Antimicrobial Finishing of Regular and Modified Polyethylene Terephthalate Fabrics

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ABSTRACT: An effective two-stage method has been developed for imparting antimicrobial properties to regular polyethylene terephthalate (R-PET), polyethylene glycol modified polyethylene terephthalate (PEG-M-PET), R-PET/Cotton blend (R-PET/C) and PEG-M-PET/Cotton blend (PEG-M-PET/C) fabrics. The method consists of partial hydrolysis of the fabrics to create carboxylic groups in PET macromolecules followed by subsequent reaction with dimethylalkylbenzyl ammonium chloride (DMABAC) under alkaline conditions. The reaction conditions such as pH, reaction temperature and time, carboxylic content, and DMABAC concentration were studied. Characterization of the finished fabrics was carried out through scanning electron microscopy (SEM) and Fourier transform infrared spectra (FTIR). All the modified PET fabrics showed excellent antibacterial activity towards Gram-positive (*Bacillus mycoides*), Gram-negative (*Escherichia coli*), and nonfilamentous fungus (*Candida albicans*). The achieved antimicrobial functions on the PET fabrics are durable in repeated laundering processes. Even after laundering 10 times the fabrics could still provide more than 85% of its antimicrobial activity against *B. mycoides*, *E. coli*, and *C. albicans*. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 109: 942–950, 2008

Key words: polyester (PET) fabric; polyethylene glycol-modified polyester (PEG-M-PET) fabrics; PET/cotton blend; PEG-M-PET/cotton blend; hydrolysis; antimicrobial treatment; QAS; SEM; FTIR; salt exhaustion; salt uptake efficiency; zone of inhibition; antimicrobial properties

INTRODUCTION

Textile materials are good media for growth of micro-organisms, particularly the drug-resistance bacteria which have caused great concern to public health.[1–2] Therefore, antimicrobial treatment for textile materials is necessary for a variety of applications. Medical products are perhaps the largest application.

Polyester fibers are of considerable use in textile and other industries. In textile industry alone its volume consumed started to exceed the volume of cotton fibers. They offer a number of favorable properties, including ease of processing, reduced weight, plasticity, and, last but not least, low cost. Accordingly, the improvement of the antimicrobial properties of these fibers is important for a wide range of industrial applications including clothing, bedding, and interior materials for automobiles.[3,4]

More promising and widely used method for imparting bioactive features to PET fibers is the preliminary modification of ready-made articles via reaction in the polymer chain or by grafting of ionogenic and nonionogenic monomers, with the objective of increasing the content or creating on the fibers new functional groups, which are able to react with biocides.[5–11] Ito et al.[12] prepared insulin-immobilized PET by the hydrolysis of PET and the subsequent reaction with insulin. Kim et al.[13] and Huh et al.[14] have prepared PET coimmobilized with insulin and heparin,[13] and chitosan and quaternized chitosan[14] using oxygen plasma glow discharge treatment followed by acrylic acid grafting and subsequent reaction with insulin and heparin or chitosan and quaternized chitosan. A simple base hydrolysis process was used as a pretreatment process for PET granules to increase the adhesion of chitosan with these granules.[15] N-vinyl-2-pyrrolidone (NVP)[16] and 8-quinolinyl acrylate (QA)[17] were graft-polymerized on to the plasma-treated PET surface. Antibacterial assessment using *S. aureus* test indicated that *S. aureus* was restrained from growing in NVP-g-nonwoven PET. Moreover, the QA-grafted PET film showed high-growth inhibition of 91% for the same microorganism.

Several attempts to immobilize antimicrobial agents in PET fibers have so far not led to their practical utilization. This is, mainly, due to the absence of suitable and simple method for attaching biocides to the fibers. One of the effective ways of obtaining antimicrobial fibers and textile articles is modifying man-made fibers with quaternary ammonium

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compounds. These substrates have a broad spectrum of antimicrobial action with respect to both fungi and bacteria.[5] Quaternary ammonium salts (QAS) are readily soluble in water, they are adsorbed well by fibers, and the definite toxicity inherent to these compounds can be considerably reduced by including them into polymer structure. Immobilization of QAS on man-made fibers and fabrics is based on reaction with active functional groups (either present or specially created) in these materials.

In light of the abovementioned the present work aims at developing a novel, simple, and practically applicable functional finishing approach for imparting antimicrobial properties to PET fabrics. In this study, four types of polyester fabrics-regular polyethylene terephthalate (R-PET), polyethylene glycol modified PET (PEG-M-PET), R-PET/cotton (R-PET/C), and PEG-M-PET/cotton (PEG-M-PET/C), were surface-modified to improve their performances in biomedical applications. Finishing approach is based on partial hydrolysis of the fabrics to create carboxylic groups in PET macromolecules followed by subsequent reaction with quaternary ammonium compound under alkaline conditions. Moreover, some related finishing conditions is discussed. Characterization of the finished fabrics was carried out through scanning electron microscopy (SEM) and attenuated total reflection fourier transform IR (FTIR) spectroscopy. The growth inhibition of the microorganisms on the surface of finished fabrics was also examined.

