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Unusual polymerization of 3-(trimethoxysilyl)-propyldimethyloctadecyl ammonium chloride on PET substrates

Samiha Mohamed Abo El Ola[a], Richard Kotek[b,*], W. Curtis White[c], John Allan Reeve[c], Peter Hauser[b], Joon Ho Kim[d]

[a]Textile Research Division, National Research Center, Dokki, Cairo, Egypt
[b]College of Textiles, Textile Engineering Chemistry and Science, North Carolina State University, Box 8301, Raleigh, NC 27695-8301, USA
[c]AEGIS ENVIRONMENTS, 2525 Washington Ave., Suite 500, Midland, MI 48642-4619, USA
[d]School of Textiles, Yeungnam University, 214-1 Daedong, Kyongsan 712-749, South Korea

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Abstract

3-(Trimethoxysilyl)-propyldimethyloctadecyl ammonium chloride (AEM 5700) is a trifunctional monomer that can be polymerized under acidic conditions at temperatures greater than 100 °C. Under such conditions, coatings based on AEM 5700 formed on PET substrates are relatively hydrophobic and exhibit rewetting times higher than 100 min. Hydrophilic coatings exhibiting a rewetting time of 2.8 min can be also formed when AEM 5700 is initially hydrolyzed under alkaline conditions and then polymerized under acidic conditions. This effect could be related to the accessibility of unreacted hydroxyl groups and quaternary ammonium groups for interaction with water. Excellent antimicrobial action was demonstrated for all coatings based on AEM 5700 on PET substrates. Polyester fabrics having AEM 5700 coating can be dyed with direct dyes. The loss of hydrophilic properties for AEM 5700 coatings is observed after dyeing with direct dyes.

Keywords: Silane polymerization; Trimethoxysilane; Ammonium salts

1. Introduction

Demand for antimicrobial fabrics in domestic and international markets has grown significantly in recent years because of public awareness of the potential threat of spreading diseases [1,2]. Bio-protective clothing such as medical, protective garments, and institutional and hygienic textiles are the main applications of antimicrobial fabrics [2].

Silanes have been well published on and their associative and bonding capacities to themselves and to other surfaces has been studied by many researchers. It was determined that antimicrobial organofunctional silanes could be chemically bound to receptive substrates such as cellulose, by what were believed to be Si–O linkage. The method was described as orienting the organofunctional silane in such a way that hydrolysable groups on the silicon atom were hydrolyzed to silanols formed chemical bonds with each other and the substrate. The resultant surface modification, when an antimicrobial moiety such as quaternary nitrogen was included, allowed the antimicrobial to be oriented away from the surface [3]. The attachment of this chemical to surfaces appears to involve two processes. First and most important is the very rapid process which coats the substrate with cationic species one molecule deep. This is an ion exchange process by which the cation of silane quaternary ammonium compound replaces protons from water on the surface. It has long been known that most surfaces in contact with water generate negative electrical charges at the interface between the water and the surface (even on surfaces which contain no functionality). The second process is unique to materials such as silane quaternary ammonium compounds, which have silicon functionality, enabling them to polymerize after they have coated the surfaces and to become almost irremovable from these surfaces. Covalent bonding to that surface may also occur, and it is also possible to have intermolecular polymerization [4]. It is

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* Corresponding author. Tel.: +1-919-515-6585; fax: +1-919-515-6532.
E-mail address: rkotek@unity.ncsu.edu (R. Kotek).

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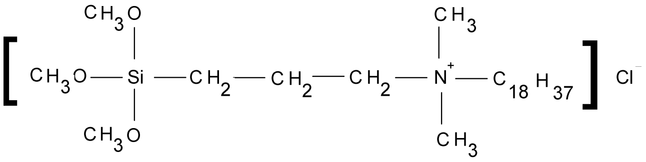

Fig. 1. Chemical structure of 3-(trimethoxysilyl)-propyldimethyloctadecyl ammonium chloride.

important to mention that coatings based on silicone polymers having Si–O–Si units have low glass transition temperature and surface tension.

The specific antimicrobial utility of 3-(trimethoxysilyl)-propyldimethyloctadecyl ammonium chloride (Fig. 1) is well described [5–9]. The compound is effective against Gram positive and Gram-negative bacteria, fungi, algae, and yeast. In most cases, antimicrobial action is usually achieved on contact in the presence of moisture.

Non-microbial trialkoxysilanes:

$R'-Si(OR)_3$

have also been extensively studied because they are useful precursors for the sol–gel polymerization processes [10,11] and organic–inorganic (O–I) hybrid materials [12,13]. Recent investigations have shown [14] that these monomers undergo not only intermolecular condensation but also excessive cyclization that lead to the formation of cage-like clusters. The resulting polymers, namely polysilsesquioxanes are therefore composed of random branched polymers, ladder polymers and also polyhedral products—'cages' [15,16]. It is important to mention that Izawa et al. [17] developed two-dimensional correlation gel permeation chromatography to study the polymerization of alkyltriethoxysilanes.

Unfortunately, little published information is available on the mode of action of trialkoxysilanes or silicon polymers on polymer films and fibrous surfaces. During the last decade, only Bereck [18–23] and Lautenschlager et al. [24,25] studied silicone polymers as softening and finishing agents for textiles. Most recently, Mahltig and Boettcher [26] reported the preparation of water-repellent materials based on 3-glycidoxypropyl triethoxysilane and fluorine containing silanes.

In this paper, a simple technique permitting the characterization of coating formation after polymerization of 3-(trimethoxysilyl)-propyldimethyloctadecyl ammonium chloride on poly(ethylene terephthalate) (PET) fabrics and films is reported. We have also studied the effect of polymerization temperature and pH on the uniformity of the coating as well as the effect of coating on dyeing with direct dyestuffs. Results of microbiological analysis of PET fabrics treated with 3-(trimethoxysilyl)-propyldimethyloctadecyl ammonium chloride under various conditions are also reported.

2. Experimental

2.1. Materials

3-(Trimethoxysilyl)-propyldimethyloctadecyl ammonium chloride (AEM 5700, see structure in Fig. 1) from AEGIS ENVIROMENTS was provided as a 42% solution in methanol. Glacial acetic acid and sodium carbonate were used as received.

Style 720, texturized Dacron 56T double knit jersey fabric from Testfabric Inc. was used in this study as a substrate for polymerization of AEM 5700. Mylar film substrates were also used when the surface characteristics of AEM 5700 coatings was investigated with atomic force microscopy (AFM).

