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ORIGINAL PAPER

A flame retardant, antimicrobial and UV protective polyester fabric by solvent crazing route

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Abstract

The multifunctional finishing effects like flame retardancy, antibacterial activity and UV protection were produced at room temperature in polyester fabric using "solvent crazing technique" with $ZnCl_2$ dissolved in acetone. The morphological changes due to incorporation of ZnO on the fabric was analysed by the SEM-EDX analysis and XPS analysis. The fabric was investigated for various mechanical and functional properties. Flammability of the functionalized fabric was investigated by limiting oxygen index (LOI) & vertical burning tests (VBT) whereas thermal behaviour was assessed by TGA-DTG and DSC. LOI up to 29 was achieved and also passed all the requisite standards for VBT for curtain fabrics. The growth of bacteria on the treated fabric was negligible. Also, 50 + UPF factor was attained for protection from UV rays. The washing fastness was good and after 10 washing cycles about 60% of the original metal content retained in the fibre.

Keywords Solvent crazing · Polyester fabric · Flame retardancy · Antibacterial activity · UV protection

Introduction

Polyester fabrics are widely used in the textile field, owing to their outstanding characteristics, i.e. high strength, high melting point, better crease resistance, high elastic modulus, better creep properties, etc. However, polyester has low moisture absorption, high static build-up, high pilling tendency, low dyeability, high flammability, low antibacterial activity, low UV protection under practical conditions due to the compact physical structure and absence of chemical active groups in polyester fibres [1]. To overcome these limitations various type of physical and chemical modification is carried out at finishing stage of wet processing treatment. New process techniques are also demanding that will allow minimum application of energy with considerable importance. Solvent crazing [2, 3] can be considered as a new approach which saves time, energy and consumption of chemicals.

Solvent crazing is stress-induced adsorption and produces bulk plasticization effect on a polymer. The basic requirement in this technique is surface active liquid environment (SALE) [4] i.e. liquid (solvent) which can lower the polymer surface tension, without swelling or dissolving polymer in the stress inducing device. This liquid produces certain effect on stress-strain curve. This technique is completed in two steps, first is craze initiation [5] where due to the viscous flow of liquid the glassy polymer starts to develop crazes near the stress regions. Second is craze propagation which is due to the plasticization effect. During the craze initiation, additives are incorporated in the bulk of polymer by viscous flow of SALE into the micro voids. In the fibre structure, the transport rate of additives is much higher in viscous flow than diffusion. As a result, the mutual dispersion and uniform blending of a polymer and an incompatible low or high-molecular-additive component may be achieved [2].

This technique has been used for improving various properties in the synthetic fibres. Previously the number of unusual material combinations such as AgCl in poly(propylene) [6], nickel in poly (propylene) [7], metallic copper in poly (ethylene terephthalate) [8], phosphonic acid amide in polyester have been prepared by solvent crazing [9].

Takeno, used solvent crazing technique for dyeing polypropylene filament with acid dyes and showed that acid dye gets easily absorbed in those crazed regions of polypropylene [10]. Previously we have tried to incorporate crude disperse dye into the polyester and polyamide filament yarns thereby

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eliminating milling process for the disperse dyes. Also simultaneously dyeing along with drawing of polyester filament at room temperature could be successfully achieved and results were comparable with the conventional [11]. This indicates that the crazing is a promising method for the development of highly dispersed porous materials with controlled structure and facilitates the easy incorporation of any modifier. Oliver Weichold also used solvent crazing technique to incorporate $ZnCl_2$ in partially orientated polyester fibres followed by thermal annealing at 80 °C for 30minto get antimicrobial effect. However due to annealing in stretched condition the tensile property of polyester fibres was affected [12].

Most of these applications were performed on the yarn or filament. If this technique is extended on to the fabric form then it will have more commercial value as small lots can be finished with the desirable properties. In the present research work, solvent crazing was explored to get multifunction finishing properties in polyester fabric. $ZnCl_2$ was first dissolved to its maximum solubility in acetone and through this solution fabric was stretched to get dispersion and deposition of $ZnCl_2$ in the fabric and then in the stretched condition only the polyester fabric was dipped in NaOH solution which nucleated $ZnCl_2$ into ZnO. The mechanical, functional and fastness properties were evaluated as per various standards [13].

Experimental

Materials

Polyester curtain fabric made up of fully oriented yarn having GSM 331, 136 X 518 denier yarn count, 51 × 40 epi × ppi (construction) and 1/3 satin weave was sourced from local market in Mumbai, India. The laboratory grade $ZnCl_2$, NaOH, acetone, acetic acid & non-ionic soap were purchased from S D fine chemical ltd., Mumbai, India.

Selection and preparation of surface active liquid environment (SALE) and its optimisation

The selection of solvent depends on the swelling of polyester and the solubility of the $ZnCl_2$. The solubility of $ZnCl_2$ was measured up to maximum concentration in various solvents like n-propanol, isopropanol, and ethylene glycol etc. It was observed that $ZnCl_2$ had maximum solubility in acetone and did not show any adverse effect on the structure of polyester [14]. Different concentrations of $ZnCl_2$ were employed in this study.