EXPERIMENTAL

Materials

- *R*-PET, PEG-M-PET, *R*-PET/C (50/50), and PEG-M-PET/C (50/50) fabrics used throughout this study were in the form of filament woven fabric cloth made from filament yarns. They were kindly supplied by Misr polyester Co., Kafr EL-Dwar, Egypt. The fabrics were scoured at 80°C for 45 min. with solution containing 2 g/L nonionic detergent, washed with cooled water, squeezed, and finally air dried.
- Dimethylalkylbenzyl ammonium chloride C_{10}—C_{12} (DMABAC) used in this work was in the form of 50% aqueous solution, and was kindly supplied by NIIPAV, Volgodonck, Russia.
- Solutions of sodium hydroxide (A.R. grade) were made up in distilled water and stored in plastic bottles; concentrations were determined by titrations.
- *Microorganisms: Bacillus mycoides* (Gram positive bacterium), *Escherichia coli* (Gram negative bacterium), and *Candida albicans* (nonfilamentous fungus) were used for estimation of antimicrobial potency of control and treated samples. Microorganisms were obtained from the culture collection of the Department of Microbial Chemistry, Division of Genetic Engineering and Biotechnology, National Research Centre of Egypt.
- *Culture medium:* Modified nutrient agar medium was used and is composed of the following ingredients (g/L): peptone (10.0), beef extract (5.0), NaCl (5.0), and agar (20.0). The pH was adjusted to 6.8. This medium was sterilized for 20 min at 121°C under pressure.[18]

Methods

- Alkaline hydrolysis of PET fabrics was carried out using the method described by Shalaby et al.[19]
- *Fixation of DMABAC on hydrolyzed PET fabrics:* The treatment of hydrolyzed PET fabrics with DMABAC was carried out using a high-temperature high-pressure laboratory dyeing machine. Required amounts of DMABAC solutions were placed in stainless-steel bowls, hydrolyzed samples were immersed in the solutions, and the sealed bowls were rotated in a closed bath containing ethylene glycol at the desired temperature. The material:liquor ratio (M:L) was 50:1. The bath temperature increased at rate of 2°C/min. After the predetermined durations, the samples were removed from the bath, rinsed repeatedly with distilled water and allowed to dry in the open air.
  The initial and final concentrations of DMABAC solutions were determined by Recording Spectrophotometer (UV-Vis, 2401 Shimadzu) at λ_{max} = 208 nm.
- The salt exhaustion on the fabrics (E%) was calculated according to the following equation: $E(\%) = [C_1 - C_2/C_1] \times 100$, where C_1 and C_2 are the concentrations (g/L) of QAS solutions before and after treatments, respectively.
- The salt uptake on PET fabrics (SUT) was calculated as follows:
  SUT (g./g. fabric) = $[V(C_1 - C_2)/W]$, where C_1 and C_2 are the concentrations (g/L) of QAS solutions before and after treatments, respectively, V is the volume of QAS solutions (cm^3) used in treatment, and W is the weight of PET fabric.
- Finishing efficiency of PET fabrics with QAS solutions (FE%) was calculated as follows: $FE(\%) = [A/B] \times 100$, where A is the salt uptake on PET sample (mg/g. Fabric), and B is the amount of salt equivalent to the carboxylic content (mg/g. fabric) in PET fabric before treatment.

Analysis

- Antimicrobial potency by diffusion was quantified by measurement in millimeters of the width of the