C.I. Direct Blue 80 (C.I. 24315) was used in this study. The chemical structure of this dyestuff is shown in Fig. 2.

2.2. Polymerization technique

In order to remove any residual amount of finish, polyester fabric was washed with hot tap water (55–60 °C) for 15 min, then dried at 70 °C for 10 min. Washed material was then rinsed with 50% (v/v) isopropyl alcohol followed by final rinsing with distilled water before drying at 70 °C for 10 min.

In a typical polymerization experiment, a 1% (wt/wt) solution of 3-(trimethoxysilyl)-propyldimethyloctadecyl ammonium chloride (AEM 57000) was prepared in distilled water and pH was adjusted to 4 with glacial acetic acid at ambient temperature. Clean, finish free polyester fabric was placed in the solution of AEM 5700 and processed trough Werner Mathis AG, type HVF 24489 padder. Wet pick up was about 100% and was calculated from Eq. (1).

$$\text{wpu} = \frac{w_f - w_o}{w_o} 100\% \qquad (1)$$

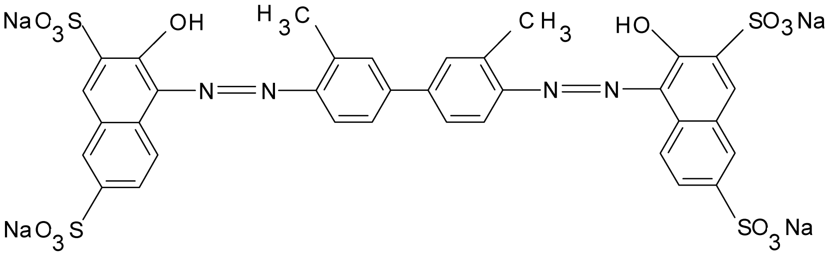

Fig. 2. Chemical structure of Direct Blue 80 dyestuff.

where:

wpu—wet pick up
w_f—weight of fabric after treatment
w_o—initial fabric weight

Excess water was then evaporated in a Werner Mathis AG, type LTF 134489 oven and polymerization was carried out in a temperature range of 70 to140 °C for 30 min [27]. Weight gain (add on) of about 1% for PET fabric samples resulting from polymerization of AEM 5700 was determined by weighing specimens before and after treatment.

2.3. Dyeing experiments

The direct dyestuff concentration was 3 g/l. The liquor ratio was set at 40:1. All the dyeing experiments were conducted on a Werner Mathis AG type BFA8 9389 laboratory dyeing machine. The temperature was increased at 1 °C /min up to 100 °C, and then the process was completed at this temperature for 10 min.

2.4. Rewetting time measurements

AATCC test method 27-1994 was used for the determination of rewetting time. The coated fabrics were kept in the conditioned lab at least 3 h before the first measurement. The temperature of the lab was kept constant at 70 °F. The relative humidity was 65%.

2.5. UV spectroscopy and color measurements

UV absorbance of the direct dye solutions was measured at $\lambda_{\max} = 568$ nm with a Varian, CARY 3E/UV Visible spectrophotometer. *K/S* values were determined with a Mini Scan XE Plus model manufactured by Hunter Associates Laboratory. *K/S* values were determined from the Kubelka–Munk equation (Eq. (2)):

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

where:

R—reflectance.

2.6. Thermal analysis

Thermal characteristics of samples were determined with a Perkin–Elmer Model 7 differential scanning calorimeter and thermogravimetric analyzer. Sample weight varied from 2.5 to 6 mg in both tests. In DSC experiments, the samples were sealed in aluminum pans and scanned by heating between 25 and 300 °C. Heating and cooling rates of 20 °C/min were used. In TGA experiments, the samples were put inside a platinum pan hanging in a furnace. Polymer weight loss was recorded while the furnace was heated from 30 to 1000 °C at 20 °C/min. Both instruments used nitrogen as a purging gas.

2.7. Infrared spectroscopy

Transmittance Fourier transform infrared spectra were recorded on a Nicolet 510P FTIR spectrometer with OMNIC software at frequencies from 800–4000 cm^{-1} with a resolution of 4.00 cm^{-1}, sample gain 128, and number of a sample scans of 64. A sodium chloride plate was used to mount the sample for FTIR analysis.

2.8. XPS analysis

XPS data were acquired with a Riber XPS system using Mg Kα X-rays (1253.6 eV, non-monochromated) from a Mg–Al dual source and a Mac 2 cylindrical mirror analyzer. The base pressure of the main analytical chamber was 2×10^{-9} Torr. Survey scans from 0 to 1200 eV were acquired with a step size of 1.0 eV and a dwell time of 0.1 s. Narrow scans of individual photo peaks were acquired with a step size of 0.1 eV and a dwell time of 0.2 s.

2.9. Atomic force microscopy

A Digital Instruments D3000 AFM with Nanoscope III controller and extender module was used to record phase lag data.

2.10. ASTM E2149-01—dynamic shake flask test

This quantitative method was used to measure the effect of exposing known concentration of a standard strain of bacteria suspended in phosphate buffer for a certain length of time. The test utilized a 150 ml Erlenmeyer flask in which 5 ml of titer of 1×10^5 CFU/ml of *Escherichia coli* (ATCC 8439) was added to 50 ml of 0.3 mM of phosphate buffer containing a 1-g sample of fibers and 0.01% of Q2-5211 wetting agent. This system was then placed on a Burrell wrist action shaker for 1 h. The concentration of colony forming units after 1 h was compared with the value at zero time in order to calculate the percent bacterial reduction [28].