Preparation of modifier or precipitating solution

Modifier was required to convert $ZnCl_2$ into ZnO. NaOH was used as a modifier at minimum concentration which could be just sufficient for nucleation and precipitation of $ZnCl_2$ and did not harm polyester. We used 5 g per litre NaOH for 5 min wherein the precipitation happened instantaneously.

Application of SALE on polyester fabric

According to the principle of solvent crazing, the sample was clamped between the fixed and movable jaws on a stretching frame and immersed in a tray containing SALE at room temperature by subjecting the sample to 30% stretch. After immersion, stress was induced in the fabric sample giving rise to crazes (Fig. 1). Through these crazes, penetration and deposition of $ZnCl_2$ inside polyester occurred. When the time interval was completed the frame

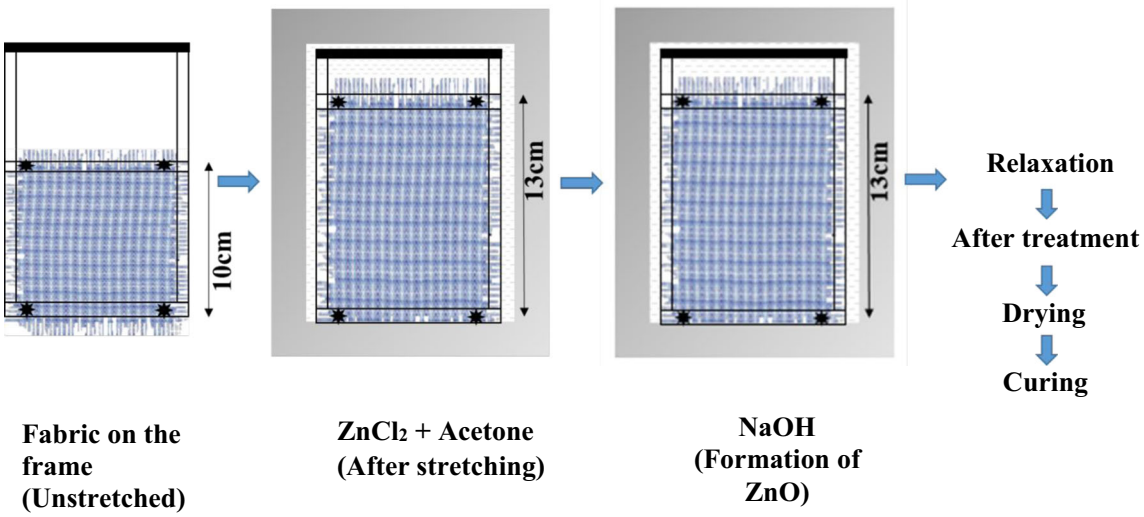

Fig. 1 Incorporation of ZnO in polyester fabric

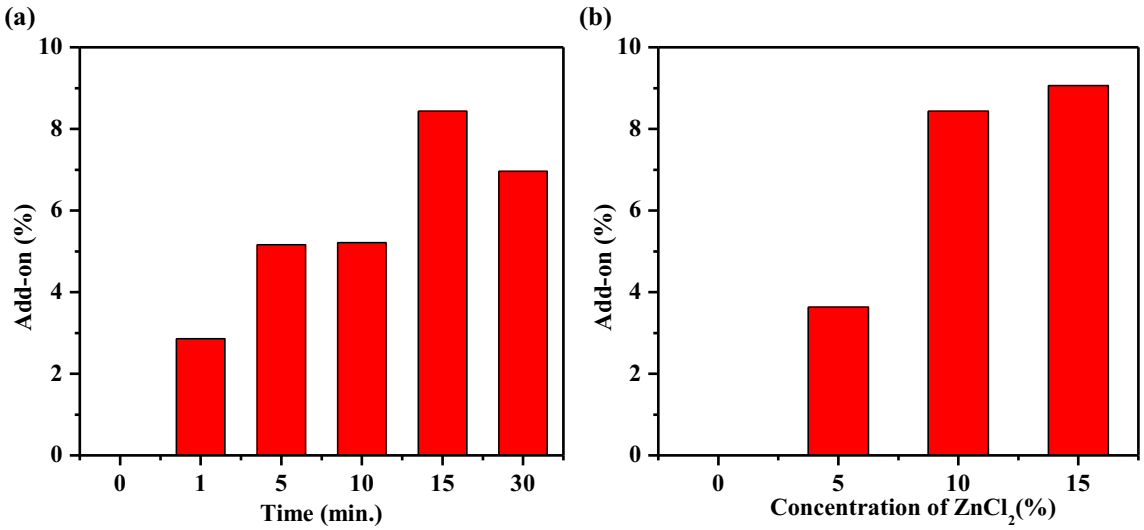

Fig. 2 Effect of (**a**) crazing time using 10% $ZnCl_2$ and (**b**) concentration of $ZnCl_2$ at 15 min on add-on percentage

was withdrawn and submerged in the modifier solution present in another tray. In the modifier solution, $ZnCl_2$ got precipitated into ZnO inside and on the surface of fabric. After this, the fabric was released from the frame and allowed to relax for 1 h.