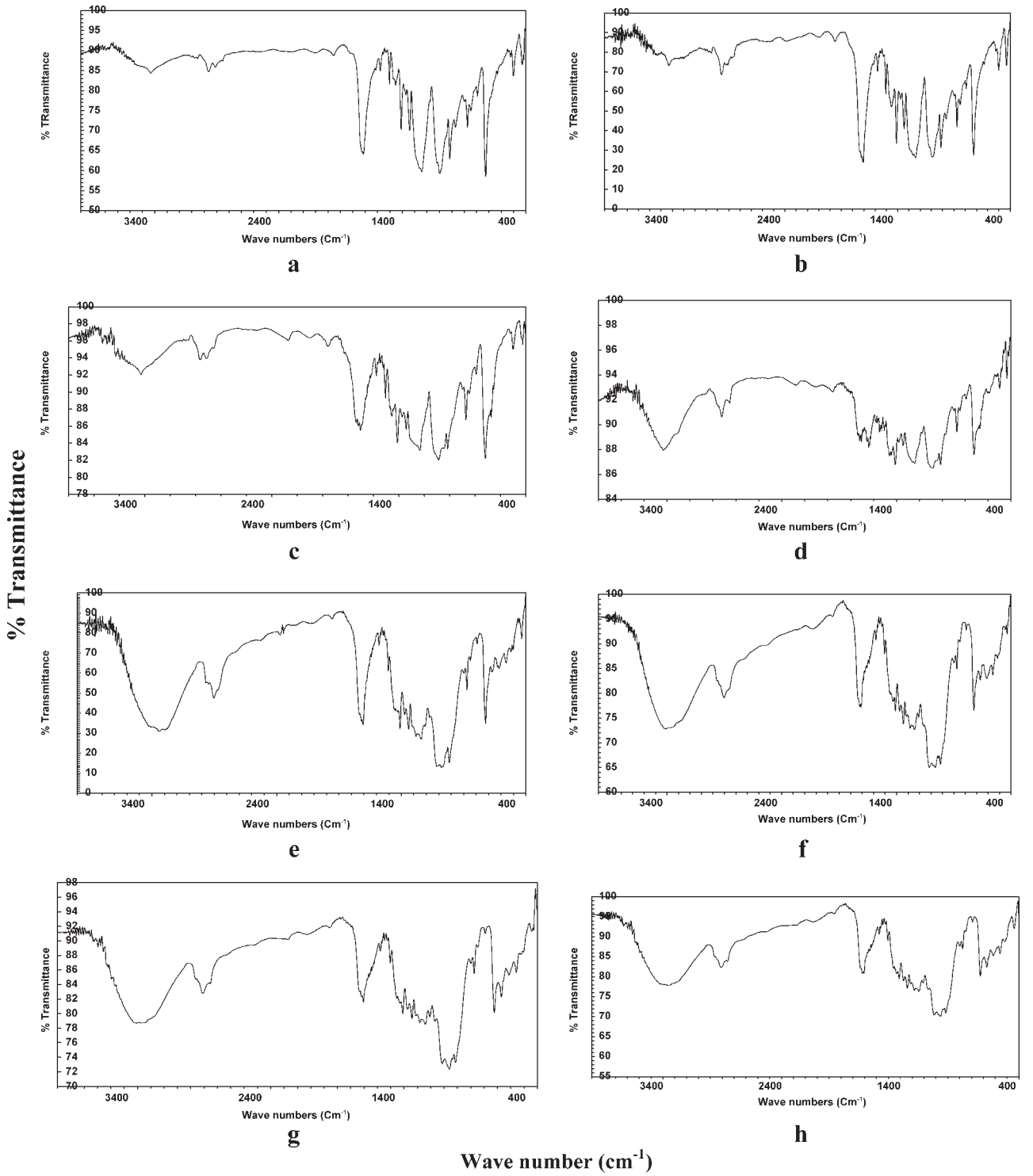


Figure 1 FTIR Spectra for hydrolyzed and treated with DMABAC R-PET and M-PET fabrics and their blends with cotton. **(a)** Hydrolyzed R-PET (Weight loss% = 25), **(**b) Hydrolyzed R-PET (Weight loss% = 25) Treated with DMABAC, **(**c) Hydrolyzed PEG-M- PET (Weight loss% = 25), **(**d) Hydrolyzed PEG-M-PET (Weight loss% = 25) Treated with DMABAC, **(**e) Hydrolyzed R-PET/C blend (Weight loss% = 10), **(**f) Hydrolyzed R-PET/C Blend (Weight loss% = 10) Treated with DMABAC, (g) Hydrolyzed PEG-M-PET/C Blend (Weight loss% = 10), **(**h) Hydrolyzed PEG-M-PET/C Blend (Weight loss% = 10) Treated with DMABMA.

zone of growth inhibition around the sample according to AATCC standard test method.[20]

- Carboxylic content was determined according to the method described in Ref. 21.
- A JEOL-Model JSM T20 scanning electron microscope (SEM) operating at 19 KVwas used to obtain photomicrographs of fibers surfaces.

- The chemical structure was determined using the Fourier transformation infrared (FTIR) spectrometer, model NEXUS 670, NICOLET USA. The measurements were carried in spectral range from 4000 to 400 cm^{-1}, by using resolution 4 cm^{-1} KBr disc technique was applied.