3. Results and discussion

3.1. Polymerization of 3-(trimethoxysilyl)-propyldimethyloctadecyl ammonium chloride

3.1.1. Direct polymerization under acidic conditions

Trialkoxysilanes and also 3-(trimethoxysilyl)-propyldimethyloctadecyl ammonium chloride (AEM 5700) are usually polymerized under acidic conditions at pH of 4. The process involves two steps. First is the partial hydrolysis of trimethoxysilyl groups followed by the

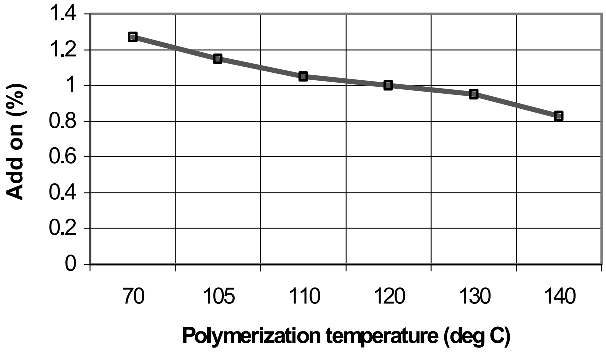

Fig. 3. Effect of polymerization temperature on add-on.

polymerization of free hydroxyl groups and the formation of –Si–O–Si– units. Since AEM 5700 contains three methoxy groups, it is expected that a crosslinked polymer can be formed. Excessive cyclization could also occur and lead to the formation of cage-like clusters or ladder polymers [14–16]. Indeed as polymerization temperature increases from 70 to 110 °C the add on gradually decreases (Fig. 3) thus indicating a loss of water, which is a byproduct of this reaction and obviously a higher degree of conversion.

At a low polymerization temperature or a low degree of conversion, one could expect more silanol groups on the fiber surface. Consequently, the coating on a PET substrate should be more hydrophilic. On the other hand, the hydrophobic coating should be anticipated at the end of polymerization when the degree of conversion is high. Therefore, a measurement of the rewetting time could be an indirect measure of the conversion. Consequently, a short rewetting time implies that the coating is hydrophilic and absorbs water more easily. A very long rewetting time might indicate that the availability of hydroxyl groups is low for interaction with water. According to AATCC test method 27-1994, the rewetting time can be described as the time required for a droplet of water to be absorbed by a fibrous surface. As a drop of water gradually is absorbed by fabric, the area of this tiny liquid mirror gets smaller and finally diminishes entirely leaving only a dull wet spot behind. Hence, the end of the wetting process is determined when the liquid on the surface of the cloth loses its specular reflective power.

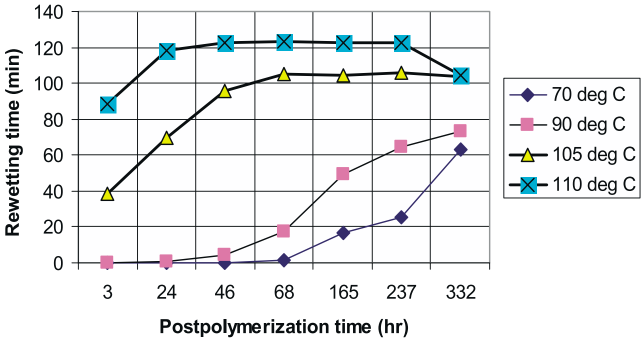

Fig. 4. Effect of post-polymerization time on rewetting time after direct polymerization of AEM 5700 under acidic conditions at various temperatures.

PET fibers as well as fabrics are very hydrophobic and therefore the rewetting time should be relatively long. Our experiments on double knit fabric show that rewetting time varies from 5 to 6 h. Because of the evaporation of water, the true rewetting time on the PET fabric could be even longer.

Fig. 4 shows the dependence of postpolymerization time on rewetting time for the direct polymerization of AEM 5700 under acidic conditions at various temperatures. After polymerization at the low polymerization temperature of 70 °C, the coating is extremely hydrophilic and the rewetting time is zero after 3, 24 and 46 h of conditioning. It is interesting to see that post-polymerization causes the rewetting time to increase up to 63 min. This could be possibly an indication that even at the ambient temperature, polymerization proceeds but at a much lower rate. A similar trend was observed for samples polymerized at 90 °C but as expected, longer rewetting time was observed. However at temperatures of 90 °C or higher, the coating formed becomes more hydrophobic and the rewetting time initially increases with an increasing postpolymerization time, and reaches a plateau at 110 and 125 min for samples polymerized at 105 and 110 °C, respectively.

3.1.2. Alkaline hydrolysis of AEM 5700 and polymerization under acidic conditions

Alkaline hydrolysis of 3-(trimethoxysilyl)-propyldimethyloctadecyl ammonium chloride is much faster than acidic hydrolysis. Free silanol groups are usually formed in a short period of time at pH of 9 [31]. Only 18.9 min are needed to convert >96% of the methoxy groups to trisilanol at 25 °C. We were interested to see how polymerization proceeds when all methoxy groups in AEM 5700 are hydrolyzed. In such an experiment, we set pH of 9 for a 1% aqueous solution of AEM 5700 with sodium carbonate and let hydrolysis occur for 60 min, before adjusting pH to 4 just before polymerization. Fig. 5 shows the effect of post-polymerization time on rewetting time after alkaline hydrolysis of AEM 5700 and polymerization under acidic conditions at various temperatures. As can be seen in this Figure, the rewetting time initially tends to decrease with increasing time and reaches a plateau, on average, at 2.8 min, regardless of the polymerization temperature. The data definitely prove that hydrophilic coating based AEM 5700 can be formed on PET fabrics. This unusual effect could be related to the availability of unreacted hydroxyl groups and quaternary ammonium groups for interaction with water. We can also postulate that these hydrophilic groups are oriented toward the surface of the coating. A similar effect for aminoethyl-aminopropyl silicones was observed by Bereck et al. [19].

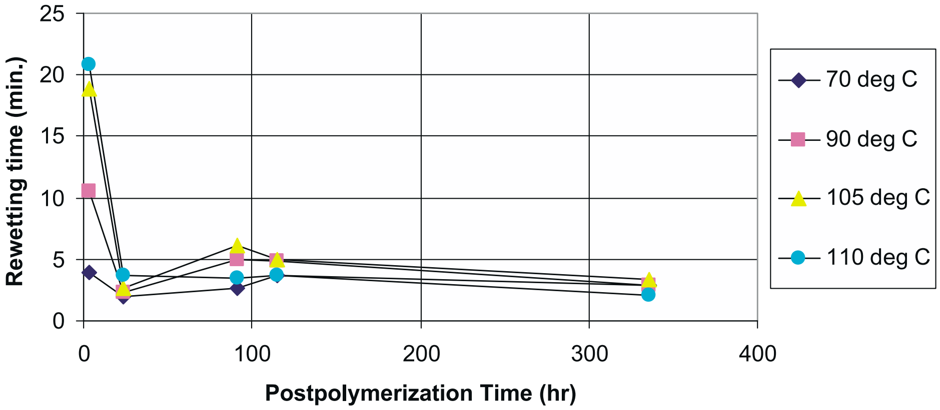

Fig. 5. Effect of postpolymerization time on rewetting time after alkaline hydrolysis of AEM 5700 and polymerization under acidic conditions at various temperatures.