Neutralization was done with 2% acidic acid at room temperature followed by soaping using 2% non-ionic solution at 100 °C for 15 min to remove unreacted chemicals. This fabric was dried at 100 °C and heat set at 190 °C for 2 min to achieve the dimensional stability.

Characterisation

Add-on percentage

After the crazing treatment, total weight gain was calculated in terms of add-on percentage using the following equation:

$$\text{add} - \text{on}\% = \frac{W_t - W_o}{W_t} \times 100\%$$

Where,

W_o Weight of polyester fabric before treatment
W_t Weight of polyester fabric after treatment.

Flame retardant characteristics

The vertical burning test was evaluated by BS EN5867-2b:2008 [15] test method. The fabric is tested before and after cleaning. A fabric sample is placed vertically on a metal frame. A flame is applied to the fabric surface for 15 s. Results of these tests were assessed on 3 parameters i.e. after flame time, i.e. the time till which the material kept burning after removal of ignition source, after glow time which is the time and char length, which is the distance from the margin of the fabric that was exposed to the flame to the end of the area which was affected by the flame. Three measurements were taken for each samples and average was reported.

Table 1 Flammability results of different polyester fabrics treated for 15 min

Polyester samples	Untreated	Treated	Treated	Treated	Treated -after 5 wash	Treated -after 10 wash	Treated -after 20 wash
$ZnCl_2$ concentration (%)	0	5	10	15	10	10	10
Add-on (%)	0	4	8	9	6	5	3
LOI (%)	21	26	29	29	27	26	25
Burning rate ± 0.2 (mm/s)	13	11	3	3	6	9	11
After flame time ± 0.2 (sec.)	8	2	0	0	1	2	3
Char length ± 2 (mm)	200	162	51	48	93	142	159
Melt-dripping	Heaviest	Heavy	Not observed	Not observed	Not observed	Not observed	Heavy

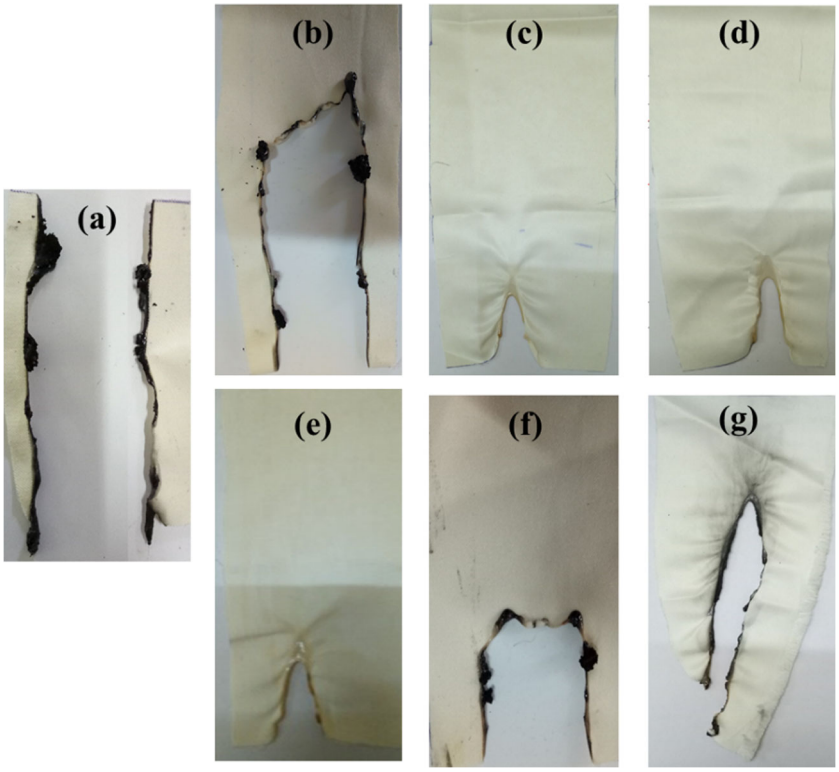

Fig. 3 Images after vertical burning test of (**a**) untreated, (**b**) 5%, (**c**) 10%, (**d**) 15% $ZnCl_2$ treated fabrics and (**e**) after 5(**f**) 10 and (**g**) 20 washes

The limiting oxygen index values were calculated by IS 13501:1992 [16] on Dynisco LOI analyser (Alpha Technologies, United Kingdom) using nitrogen and oxygen gases. The LOI values were measured by following equations:

$$LOI\% = \frac{(O_2)}{(O_2 + N_2)} \times 100$$

Thermal properties

The thermal stability study of fabrics was done by thermogravimetric analyser, TG-60H (Shimadzu, Japan) and DSC on DSC-60 (Shimadzu, Japan). Samples were analysed under nitrogen condition at 100 mL/min flow rate and 10 °C/min heating rate from 25 °C to 500 °C (for TGA analysis) and 25 °C to 300 °C (for DSC analysis).