RESULTS AND DISCUSSION

It has been mentioned above that several attempts to immobilize antimicrobial agents in PET fibers have so for not led to their practical utilization. This is, mainly, due to the absence of suitable and simple method for attaching biocides to the fibers. In the present work experiments were designed to develop an efficient and generally applicable method for imparting PET fabrics antimicrobial properties, as follows:

1. The PET samples were introduced into thermostated dimethylalkyl benzyl ammonium chloride (DMABAC) water solution. The treatment was allowed to proceed at the required temperature for the desired reaction time. It was observed that no salt uptake on PET fabrics took place when PET/DMABAC/H_2O system was used irrespective of the conditions of the treatment.
2. The PET samples were treated with NaOH solutions at a temperature higher than T_g of the polymer for creation of carboxylic groups, and then treated with DMABAC water solutions under the same conditions indicated in (1). It was observed that there was a salt uptake on the PET fabrics; a point which signifies the role of alkaline treatment of the samples prior to reaction with QAS. A part of this salt uptake was still existing even after several extraction cycles of the samples with water which is the solvent of the salt. The salt uptake is unequivocally due to inclusion of QAS within the PET samples.

It would be of interest to find out whether the coating or immobilization of DMABAC on hydrolyzed fabrics is through physical or chemical interactions. Therefore, characterization of the so finished PET fabrics was carried out through scanning electron microscopy (SEM), and FTIR microscopy.

SEM micrographs showing the surface morphologies of PET samples after the hydrolysis reaction and the samples with immobilized DMABAC layer on the surface are presented in Figure 1. From Figure 1(a,c,e,g), it is seen that after hydrolysis, a few pits appeared on the surfaces and the latter became much rougher. The rougher surfaces can be expected to improve the immobilization of DMABAC on the hydrolyzed samples. Figure 1(b,d,f,h) shows the QAS layer immobilized on the PET fabrics. The surfaces of the samples were covered by salt and the surfaces morphology of QAS layer immobilized on different PET samples did not show a noticeable difference.

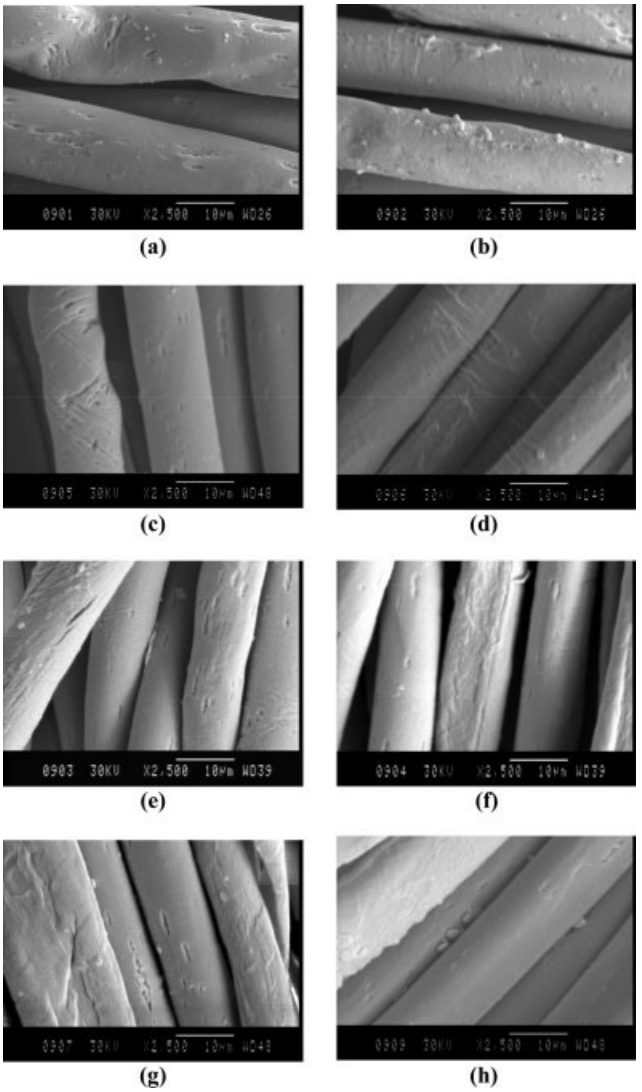


Figure 2 Scanning electron micrographs for hydrolyzed and treated with DMABAC R-PET and M-PET fabrics and their blends with cotton. (a) Hydrolyzed R-PET (Weight loss% = 25), (b) Hydrolyzed R-PET (Weight loss% = 25) Treated with DMABAC, (c) Hydrolyzed PEG-M-PET (Weight loss% = 25), (d) Hydrolyzed PEG-M-PET (Weight loss% = 25) Treated with DMABAC, (e) Hydrolyzed R-PET/C blend (Weight loss% = 10), (f) Hydrolyzed R-PET/C Blend (Weight loss% = 10) Treated with DMABAC, (g) Hydrolyzed PEG-M-PET/C Blend (Weight loss% = 10), (h) Hydrolyzed PEG-M-PET/C Blend (Weight loss% = 10) Treated with DMABMA.