3.2. FTIR spectroscopy

Table 1 lists major IR bands and their possible assignments and Fig. 6 shows four spectra for AEM 5700 polymerized at the ambient temperature and 110 °C. All these spectra show that the band splits into a doublet at 1129–1035 cm^{-1}. Such a split is known to occur when siloxane chains become longer [30]. The band near 920 cm^{-1} is assigned to the Si–O stretch of Si–OH. All samples also show a broad peak at 3400 cm^{-1}, which can attributed to the presence of moisture but it could also be related to free silanol OH stretching vibration [29].

Spectra of coatings (Fig. 6c and d) based on AEM 5700 formed after alkaline hydrolysis and polymerization show new bands. The C=O stretching for the acetyl group gives a strong infrared absorption band in the 1770–1700 cm^{-1} region. However, the acetyl group on the silicon atom is moisture sensitive and it reacted with water later on to give acetic acid and –Si–OH.

3.3. XPS surface analysis

XPS technique is a good tool to determine the content of various elements at a depth less than 20 nm; obviously the thickness of our coatings is much greater. Table 2 presents the atomic ratios O/C, N/C and Si/C on the surface of polyester treated with AEM at 110 °C. Two coatings were analyzed; one was formed directly under the acidic condition and the other was produced after an alkaline hydrolysis and subsequent polymerization. Interestingly, in both cases, data show that the quaternary nitrogen is present in the upper layer. In the first case the atomic ratio N/C of 0.049 is in good agreement with the calculated values of 0.0449. However, in the second case, the N/C ratio is only 0.024. The corresponding O/C ratio for this coating is 0.69. This value is much higher than the calculated one of 0.154. If one recalls that these coatings were hydrophilic, we can perhaps speculate that this effect is attributed to the increased concentration of hydroxyl groups present in the upper layer of these coatings. The experimental values for Si/C ratio of 0.09 and 0.11 match perfectly with the calculated value for this atomic ratio. It is also worth mentioning that an atomic O/C ratio of 0.80 was found for the untreated PET fabric, and the calculated O/C ratio is 0.53.

Table 1
IR band assignments of coatings based on 3-(trimethoxysilyl)-propyldimethyloctadecyl ammonium chloride [30]

Peak (cm^{-1})	Assignment
2919 (s)	In plane CH_2 str.
2850 (s)	Out of plane CH_2 str.
1467 (m)	CH_2 deformation
1193 (m)	CH_3 rock of OCH_3
920 (v)	Si–O str. of Si–OH
825 (m)	Si–O–C sym. str.

3.4. AFM surface analysis

We used AFM technique to determine whether our coatings show any micropores and to characterize the coating roughness. It is very well known that fiber porosity or roughness generally improves the moisture transport and therefore could explain the wettability of our coatings. Fig. 7a shows the typical AFM texture of a coating after polymerization under acidic conditions on Mylar film at the ambient temperature. As can be seen in this Figure, the surface exhibits a ‘grainy’ texture. Interestingly, such texture was not observed after AEM 5700 polymerization at 110 °C (Fig. 7b). No micro or nanopores were found in the hydrophilic or hydrophobic coatings.

$$R_q = \sqrt{\frac{\sum (Z_i - Z_{\mathrm{ave}})^2}{N}} \qquad (3)$$

The roughness of the coating surface can be a significant

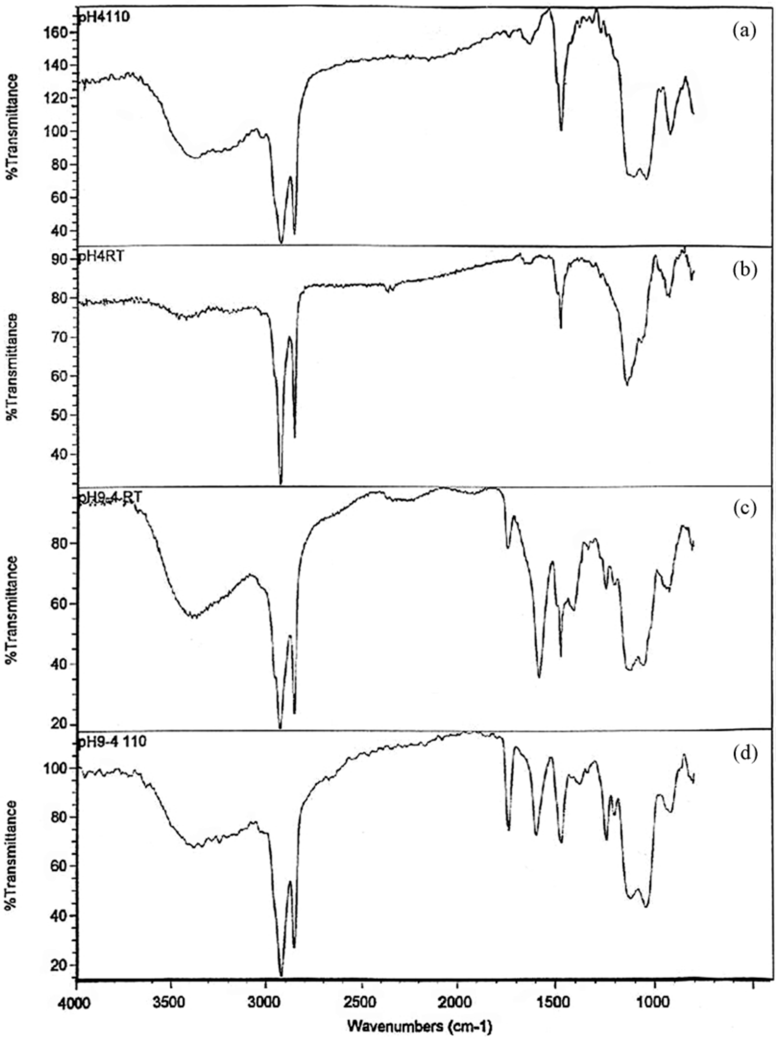

Fig. 6. Infrared spectra after AEM 5700 polymerization under acidic conditions at (a) 110 °C, (b) ambient temperature, (c) ambient temperature after alkaline hydrolysis, (d) 110 °C after alkaline hydrolysis.

factor affecting wettability or moisture absorption. If one assigns a vertical distance Z on a rough surface, the standard deviation R_q given by Eq. (3) can be a measure of roughness where:

Z_{ave}—the average of the Z values within the given area
Z_i—the current Z value
N—the number of points within the given area

Only a small number of points were tested on the coatings. R_q values for AEM 5700 coatings directly polymerized under acidic conditions were as low as 1.196 nm but not higher than 5.069 nm. The coatings formed after alkaline hydrolysis of AEM 5700 and polymerization under acidic conditions were rougher and had higher R_q values in the range from 1.413 to 44.504 nm.