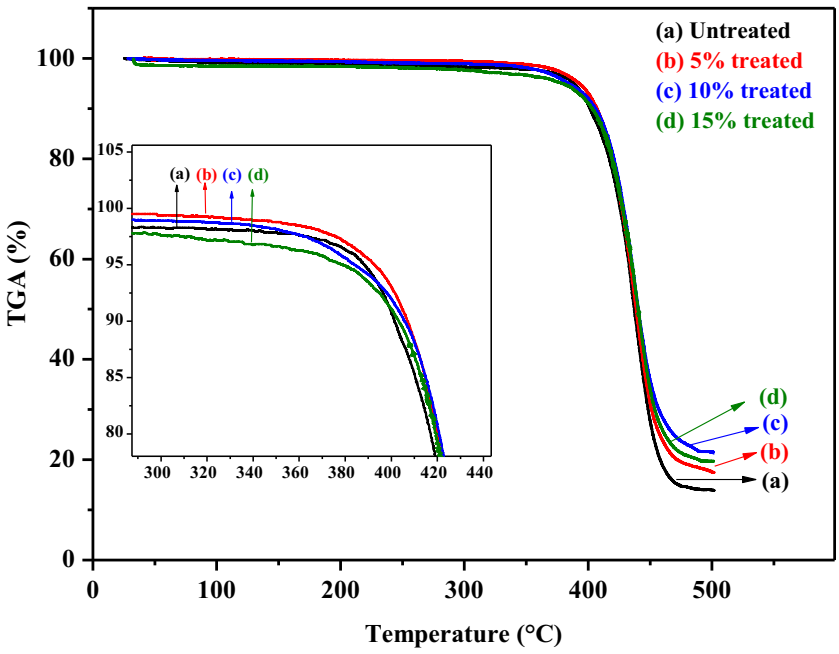

Fig. 4 TGA curves of untreated and treated polyester samples

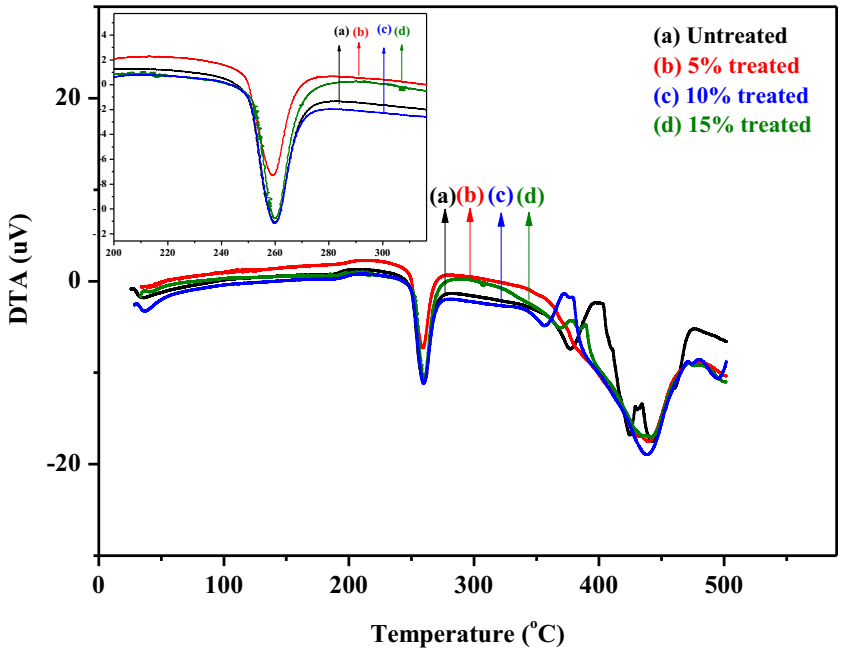

Fig. 5 DTGA curves of untreated and treated polyester

Antibacterial study

The antibacterial property of untreated, treated and after ten wash fabric was investigated using AATCC100–2012 test method [17]. This method gives inhibition against *S.aureus* (strain no. ATCC 6538), gram positive bacteria and *K.pneumoniae* (strain no. ATCC 4352), gram negative bacteria. The total number of colonies recovered at '0' h and '24' h contact time on the agar plates were counted and antibacterial activity was expressed as the percentage reduction of micro-organisms using following equation:

$$\text{Antibacterial activity (R)} = \frac{B - A}{B} \times 100$$

Where,

A Number of colonies recovered at '24' h contact time
B Number of colonies recovered at '0' h contact time