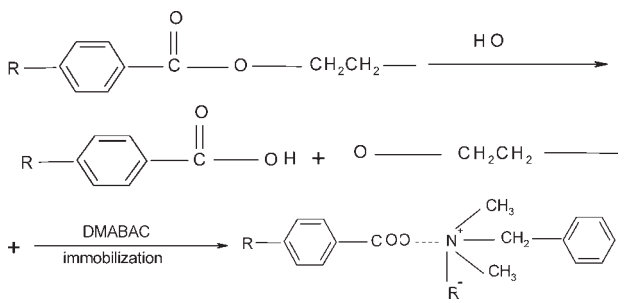
Figure 2 shows the FTIR spectra of hydrolyzed PET samples before (a,c,e,g) and after (b,d,f,h) treatment with DMABAC in the region from 400 $\sim$ 4000 cm^{-1}. From this figure, it is clear, that the C=O stretching vibrational band, in case of hydrolyzed fabrics, shows a frequency shift in the following order: R-PET (1713) > R-PET/C (1708) >PEG-M-

TABLE I
Relative Absorbance of Hydrolyzed PET Fabrics Before and After Treatment with DMABAC

Sample	Relative absorbance (1702–1713 cm^{-1}/873 cm^{-1})
R-PET	3.93
R-PET-tr. DMABAC[a]	4.10
PEG-M-PET	2.55
PEG-M-PET-tr. DMABAC[a]	2.82
R-PET/C	2.94
R-PET/C-tr. DMABAC[a]	3.46
PEG-M-PET/C	2.86
PEG-M-PET/C- tr. DMABAC[a]	3.57

[a] Treated with DMABAC.

PET/C (1704) > PEG-M-PET (1703). The shift towards lower wave number indicates that the hydrogen bonding intermolecular interaction in PEG-M-PET and PEG-M-PET/C is less than of R-PET and R-PET/C. After treatment of the abovementioned samples with DMABAC, the C=O stretching vibrational band shifts, to some extent, towards higher wave number. Based on FTIR spectra shown in Figure 2 the mean values of relative absorbance (band intensity of C=O of carboxylic group relative to absorption peak at 873 cm^{-1}) was calculated by applying base line method described by Heigl et al.[22] (Table I). It is clear, from Table I, that treatment of hydrolyzed PET samples with DMABAC leads to a significant increase of the value of relative intensity irrespective of the type of treated PET fabric. These results, therefore, suggested the formation of salt structure of ($R\text{-}^{+}NH_4.....^{-}OOC$) between the hydrolyzed PET fabrics and the immobilized DMABAC at the interface, probably attributable to the acid–base ionic binding between the carboxyl groups on the hydrolyzed PET fabrics and the quaternary ammonium groups in DMABAC. Therefore the hydrolysis process of the PET fabrics and the immobilization of DMABAC can be schematically proposed as follows:



Based on the abovementioned, one can conclude that, the immobilization of dimethylalkyl benzyl ammonium chloride on the hydrolyzed PET fabrics is at least partly effected through chemical interactions. Similar mechanism was proposed for the immobilization of chitosan on the hydrolyzed nylon 66 or PET granules.[15]

Presented below are the different factors that affect the antimicrobial finishing of regular and modified polyethyleneterephthalate and its blends with cotton, with dimethylalkyl benzyl ammonium chloride (DMABAC).

Effect of pH

The effect of pH value of the treatment solution on the salt uptake of DMABAC on the PET fabrics and finishing efficiency was studied under three different values, mainly, 4, 7, and 11. The results obtained are listed in Table II. It can be seen that, acidic treatment conditions did not result in significant salt uptake of QAS on the fabrics, irrespective of the type of PET used. In contrast, the higher pH solution led to a higher exhaustion and higher efficiency, since the cationic DMABAC is more attractive to the negatively charged carboxylate groups under the basic conditions. Because of the ionic interactions the DMABAC was quickly absorbed onto and then diffused into the PET fabrics. However, a higher salt uptake was observed in the case of PEG-M-PET and PEG-M-PET/C fabrics. This may be due to the higher susceptibility of these fabrics to finishing conditions, since it contains PEG moiety in the polymer chain itself. The difference in internal structure of PEG-M-PET in comparison with the *R*-PET would also be responsible for this behavior.

