white powder (50% yield).

3-chloro-2-hydroxypropylbisphenol A (50 g, 0.166 mol) and polyethylene glycol 1000 (332 g, 0.332 mol) was added into 100 mL of water under continuous stirring at room temperature. Then, sodium hydroxide flake (15 g, 0.375 mol) was portion-wise added to the mixture and the reaction was continued for 5 h to obtain the clear solution. The structure of PEGBPA was purposed as shown in Scheme 1. The obtained solution was applied onto PET fabric without purification. Before application, the solid content was adjusted to 50 wt % by addition of water. Proton NMR analysis (taken on Bruker DPX-300 spectrometer) of PEGBPA sample confirmed the structure of PEGBPA by the presence of aromatic proton signals at 7.5 ppm and 6.5 ppm, and methylene ($-\text{CH}_2-$) proton signal at 3.5 ppm. Typical ^1H NMR of PEGBPA is shown in Figure 1.

Application of PEGBPA onto polyester fabric

The treatment of the PET fabrics with PEGBPA was carried out using a pad-dry cure application method. First, a series of PEGBPA padding solutions with concentrations varying from 5 to 20 g/L were prepared and each solution was adjusted to pH 6 using acetic acid. The PET fabric was then treated with the prepared solution using a pad mangle with the pressure-nip set to give 80% wet pickup, then dried at 100°C for 5 min and then thermofixed at temperatures between 150–180°C for 3 min using a

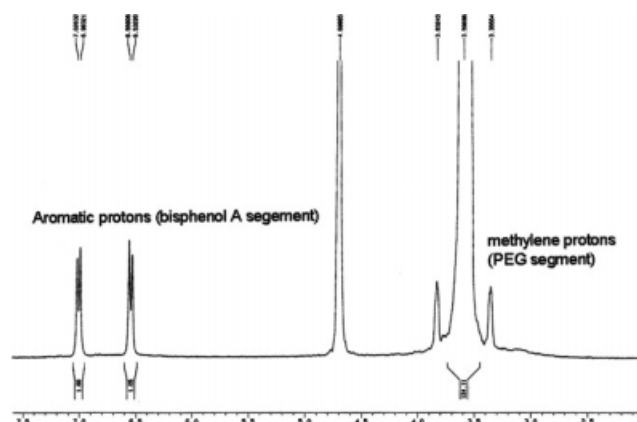


Figure 1 ^1H -NMR of polyethylene glycolated bisphenol A.

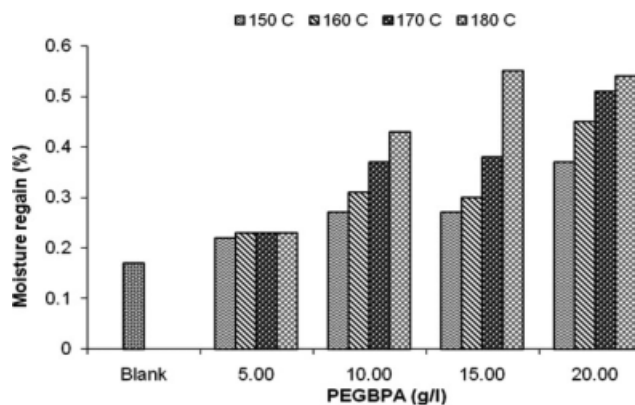


Figure 2 Effect of PEGBPA concentration and fixation temperature on the percent moisture regain of the resultant treated PET fabric.

mini-stenter machine. The treated fabrics were rinsed with tap water and dried in open air. Following that, the treated fabrics were subjected to surface performance evaluation, including moisture regain, wettability, and durability as outlined below.

Evaluation of properties of treated polyester fabrics

Determination of moisture regain

Moisture regain is the percent weight of moisture compared to the initial fabric dry weight and was determined by Infrared moisture determination balance (AND AD-4715). The regain of the material is considered to be the amount of moisture after 24 h under the standard conditions of $27 \pm 2^\circ\text{C}$ and a relative humidity of $65 \pm 2\%$.

Evaluation of the wettability of treated PET fabrics

The wettability of untreated and treated PET fabrics was evaluated by the wicking test. The PET fabric was cut to samples of 2.5×17.5 cm and then clipped at the top and bottom margin by 0.6 cm with a 2–3 g clip. After that the edge of the PET fabric was immersed to 0.6 cm depth into a 1% (w/v) water soluble dye (a reactive blue dye) solution (400–600 mL in a 1000 mL beaker) for 3 min. The distance the dye solution had moved up the fabric was then measured.