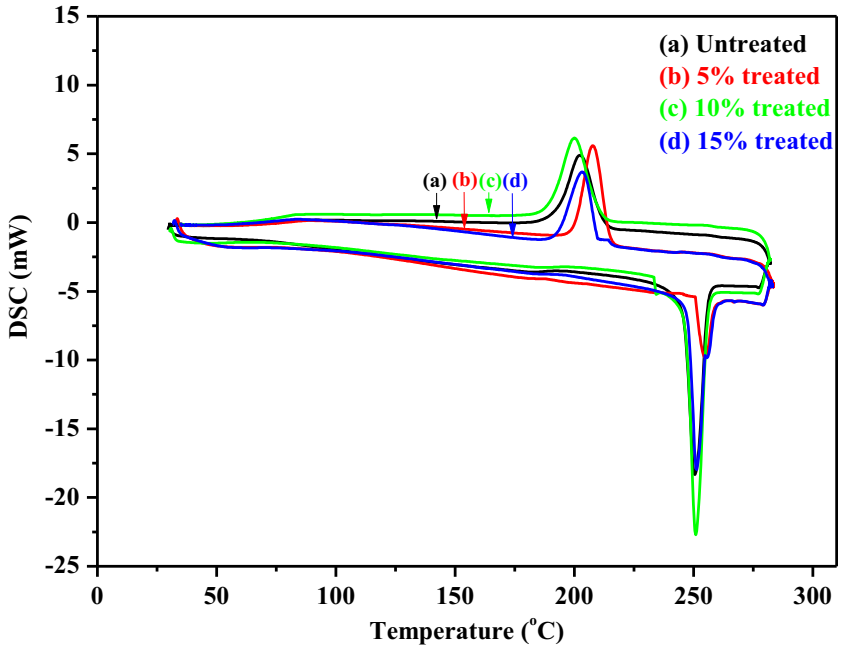

Fig. 6 DSC curves of untreated and treated polyester samples

Table 2 Onset decomposition temperature (T_{onset}), maximum decomposition temperature (T_{dmax}), endset decomposition temperature (T_{endset}) and residual weight of untreated and treated polyester fabrics (10% $ZnCl_2$)

Samples	$ZnCl_2$ concentration, %	T_{onset} (°C)	$T_{dmax.}$ (°C)	T_{endset} (°C)	Residual (wt%)
Untreated	0	406	431	436	13
Treated	5	409	433	437	17
Treated	10	420	432	450	21
Treated	15	426	432	451	21

UV protection analysis

The treated fabrics have ability to obstruct UV light which is denoted by ultraviolet protection factor (UPF) values. The UPF was measured by AS/NZ 4399:2017 standard [18] on a UV spectrophotometer UV-2600 (Shimadzu, Japan) using integrating sphere in the range of 290 nm to 400 nm at an interval of 5 nm. The percentage obstruction of UV-A (315–400 nm) and UV-B (280–315 nm) were computed by the transmittance data. UVA, UVB and UPF values were measured using the following equations [19, 20]

$$UVA = \frac{T_{315} + T_{320} + T_{325} + \ldots + T_{395} + T_{400}}{18}$$

$$UVB = \frac{T_{290} + T_{295} + T_{300} + T_{305} + T_{310} + T_{315}}{6}$$

$$UPF = \frac{\sum_{y=290}^{400} E_\lambda \times E_\lambda \times \Delta\lambda}{\sum_{y=290}^{400} E_\lambda \times E_\lambda \times T_\lambda \times \Delta\lambda}$$

Where,

E_λ	Erythematous spectral coefficient
S_λ	Solar spectral irradiance
T and T_λ	Transmittance
$\Delta\lambda$	Measured wavelength interval (nm).

Morphological study

The surface morphology of fabrics was analysed using SEM analysis on XL 30 (Philips, The Netherlands) and EDX (energy dispersive X-ray spectroscopy) analysis on JSM-6380 (JEOL ltd., Japan). EDX analysis was used to study the uniformity of ZnO deposition. For scanning, each sample was coated with gold by sputter coater for scattering of static charge generated during the electron bombardment.

X-ray photoelectron spectroscopy (XPS) study

XPS patterns were analysed by XPS spectroscopy using ESCA+, (omicron nanotechnology, Oxford Instrument Germany) equipped with monochromactic Aluminium Source.

X-ray diffraction (XRD) study

XRD patterns were analysed by XRD-6100 (Shimadzu, Japan), using Cu-Ka radiation at 40 kV and 30 mA in the angular range (2θ) from 5 to 80°. XRD of the ZnO powder was also taken after dissolving 10% $ZnCl_2$ in acetone and precipitating in NaOH.

Mechanical properties

Tensile property of fabrics was investigated by Tinius Olsen Inc., (Model H5KS, USA) with 10 kN load cell and 2 mm/min cross-head speed according to ASTM D5035 [21]. Measurement was repeated 3 times for each sample and average was reported.

Washing fastness study

The washing fastness was investigated as per AATCC 61-1A:2013 [22] using 0.37 wt.% detergent on Laundrometer by Rossari Lab Tech Pvt. Ltd. Mumbai for 5, 10 and 20 washing cycles.