Effect of finishing temperature

The effect of finishing temperature on the DMABAC uptake on the PET fabrics and finishing efficiency was examined. The obtained results (Table II) show that, irrespective of the type of treated PET fabric, the salt uptake was increased as the temperature was increased from 70 to 90°C. The increase of salt uptake was more significant in the temperature range around the T_g of the PET fiber. At 90°C the exhaustion of DMABAC reached its higher rates at the chosen conditions. The results obtained show the same trend but the values are much greater in the case of PEG-M-PET and PEG-M-PET/C fabrics. Therefore, 90°C was selected as the temperature used in subsequent experiments since it could be easily controlled in water bath shaker. Above T_g the amorphous regions in the PET will provide more volume to accept the salt. In addition, a swelling effect resulted from the higher temperatures and basic conditions should facilitate diffusion of DMABAC in PET substrates. Consequently, there would be an increased amount of the salt present within

TABLE II
Effect of pH, Finishing Temperature, and Finishing Time on Salt Uptake (SUT) and Finishing Efficiency (FE)

	R-PET(1)		PEG-M-PET(2)		R-PET/C(3)		PEG-M-PET/C(4)	
pH[a]	SUT (mg/ g.fabrics) (1)	FE (%) (2)	SUT (mg/ g. fabrics) (3)	FE (%) (4)	SUT (mg/ g.fabrics) (5)	FE (%) (6)	SUT (mg/ g. fabrics) (7)	FE (%) (8)
4	0.5	0.8	2.0	2.4	2.2	3.3	2.5	3.6
7	4.5	7.5	6.6	8.0	7.5	11.7	8.2	11.6
11	9.1	15.2	10.0	16.0	11.3	17.6	13.8	18.6
Finishing temperature (°C)[b]	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
70	3.3	5.5	3.9	4.8	4.2	6.5	5.1	7.4
80	5.2	8.6	8.2	10.0	9.5	14.8	11.5	16.7
90	9.1	15.2	10.0	16.4	11.3	17.6	13.8	18.7
Finishing time (min)[c]	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
20	2.3	3.8	2.5	3.1	2.8	4.3	3.5	5.1
40	3.6	6.0	4.0	4.9	4.5	7.0	5.5	3.7
60	9.1	15.2	10.0	16.4	11.3	17.6	13.8	18.7

Carboxylic content (meq/100 g fabric): (1) 19. 8; (2) 27.0; (3) 21.2; (4) 22.6.
[a] [DMABAC], 0.11%; Temperature, 90°C; Time, 60 min; M : L, 1 : 50.
[b] [DMABAC], 0.11%; pH, 11; Time, 60 min; M : L, 1 : 50.
[c] [DMABAC], 0.11%; pH, 11; Temperature, 90°C; M : L, 1 : 50.

the fabrics, which provide good washing durability of the antimicrobial efficacy.

Effect of finishing time

The majority of PET structure is crystalline, which is tightly packed and difficult to penetrate for chemical reagents. Thus, the finishing time is quite critical to the exhaustion of DMABAC on PET. The effect of the treatment time on the salt uptake is listed in Table II. It is clear that the increase in reaction time from 20 to 60 min is accompanied by a substantial increase in the salt exhaustion and salt uptake. As the treatment time increased, the diffusion of DMABAC on the PET fabrics could be improved significantly, which will result in high durability of the antibacterial functions.

Effect of weight loss

Stemming from the fact that the present finishing method is based on alkaline treatment of the PET fabrics before treatment with DMABAC; keeping in mind that weight loss % obtained during the hydrolysis reaction depends upon alkali concentration, reaction duration and reaction temperature; aiming to decrease the consumption of energy and alkali, it would be of great importance to find out the optimum weight loss, which paves the way for imparting high antimicrobial activity to PET samples. It is seen from the data listed in Table III that the increase in weight loss % from 11 to 25% in the case of R-PET and PEG-M-PET and from 10 to 20% in the case of blends is not accompanied by a noticeable increase neither in the salt uptake on the fabrics nor in the antimicrobial activity of the finished samples (Table V). Vainburg et al.[5] have declared that a stable antimicrobial effect on cellulosic textile materials is obtained at a low DMABAC content: 4 to 6 mg of preparation per gram of fabric. Based on the abovementioned one can conclude that a weight loss of 5–7.5% quiet enough for imparting high biological activity to PET fabrics, irrespective of the type of sample used.

TABLE III
Effect of Weight Loss (%) on Salt Uptake (SUT), and Finishing Efficiency (FE)

Fabric	Weight loss (%)		SUT (mg/g fabrics)	FE (%)
R-PET	Unhydrolyzed		0.0	0.0
	11.0	(1)	7.4	23.5
	25.0	(2)	9.1	15.2
PEG-M-PET	Unhydrolyzed		0.0	0.0
	10.0	(3)	8.5	21.2
	25.0	(4)	10.0	16.0
R-PET/C	Unhydrolyzed		0.0	0.0
	10.0	(5)	11.3	17.6
	20.0	(6)	16.0	19.0
PEG-M-PET/C	Unhydrolyzed		0.0	0.0
	10.0	(7)	13.8	18.7
	20.0	(8)	16.5	18.8

Reaction conditions: [DMABAC], 0.11%; Temperature, 90°C; Time, 60 min; M : L, 1 : 50. Carboxylic content (meq/100 g. fabric): (1) 10.4; (2) 19.8; (3) 13.2; (4) 27.0; (5) 21.2; (6) 27.7; (7) 22.6; (8) 28.9.