Table 2
XPS analysis of PET and PET surfaces treated with AEM 5700 after hydrolysis under different conditions

Coating formed	O/C	O/C calcd	Si/C	Si/C calcd[a]	N/C	N/C calcd[a]
Untreated PET	0.80	0.53[b]	0.029	0	0	0
After acidic hydrolysis and polymerization	0.47	0.154[a]	0.11	0.09	0.049	0.0449
After alkaline hydrolysis and polymerization under acidic condition	0.69	0.154[a]	0.09	0.09	0.024	0.0449

[a] Calculated from the chemical formulae of 3-(trimethoxysilyl)-propyldimethyloctadecyl ammonium chloride (see Fig. 1).

[b] Calculated from the chemical formulae for the repeating unit of poly(ethylene terephthalate).

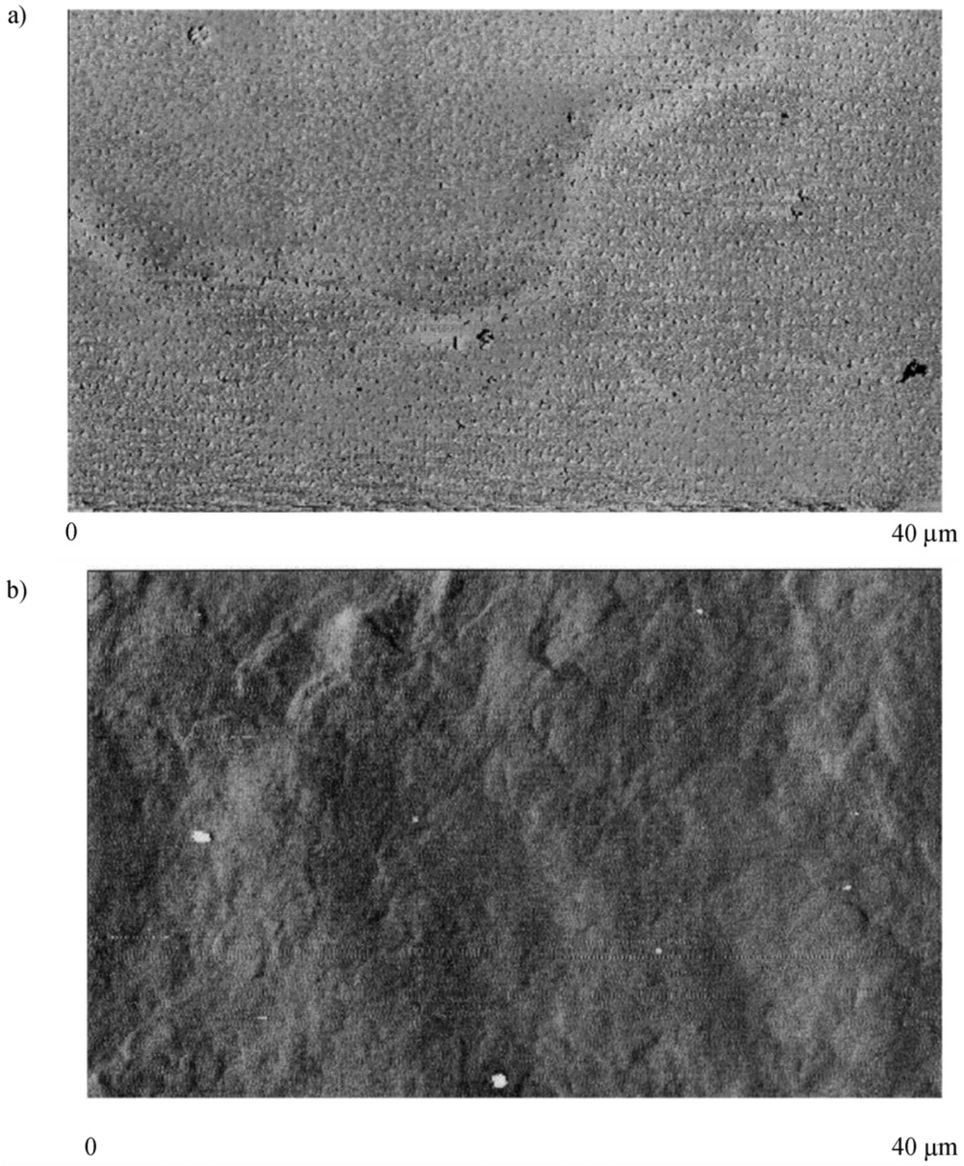

Fig. 7. AFM texture of a coating after polymerization under acidic conditions on Mylar film at (a) ambient temperature, (b) 110 °C.

3.5. Dyeability after treatment of PET fabrics with AEM 5700

A measurement of the absorbance for the direct dye solution after dyeing gives an indication of the degree of hydroxylation of the treated surface, provided the structure is sufficiently accessible to the dye molecule. This effect can also demonstrate whether the dye sites i.e. positively charged quaternary nitrogen atoms are accessible for the direct dye. As can be seen in Fig. 8 the UV absorbance of the remaining Direct Blue 80 dye solution is very low thus indicating the direct dye uptake for the fabric coating. Less dye uptake can be seen when the polymerization temperature is lower than 100 °C. Furthermore, Fig. 9 clearly proves that these coatings reached the deep blue color as shown by *K*/*S* values and demonstrates the binding of sulfonic groups

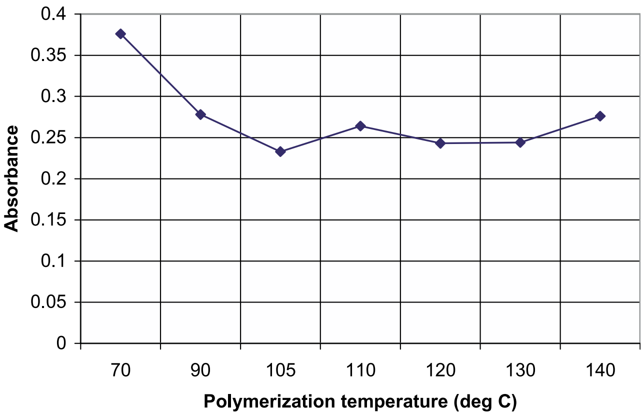

Fig. 8. Effect of polymerization temperature on color absorbance for Direct Blue 80 dyestuff.