Table 3 DSC data of untreated and treated fabrics

Samples	$ZnCl_2$ concentration, %	T_g (°C)	T_m (°C)	T_c (°C)	ΔH_f (J/g)
Untreated	0	84	251	203	56
Treated	5	87	253	203	57
Treated	10	86	252	200	63
Treated	15	86	252	202	61

Table 4 Antibacterial activity of different polyester fabrics

Samples	Test culture	No. of colonies recovered at '0' h (B)	No. of colonies recovered at '24' h (A)	Reduction of microorganisms (R)
Untreated	*S.aureus*	1.7×10^5	1.8×10^7	No activity
	K.pneumoniae	1.7×10^5	1.9×10^7	
Treated	*S.aureus*	1.7×10^5	1.1×10^4	94%
	K.pneumoniae	1.6×10^5	1.1×10^4	93%
After 10 wash	*S.aureus*	1.6×10^5	1.7×10^4	90%
	K.pneumoniae	1.6×10^5	1.9×10^4	88%

Results & discussions

Add-on percentage

ZnO add-on percentage was evaluated with the varying concentration of $ZnCl_2$ in SALE and at the different crazing times (Fig. 2a and b). It was observed that the add-on percentage increased with the concentration of additive from 4 to 99%. At a fixed 10% $ZnCl_2$ concentration, as crazing time increased, ZnO loading in polyester fabric also increased. However beyond 15 min, there was hardly any improvement. This has happened as the fabric became saturated with the Zn ions which occupied every available micro void and interstice space generated in the fabric due to crazing. There was no more space available for accommodating more amounts of these ions even if the crazing time was increased. Thus 10% $ZnCl_2$ and 15 min. Treatment time were considered as the optimized parameters.

Flame retardancy properties

The flame-retardant properties and images of burnt polyester samples for different $ZnCl_2$ concentration along with 5, 10 and 20 washes are shown in Table 1 and Fig. 3. From Table 1 we could observe that the LOI of treated samples increased from 26 to 29 with the add-on percentage. For the untreated fabric sample, the value was 21. Thus the incorporation of ZnO imparted the flame retardant property to the polyester fabric up to certain concentration. During the solvent crazing treatment, Zn^{2+} ions penetrated into the micro voids and these Zn^{2+} ions formed ZnO, during NaOH treatment, and get entrapped in the fibre structure. The upper limit in the LOI value can be related to the saturation of the fabric with ZnO. It has been already reported in the literature that ZnO imparts flame retardant property to the polyester fabrics [23]. However for the washed samples the LOI value decreased to 25 after 20

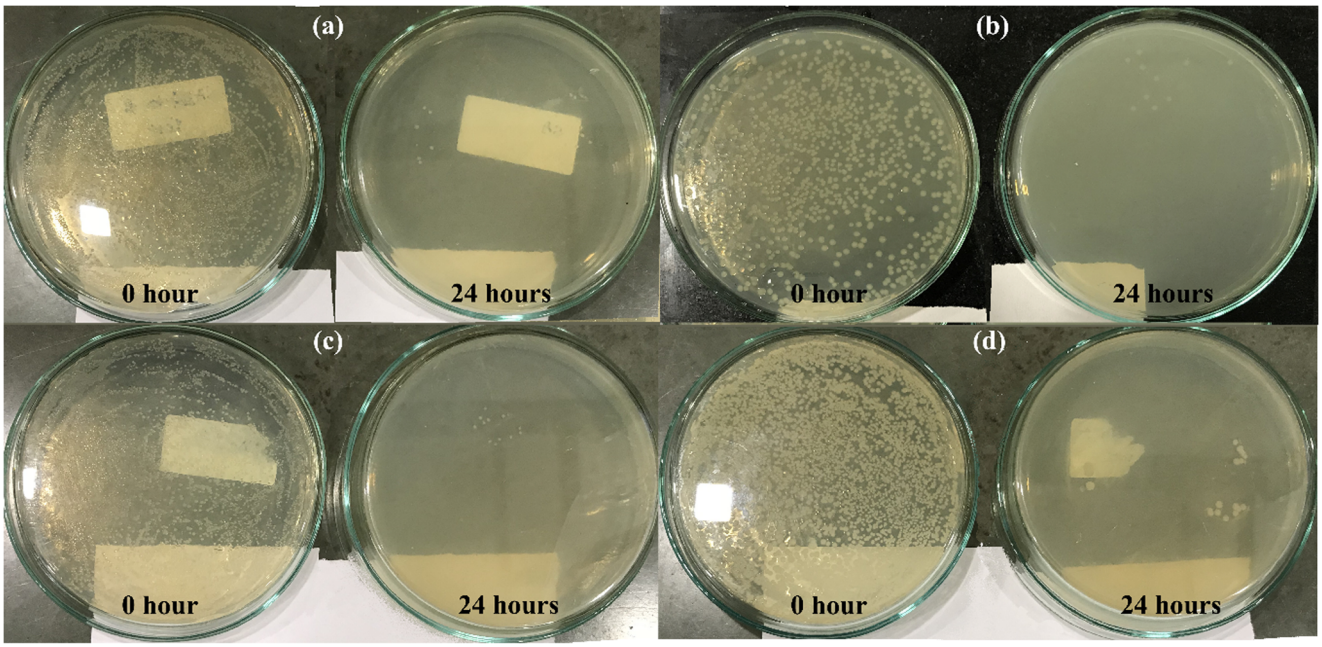

Fig. 7 Antibacterial activity of treated sample [against (**a**) *S.aureus*, and (**b**) *K.pneumoniae*] and same after 10 wash [against (**c**) *S.aureus*, and (**d**) *K.pneumoniae*]