TABLE IV
Effect of [DMABAC] on Salt uptake (SUT) and Finishing Efficiency (FE)

	R-PET (1)		PEG-M-PET (2)		R-PET/C (3)		PEG-M-PET/C (4)	
[DMABAC] (%) (a)	SUT (mg/g. fabrics)	FE (%)	SUT (mg/g. fabrics)	FE (%)	SUT (mg/g. fabrics)	FE (%)	SUT (mg/g. fabrics)	FE (%)
0.00	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
0.07	1.40	2.4	1.50	2.0	1.7	2.6	1.7	2.6
0.11	9.11	15.2	10.0	16.0	11.3	17.6	13.6	18.6
0.17	10.0	16.6	13.0	15.8	16.0	24.8	20.0	29.1
0.58	48.0	79.6	57.3	70.3	51.0	79.1	60.0	87.8
1.67	58.4	97.0	71.0	86.5	62.0	97.2	65.0	97.2

Reaction conditions: pH, 11; Temperature, 90°C; Time, 60 min; M : L, 1 : 50. Carboxylic content (meq/100 g. fabric): (1) 19.8; (2) 27.0; (3) 21.2; (4) 22.6.

Effect of dimethylalkyl benzyl ammonium chloride concentration

To study the effect of DMABAC concentration on the finishing reaction of the PET fabrics, the exhaustion of the QAS on the fabrics was examined with varied concentrations of the salt under the optimized pH condition, temperature, and time based on the previous findings. Table IV displays the amount of the salt uptake on the fabrics and finishing efficiency corresponding to different concentrations of the QAS. It was observed that the salt uptake on the fabrics and finishing efficiency increased with an increase in DMABAC concentration. Moreover, at a concentration of the salt equal to 0.58% in reaction medium, there is a significant increase in the salt uptake and finishing efficiency. However, further increase in the QAS concentration has a little effect on the salt uptake, especially in the case of PEG-M-PET/C fabrics. At any concentration of the salt in reaction medium, the values of the salt uptake on the fabrics for PEG-M-PET/C and PEG-M-PET are always higher than those for R-PET/C and R-PET.

Antimicrobial properties

The antimicrobial activity of hydrolyzed and treated with DMABAC PET fabrics against *B. mycoides, E. coli*, and *C. albicans* was investigated. The activity by diffusion is quantified by the measurement in millimeters of the width of the zone of inhibition around the sample. Tables V and VI indicate the antimicrobial activity of hydrolyzed and treated with DMABAC PET fabrics. It is seen from the data listed in these tables that all PET fabrics bounded with QAS showed high antimicrobial activities against the

TABLE V
Effect of Weight Loss (%) on Antimicrobial Activity of Hydrolyzed and Treated with DMABAC PET Fabrics

Tested fabric	Weight loss (%)	Inhibition zone diameter (mm) in case of tested microbes		
		B. mycoides	*E. coli*	*C. albicans*
R-PET	0.0	0.0	0.0	0.0
	7.5	14	13	13
	14.0	13	13	12
	22.0	13	13	12
PEG-M-PET	0.0	0.0	0.0	0.0
	11.0	14	14	13
	21.0	13	13	12
	30.0	13	14	12
R-PET/C	0.0	0.0	0.0	0.0
	5.0	16	18	14
	8.5	15	16	13
	11.0	16	17	15
PEG-M-PET/C	0.0	0.0	0.0	0.0
	4.5	16	19	14
	70	15	18	14
	11.0	16	18	16

Reaction Conditions: [DMABAC], 1.67%; pH, 11; Temperature, 90°C; Time, 60 min; M : L, 1 : 50.

TABLE VI
Effect of DMABAC Concentration on Antimicrobial Activity of Hydrolyzed and treated with DMABAC PET Fabrics

Tested fabric	Tested microbes	Inhibition zone diameter (mm) at [DMABAC] (%)		
		0.17	0.65	1.67
R-PET (1)	*B .mycoides*	13	14	14
	E. coli	13	13	14
	C. albicans	12	12	12
PEG-M-PET (2)	*B. mycoides*	13	14	14
	E. coli	12	13	13
	C. albicans	12	12	12
R-PET/C (3)	*B. mycoides*	16	17	18
	E. coli	16	17	17
	C. albicans	14	14	16
PEG-M-PET/C (4)	*B. mycoides*	16	16	17
	E .coli	17	18	19
	C. albicans	15	15	17

Reaction Conditions: pH, 11; Temperature, 90°C; Time, 60 min; M : L, 1 : 50 Carboxylic content (meq/100 g. fabric): (1) 19.8; (2) 27.0; (3) 21.2; (4) 22.6.