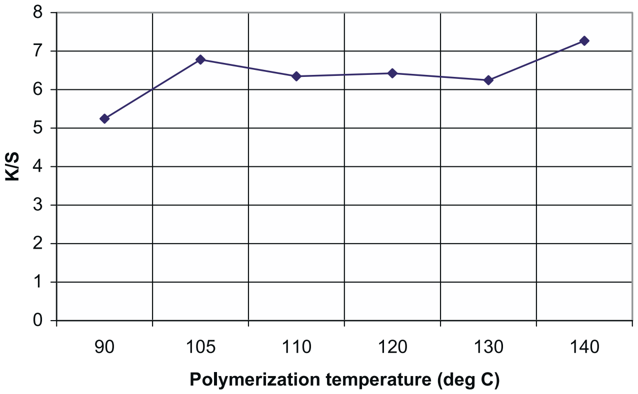

Fig. 9. Effect of polymerization temperature on the color strength (*K*/*S*) after dyeing with Direct Blue 80 dyestuff.

of the direct dye to the quaternary nitrogen sites in the AEM 5700 coating.

Interestingly, Fig. 10 shows that dyeing uniformity improves with increasing polymerization temperature. After polymerization at 70 °C, the AEM 5700 coating is not uniform but still absorbs the direct dye very well. Furthermore, Fig. 10a also shows little direct absorption by PET fabric. Good dye uniformity is accomplished when the polymerization is carried out at temperatures equal to or higher than 100 °C. However, these dyed fabrics loose their hydrophilic properties.

3.6. Thermal analysis

Even though PET fibers are thermally stable up to 300 °C, coatings based on AEM 57000 undergo some structural changes and significant weight loss occurs at much lower temperatures as shown in Figs. 11–13. A typical DSC graph (Fig. 11) shows three exotherms at 51, 93 and 228 °C for the coating formed under acidic conditions. The first exotherm at 51 °C can possibly be attributed to the small amount of methanol bound in the coating but the origin of this peak has yet to be determined. The second broad peak at 93 °C can be caused by the loss of water and the last one at 228 °C is an indication of the initial thermal decomposition of the AEM 5700 coating. A similar behavior can be seen for the AEM 5700 coatings that were formed by the alkaline hydrolysis and subsequent polymerization (Fig. 12). However, the third exotherm is not clearly defined yet corresponds to the onset of a major weight loss on the TGA curve at 218 °C (Fig.13).

3.7. Microbiological analysis

Results of microbiological analysis of PET fabrics treated with AEM 5700 under various conditions are presented in Table 3. As can be seen in this Table, excellent antimicrobial performance was demonstrated while testing the fabrics with high level of 1% and also at very low loading of 0.15% of this compound. Such low levels are

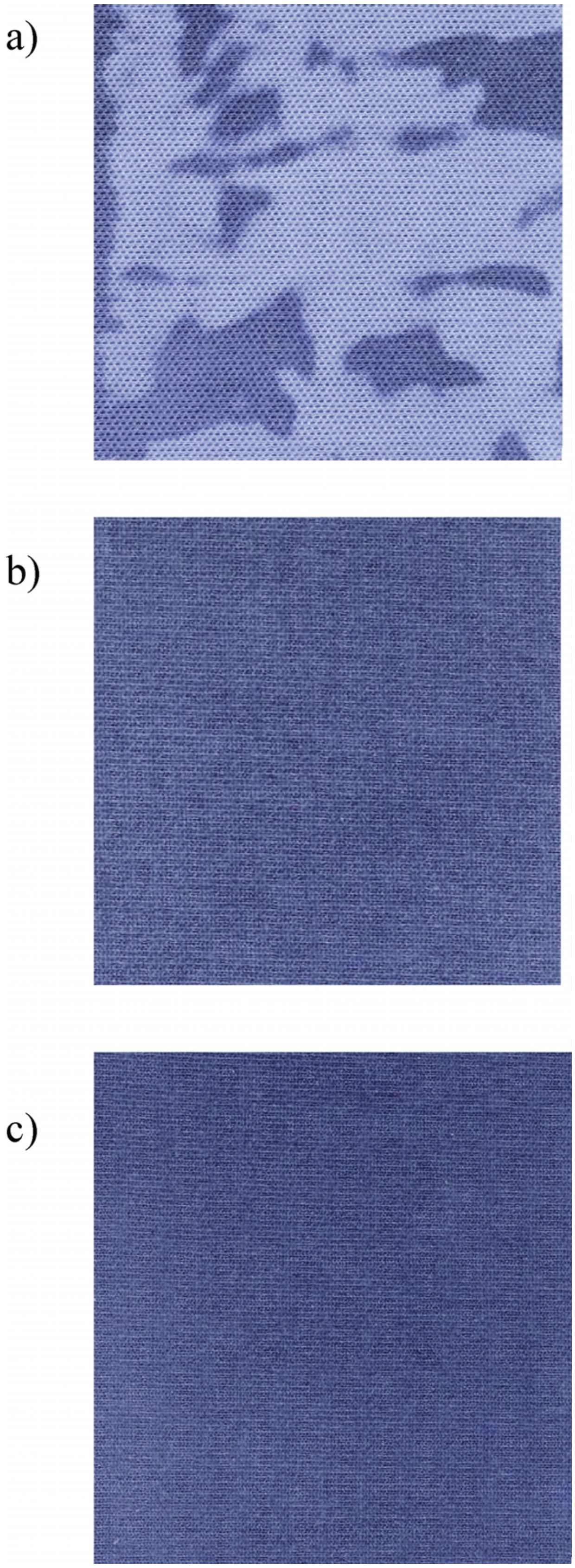

Fig. 10. Dyed polyester fabrics with Direct Blue 80 after treatment with AEM 5700 at (a) 70, (b) 105 and 140 °C.