Table 5 UV protection test of polyester fabric treated for 15 min

Samples	Untreated	Treated	Treated	Treated	Treated -after 5 wash	Treated -after 10 wash	Treated -after 20 wash
$ZnCl_2$ Concentration	0	5	10	15	10	10	10
Add-on (%)	0	4	8	9	6	5	3
UV-A	5	4	2	2	2	3	4
UV-B	0.3	0.2	0.1	0.1	0.1	0.2	0.2
UPF	50 + (239)*	50 + (323)	50 + (606)	50 + (699)	50 + (489)	50 + (369)	50 + (293)

*Actual value

washes which is quite acceptable [24, 25]. The lowering of the LOI was because of some of the ZnO being removed during the washing treatment as indicated by the lower add on percentage shown in the table. When ignition source was removed, the untreated fabric was burntcompletely with the burning rate of 13.33 mm/s. $ZnCl_2$ treated fabrics retarded the spreading of the fire having the burning rate of 3.2 mm/s. Melt dripping and after flame time of more than 2 s and was observed only for untreated, 5% $ZnCl_2$ and after 20 wash samples. Similarly the char length was relatively higher for all these samples. From the images of the burnt samples also, we can note the same behaviour. Thus in general, we can say that the fabric became flame retardant after using 10% $ZnCl_2$ and was suitable upto 10 washes. Also it is noted that after 10 washing cycles about 60% of ZnO (5%) from the initial add on of (8%) was retained in the fibre.

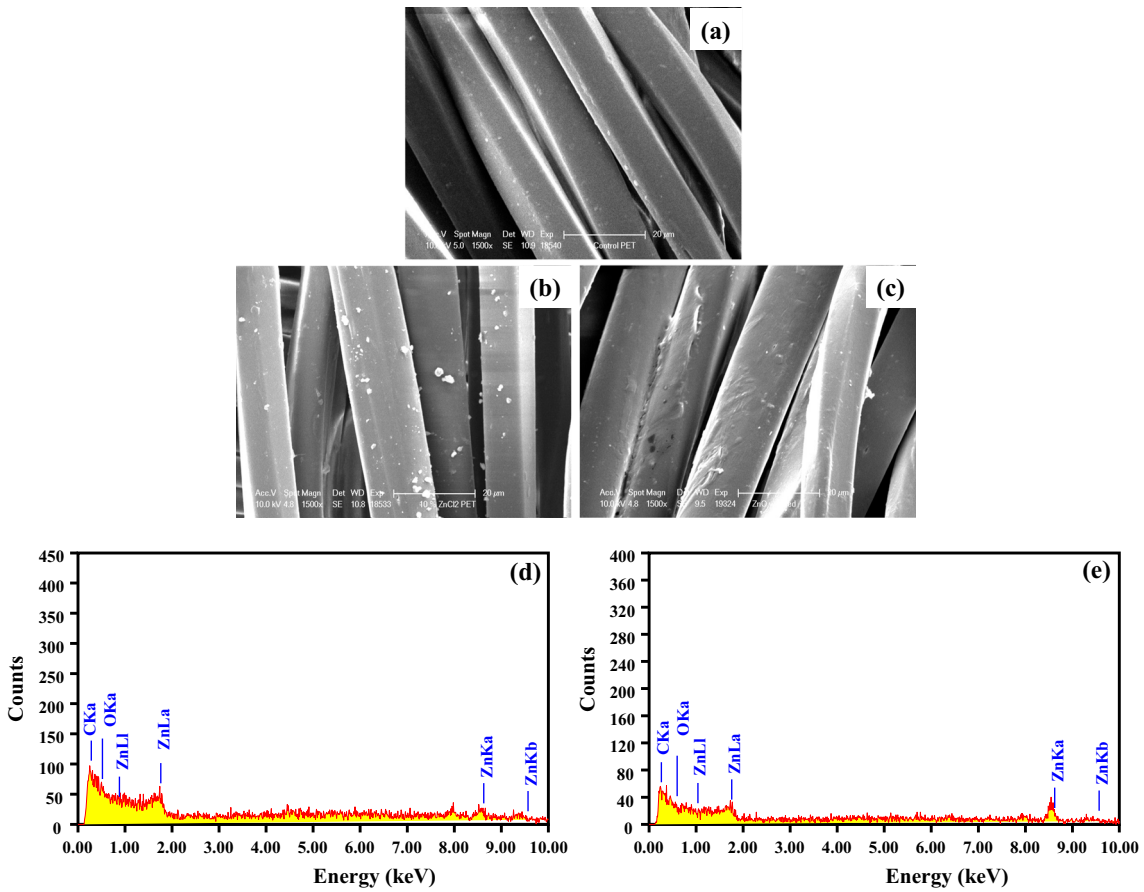

Fig. 8 SEM and EDX images of (**a**) untreated fabric, (**b**) treated fabric with 10% $ZnCl_2$ (**c**) same treated after 10 washes at 1500X (**d**) treated fabric with 10% $ZnCl_2$ and (**e**) same treated after 10 washes