previously mentioned three microorganisms. In fact, the inhibition zones for all tested PET samples are significant, whereas it is null for all the unhydrolyzed ones. Significant differences were found between PET and PET/C fabrics. Diameters of inhibition zones are ranging between 12 and 13 mm are case of R-PET and PEG-M-PET, in contrast with 16–18 mm in case of PET/C blends. The most striking structural difference between PET/C blends and PET is the higher hydrophilicity of PET/C blend fabrics, which might be the key factor for the difference in antimicrobial functions. When the tested samples were placed on the culture medium inoculated with microorganism, the liquid could diffuse into its inner parts, and thus both of the inner and surface DMABAC could contribute to the killing power of the samples in the antimicrobial test. The hydrophobic feature of the PET fabrics prevents good contact between the culture medium inoculated with the microorganisms and the surface of the fabrics. Whereas only the surface DMABAC on the materials can directly contact microorganisms and provide the antimicrobial properties, the DMABAC inside of the fabrics cannot make contact with the microorganisms and will require time to migrate to the surface areas from inside after the outside QAS is consumed.

Table VII shows the extent of antimicrobial functions demonstrated on the four finished samples treated with DMABAC and the washing durability of the fabrics subjected to 1, 5, and 10 repeated Launder-Ometer washing tests. It is seen from these data that washing durability of the functions exceeds more than 10 Launder-Ometer washes. Although the functions on the treated fabrics were reduced to certain level after 10 Launder-Ometer washes, the fabrics treated with the concentrations of 0.65 and 1.67% DMABAC could still provide more than 85% of its antimicrobial activity against *B. mycoides*, *E. coli*, and *C. albicans*. These results show that, regardless of the finishing concentrations used in the study, the existence of DMABAC salt on PET fabrics will provide antimicrobial activities.

In this concern Buchenska et al.,[23] reported that drug-loaded fibers prepared by incorporation of carboxylic groups into fibers by poly (acrylic acid) grafting polymerization followed by impregnation of the fibers with cephalosporins were bioactive against gram-positive and gram-negative bacteria. In our study we demonstrated that the modified polyethylene terephthalate (PET) fabrics prepared by the reaction with QAS (DMABAC) showed excellent antifungal activity towards *C. albicans* besides its gram-positive and gram-negative antibacterial activity towards *B. mycoides*, and *E. coli*, respectively. In addition the prepared fabrics could still be more than 85% of its antimicrobial activities against the previously mentioned strains after laundering 10 times.

TABLE VII
Effect of DMABAC Concentration on Antimicrobial Activity

	Antimicrobial activity (%)															
	B. mycoides (Gram +ve)						*E. coli* (Gram –ve)						*C. albicans* (non filamentous fungi)			
	0.65%[a]			1.67%[a]			0.65%[a]			1.67%[a]			0.65%[a]			1.67%[a]
Fabrics	1*	5*	10*	1*	5*	10*	1*	5*	10*	1*	5*	10*	1*	5*	10*	1*	5*	10*
R-PET	100	92.9	92.9	92.9	92.9	92.9	92.3	92.3	84.6	92.8	85.7	85.7	100	91.7	91.7	100	100	100
PEG-M-PET	100	92.9	92.9	92.9	92.9	92.9	92.3	92.3	84.6	92.3	92.3	92.3	100	100	91.7	100	100	100
R-PET/C	94.1	82.3	88.3	94.4	88.9	88.9	94.1	88.2	82.3	100	94.1	94.1	100	100	92.9	93.8	87.5	87.5
PEG-M-PET/C	100	93.8	93.8	100	94.1	94.1	100	100	94.4	94.7	94.7	94.7	100	93.3	93.3	94.1	88.2	88.2

Reaction conditions: pH, 11; Temperature, 90°C; Time, 60 min; M : L, 1 : 50.
[a] Concentration of DMABAC solution.
* After one, five, and ten times Launder–Ometer washings; AATCC Test method (61-1989).

CONCLUSIONS

A simple, efficient, and practically applicable functional finishing approach for imparting antimicrobial properties to R-PET, PEG-M-PET, R-PET/C, and PEG-M-PET/C fabrics was developed. This finishing approach is based on partial hydrolysis of the fabrics to create carboxylic groups in PET followed by subsequent reaction with DMABAC under alkaline conditions. The optimum reaction conditions were determined. The finished fabrics were characterized by SEM and FTIR. All PET fabrics bounded with DMABAC showed high antimicrobial activities against *B. mycoides*, *E. coli*, and *C. albicans*. The achieved antimicrobial functions on the PET fabrics are durable in repeated laundering processes. After 10 washes the fabrics could still provide more than 85% of its antimicrobial activity against *B. mycoides*, *E. coli*, and *C. albicans*.

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