Evaluation of the durability of treated PET fabrics

The evaluation of washing fastness of the treated PET fabrics was performed according to the standard AATCC Test Method No. 61-2001 using a laundering machine (Gyrowash) at 40°C in a 500 mL stainless-steel lever-lock canister. The fabric was leached in 200 mL of 0.37% (w/v) aqueous solution of the AATCC standard reference detergent. Fiber

surface morphology was obtained by scanning electron microscopy (SEM; Model JSM-5410LV, JEOL, Tokyo, Japan), operated at 15 kv accelerating voltage. ATR/FTIR spectroscopy was recorded on a Perkin–Elmer FT-2000 Fourier transform spectrophotometer.

RESULTS AND DISCUSSION

Surface properties of treated polyester fabric

The results for the standard moisture regain evaluation for treated and untreated PET fabrics after 24 h at a relative humidity of 65% are shown in Figure 2. Untreated PET fabrics revealed low moisture regain of only 0.17%, consistent with the hydrophobic nature of PET. However, the PEGBPA treated PET fabrics showed a much and significantly higher moisture regains, being found within the range of 0.2–0.6%. The increase in the treated PET fabric moisture regain was clearly dose-dependent, increasing with an increase in PEGBPA concentration. It is noted that the concentration in the range of 10–20 g/L coating agent is enough to improve the moisture regain property. The results indicate that the PET fabric after treatment with PEGBPA is more hydrophilic, and is likely to be due to the presence of the PEG segment on fiber surface.

The results of the vertical wicking test on the untreated and treated PET fabric samples (Fig. 3) are in good agreement with the percent moisture regain, where the treated PET fabrics showed a much greater wicking distance (7–8 cm) than that observed for the untreated PET (2.35 cm). In the fixed assay time period, the dye solution traveled further for the treated PET samples, again, suggesting that the

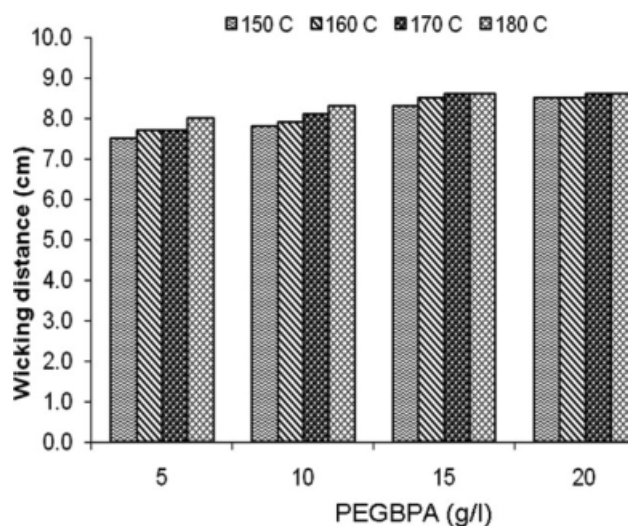


Figure 3 Effect of PEGBPA concentration and fixation temperature on the wicking distance of the resultant treated PET fabric.

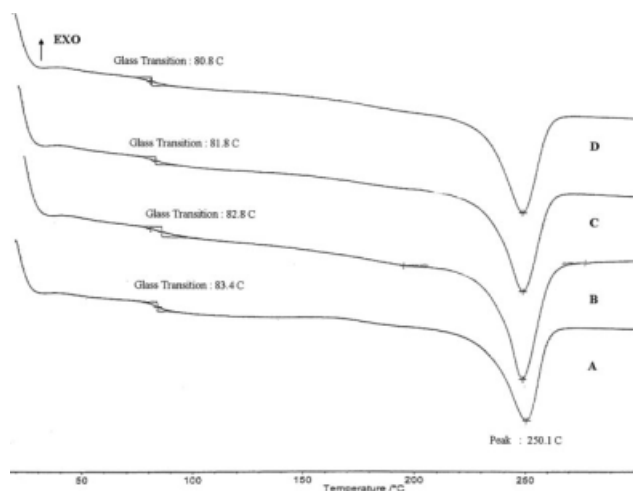


Figure 4 DSC thermograms of untreated PET (A), PET heat treated at 160°C (B), 170°C (C), and 180°C (D)

treated PET fabrics exhibited a significant improvement in wettability when compared with untreated PET, consistent with an improved hydrophilic nature. The improved wickability and moisture regain are indicative of the ability of the treated PET fabrics to transfer moisture, i.e., perspiration, away from the body, which would provide the wearer with comfort.