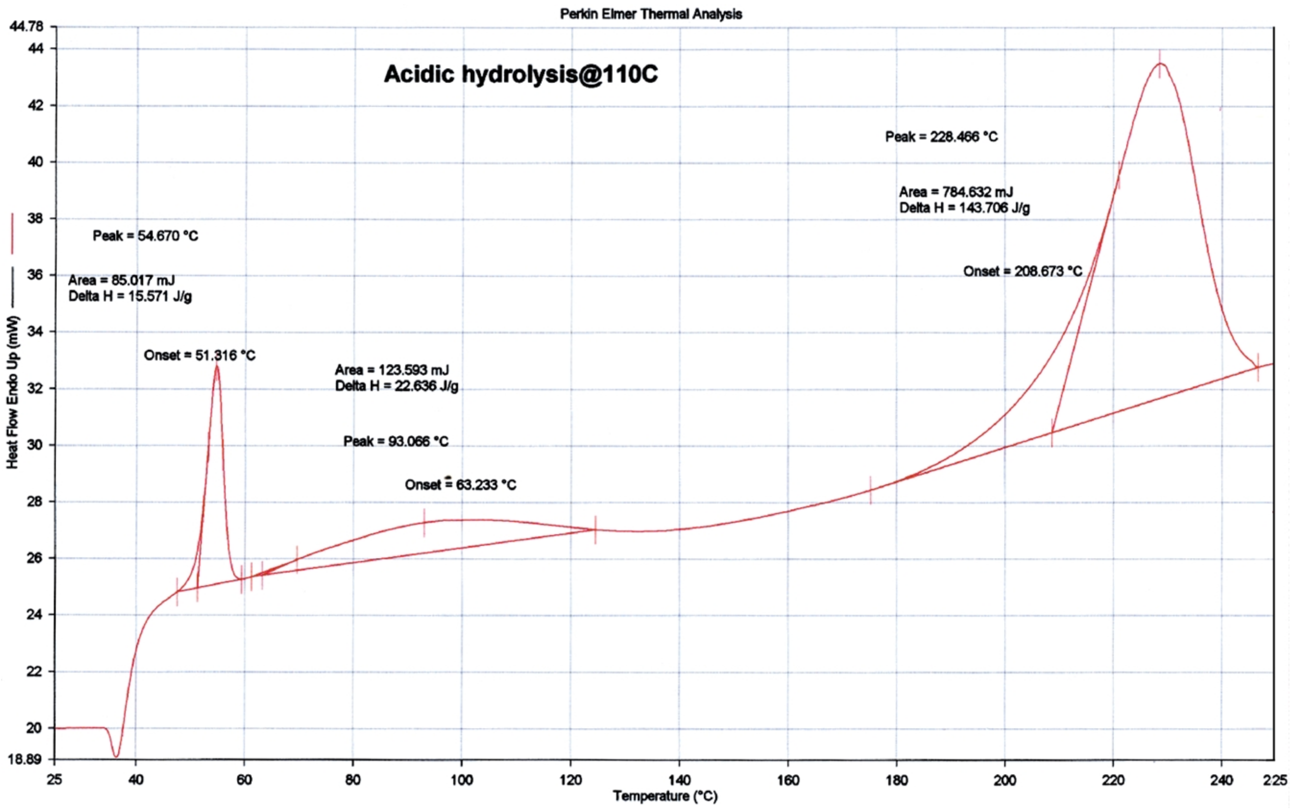

Fig. 11. DSC of coating after polymerization of AEM 5700 under acidic conditions at 110 °C.

usually recommended for commercial end uses. Our studies also showed that polymerization temperature does not affect the antimicrobial action of coating based on AEM 5700 on polyester substrates.

4. Conclusions

3-(Trimethoxysilyl)-propyldimethyloctadecyl ammonium chloride (AEM 5700) is a trifunctional monomer that can be polymerized under acidic conditions at temperatures greater than 100 °C. Under such conditions, coatings based on AEM 5700 formed on PET substrates are relatively hydrophobic, exhibiting rewetting times higher than 100 min.

Hydrophilic coatings exhibiting a rewetting time of 2.8 min can be also formed when AEM 5700 is initially hydrolyzed under alkaline conditions, and then polymerized under acidic conditions. This effect could be related to the accessibility of unreacted hydroxyl groups and quaternary ammonium groups for interaction with water.

Excellent antimicrobial action was demonstrated for all coatings based on AEM 5700 on PET substrates.

Table 3
Results of microbiological analysis of PET fabrics treated with AEM 5700 under various conditions

Sample #	Amount of AEM 5700 (%)	Polymerization temperature (°C)	First run	Second run	Pass/fail
1	1	70	>99.5	>99.5	Pass
2	1	70	>99.5	97.3	Pass
3	1	110	>99.5	>99.5	Pass
4	1	110	>99.5	>99.5	Pass
5	1	130	>99.5	>99.5	Pass
6 Control PET fabric	0	–	0	0	Fail
7	0.15	110	>99.5	93	Pass

Note. All coatings were formed directly after adjusting pH to 4. Polymerization time was 30 min.