The effects of the fixation temperature on the subsequent wettability of the treated PET fabric were investigated and the results are also shown in Figures 2 and 3. The treatment temperature is found to play a dominant role in fixing PEGBPA onto the PET surface. The treatment temperature below 150°C results in a slight increase in the moisture

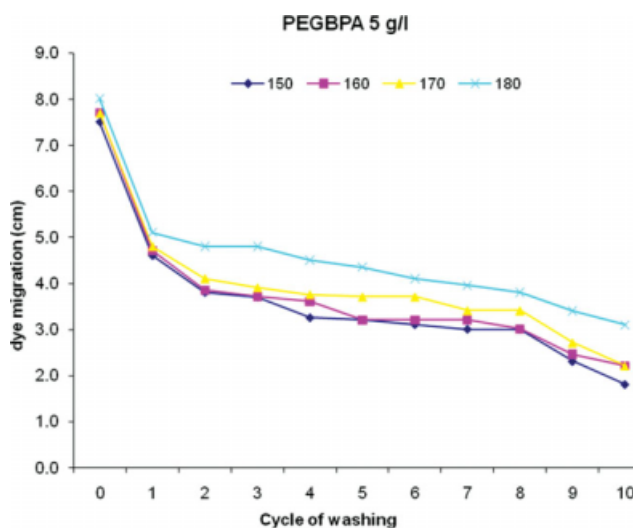


Figure 5 Plot of wash cycle against wicking distance: Samples from 5 g/L PEGBPA -PET fabrics with heat treatment temperatures of 150°C, 160°C, 170°C, and 180°C. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

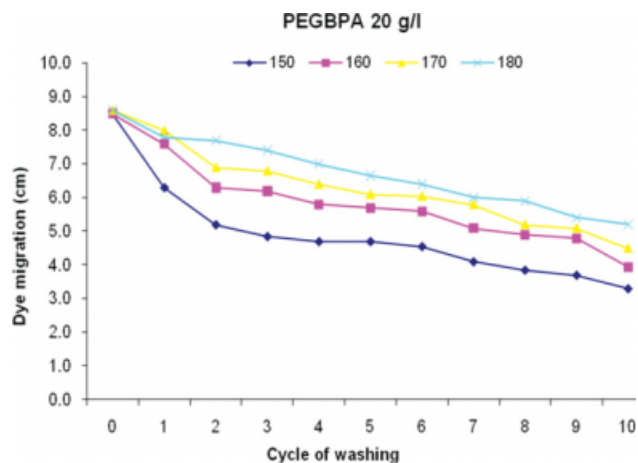


Figure 6 Plot of wash cycle against wicking distance: samples from 20 g/L PEGBPA-PET fabrics with heat treatment temperatures of 150°C, 160°C, 170°C, and 180°C. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

absorption ability over that seen with untreated PET fabrics. At the treatment temperature of 150°C and above the subsequent moisture absorption as well as the wicking distance of the treated PET fabrics significantly increases. The reason for this is believed to be that as the PET is thermoplastic, then heat treatment of the PET coated with PEGBPA (a compound having penetrating ability) will result in softening of the PET surface, then allowing penetration of the hydrophobic segment of PEGBPA into the PET substrate. By anchoring the hydrophobic moiety of PEGBPA into the PET surface and leaving the hydrophilic segment on the PET surface, a durable hydrophilic coating is attained.

Differential scanning calorimetry (DSC) was performed to study the thermal behavior of polyester

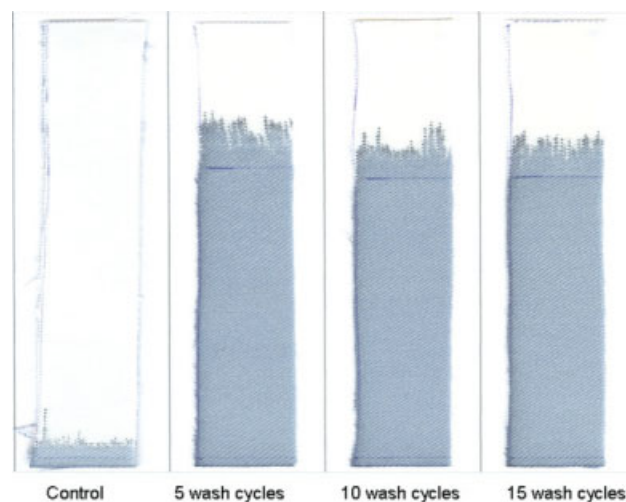


Figure 7 Photographs of fabric strips after durability test. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

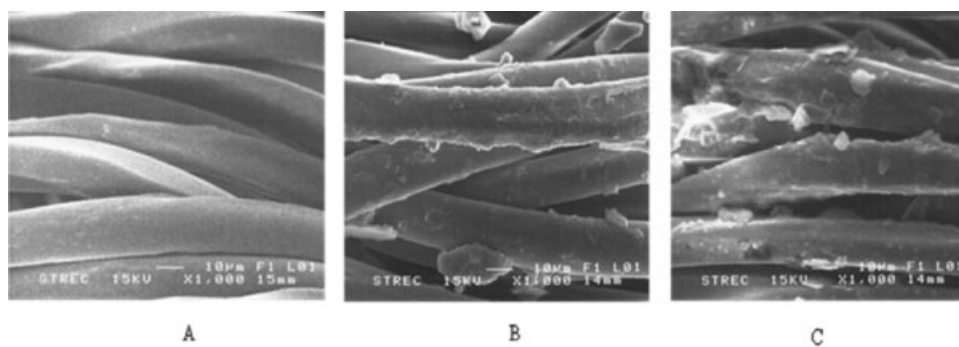


Figure 8 Representative SEM images of the untreated fiber (A) and treated (20 g/L PEGBPA coated and heat fixed) PET fiber surface before (B), and after (C) ten repeated washings.

heat treated at various temperatures. The glass transition temperatures (T_g), obtained from the DSC analyses, are indicated in Figure 4. The T_g of untreated PET at 83.4°C was relatively higher than that observed for the PEGBPA coated PET samples, which varied from 82.8°C to 80.8°C for the treated PET samples. The decrease in T_g values during heat treatment in the presence of PEGBPA suggests the formation of a less packed structure due to the penetration of the hydrophobic segment of the PEGBPA. As a consequence, heat-treated PET with PEGBPA is likely to soften faster upon heating; the higher the treatment temperature the lower the T_g value of treated fabric.

Durability evaluation

High fastness properties of any finishing agent on the textile surface are one of requirements from a commercial point of view. Here, the wash fastness of the PEGBPA coating on the PET fabric surface was evaluated using the standard washing test (AATCC test method 61-2001). Fabric samples were subjected to up to 10 repeated washings. It should be noted that 0 wash cycle represents rinsed fabric, which exhibits the highest wettability. The vertical wicking distance was measured after every wash cycle as a marker for hydrophilicity and thus coating integrity, and the results are graphically shown in