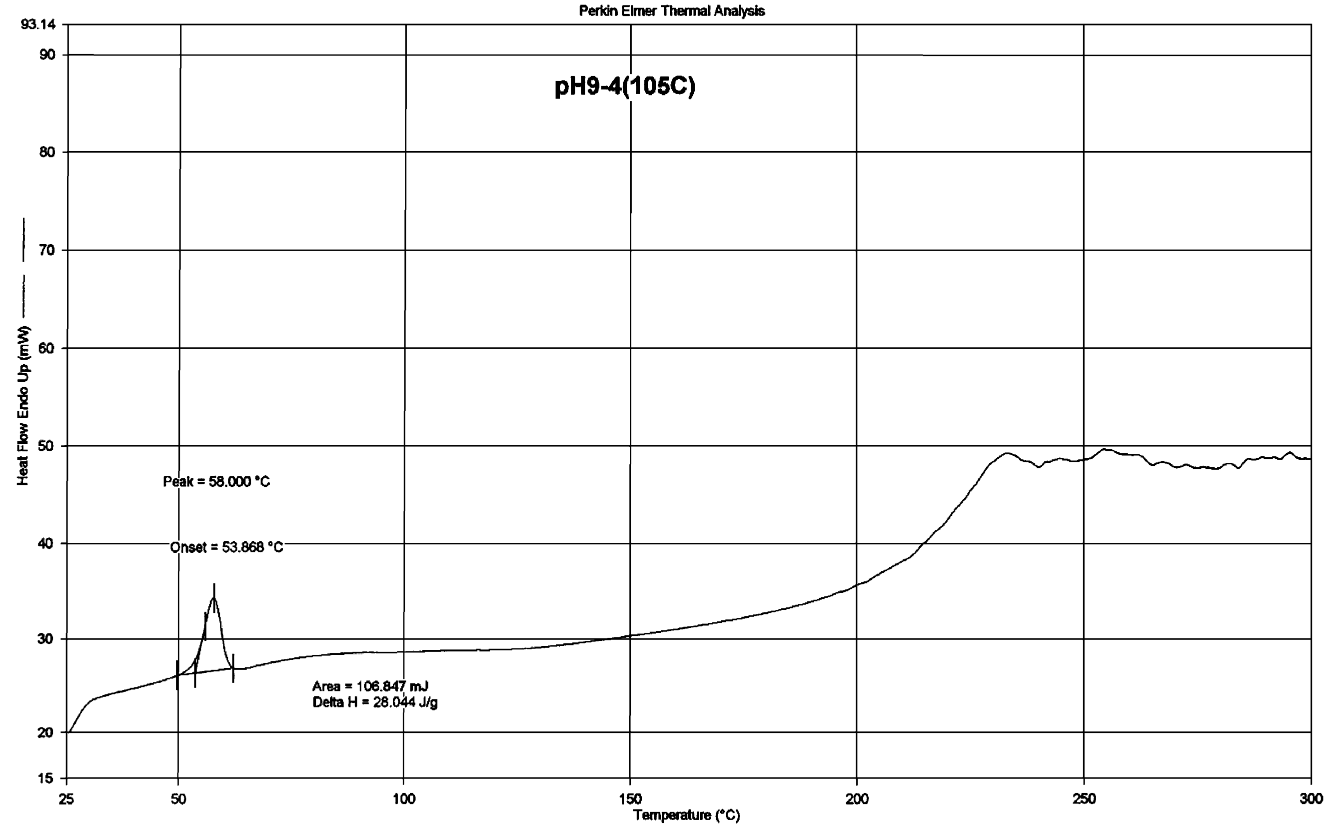

Fig. 12. DSC of coating after alkaline hydrolysis of AEM 5700 and polymerization under acidic conditions at 105 °C.

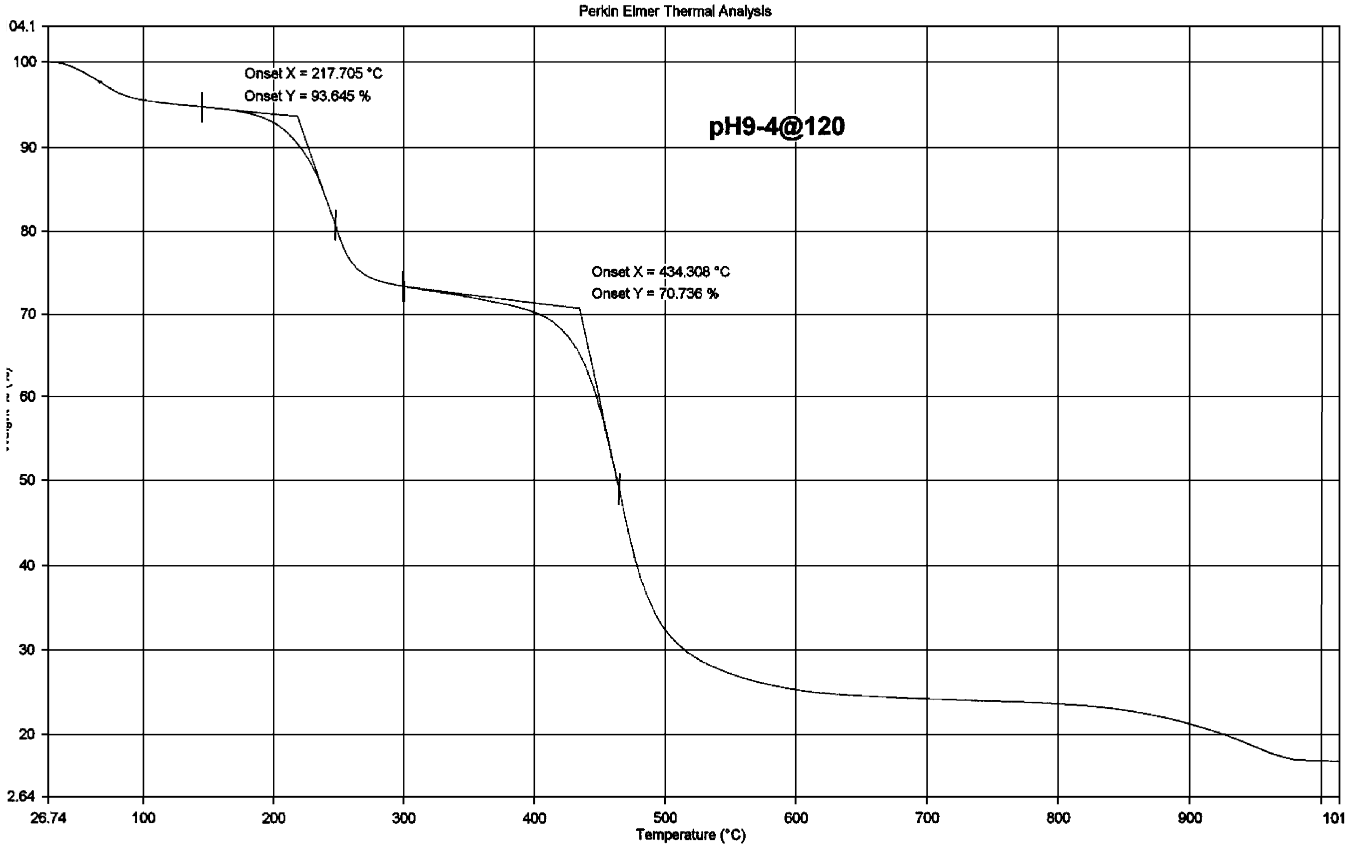

Fig. 13. TGA of coating after alkaline hydrolysis of AEM 5700 and polymerization under acidic conditions at 120 °C.

Polyester fabrics having AEM 5700 coating can be dyed with direct dyes.

The loss of hydrophilic properties for AEM 5700 coatings is observed after dyeing with direct dyes.

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