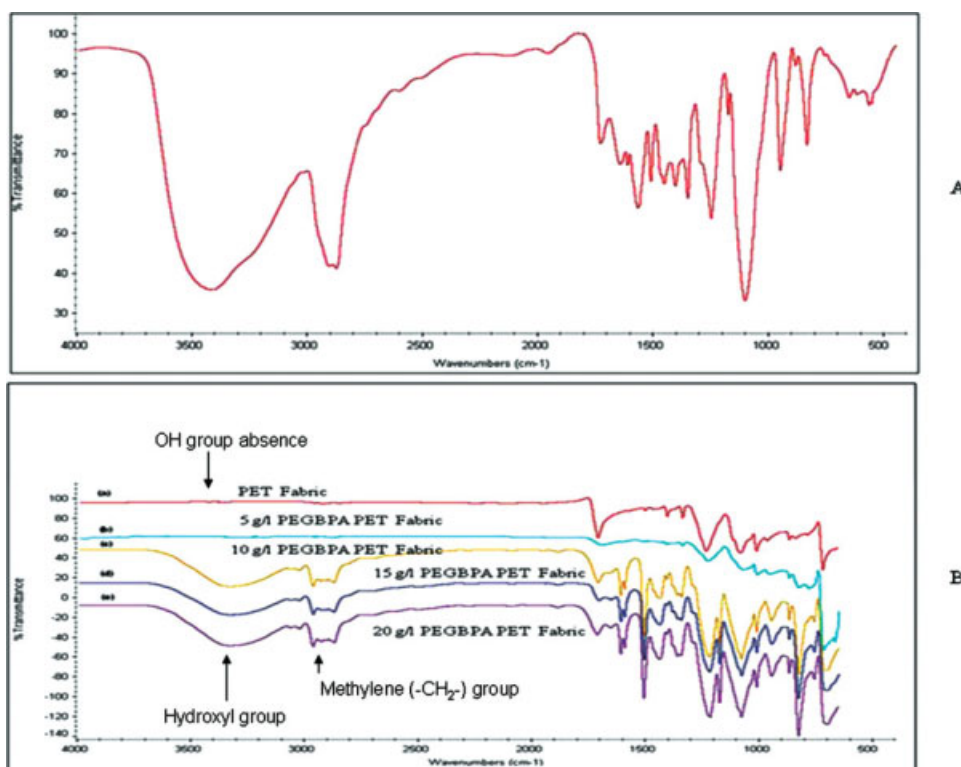


Figure 9 Representative IR spectrum of PEGBPA (A) and ATR/FTIR spectra (B) of PET fabrics treated with various PEGBPA concentrations. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Figures 5 and 6. Additionally, PET fabric strips after 5, 10, and 15 wash cycles were photographed. The photographic images are illustrated in Figure 7. For all the samples tested, which included the PEGBPA coatings attained at different application doses and at different subsequent heat treatments, the measured wicking distance decreased gradually with increasing numbers of wash cycles. For instance, PET fabric treated with 5 g/L of PEGBPA coating and fixed at 180°C, revealed a wicking distance before and after 10 wash-off cycles of 8.0 and 3.1 mm, respectively. Despite the gradual decrease with each washing cycle, the wicking distance of 5.6 mm obtained after 10 wash cycles in the best treated PET sample (20 g/L PEGBPA coating and 180°C fixation) is still markedly higher than that of 0.3 mm for the untreated PET fabric. In addition, the fiber surface morphology, when analyzed by SEM, was smooth for untreated PET fabrics and rough for PEGBPA treated PET both before and after ten washes (Fig. 8). Taking the rough surface morphology to represent the presence of PEGBPA on the fiber surface, the remaining rough surface after 10 wash cycles suggests the fastness property of PEGBPA on the PET surface. However, to confirm the actual existence of PEGBPA, the same samples were also analyzed by ATR/FT-IR spectroscopy, with representative spectra shown in Figure 9. The hydroxyl band found in the region of 3500–3300 cm^{-1} in case of the PEGBPA coated (treated) PET fabrics is indicative of the presence of PEGBPA. This absorption band remained with a strong intensity after the fabrics had been repeatedly washed for 10 cycles, indicating the durability of the PEGBPA coating. For this system, the adhesion of coating solely depends on the physical means, such as hydrophobic–hydrophobic interaction between the bisphenol A segment of PEGBPA and the aromatic segment of PET.

CONCLUSION

A durable hydrophilic property was conferred upon PET fabrics by coating with PEGBPA, a hydrophilic–

hydrophobic compound. After PEGBPA coating, the treated PET fabric was changed from a strong hydrophobic to a more hydrophilic nature, as evidenced by surface properties such as an increased moisture regain and wicking ability. Treatments of PET fabric with PEGBPA at a concentration of between 10 and 20 g/L PEGBPA, and a fixation temperature within the range of 160–180°C for 3 min were found to be sufficient to alter the surface property of the treated PET fabric from hydrophobic to hydrophilic. A standard wash fastness evaluation showed that the PEGBPA exhibited satisfactorily good adhesion on the PET surface and was capable of withstanding repeated washings. It was concluded that the adhesion of coating solely depended on the physical aspects, such as the hydrophobic–hydrophobic interaction between the bisphenol A segment of PEGBPA and the aromatic segment of PET.

Authors would like to thank National Science and Technology Development Agency, Thailand.

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