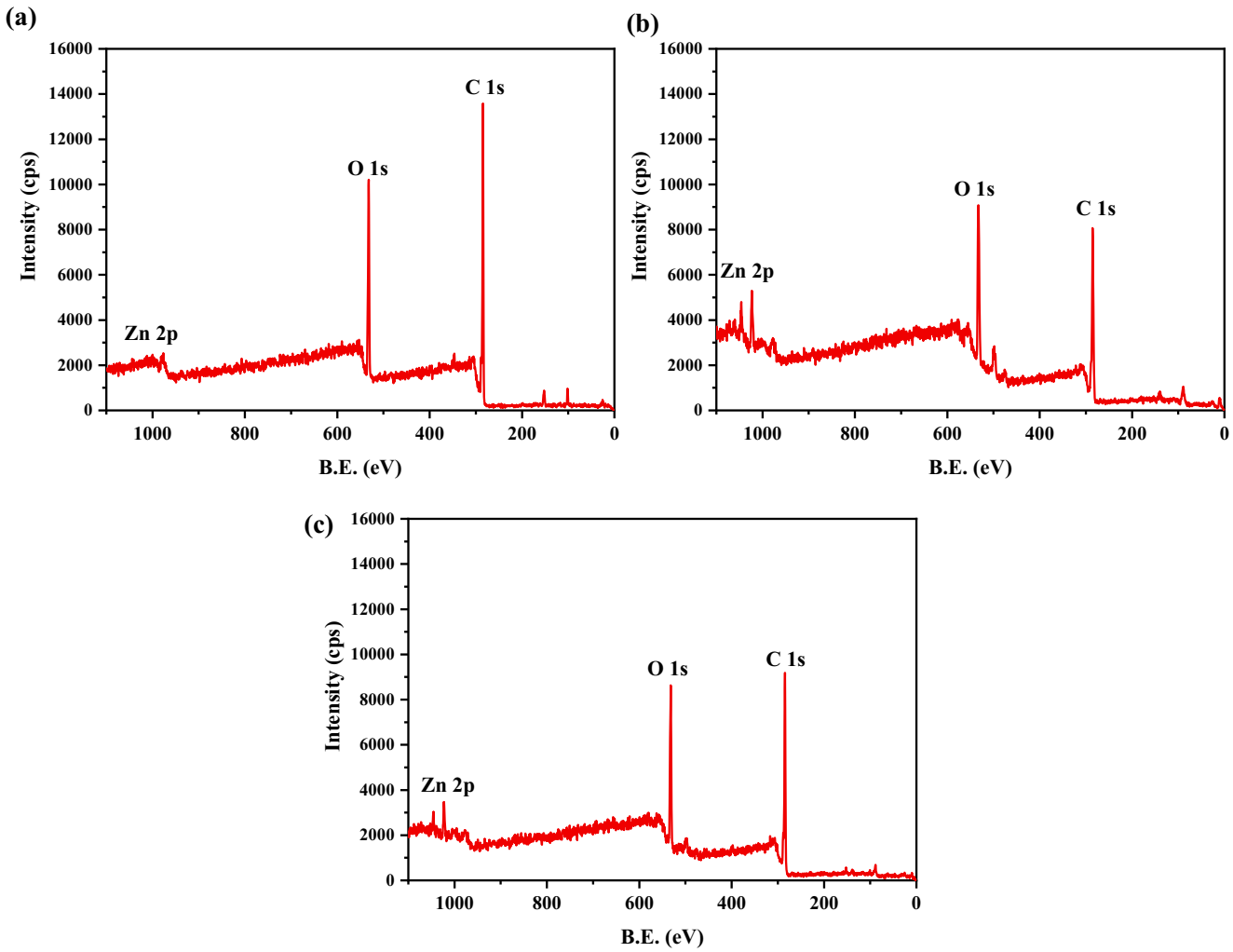

Fig. 9 XPS analysis of (**a**) untreated fabric, (**b**) treated fabric and (**c**) After 10 wash samples

Thermal behaviour

TGA, DTG and DSC were performed to study the thermal behaviour of the treated polyester samples as shown in the Figs. 4, 5, and 6 and Tables 2 and 3. The degradation of untreated and treated fabrics was observed as a single step process and was accompanied by weight loss owing to the depolymerisation of the polyester chain caused by β-H chain-transfer reactions. The polyester generates benzoic acid, carbon dioxide, carbon monoxide, benzoic acid derivatives, oligomers and polycyclic aromatic hydrocarbons upon high temperature decomposition [26]. The onset degradation temperatures increased from 406 °C to 426 °C while the endset temperature from 436 °C to 451 °C. The amount of residual weight also increased from 13% to 21% for the treated fabric. Thus the formation of ZnO in polyester significantly improved its thermal stability. The addition of ZnO affected the critical thermal behaviour slightly in terms of glass transition, melting point and crystallization temperature. There was increase in the enthalpy value due to the interference of ZnO in the crystallization that enhanced the flame retardancy and slowed down the polymer degradation [27].

Table 6 Element content and element ratio of untreated treated and after 10 wash samples

Sample	Element content (%)			Ratio		
	C 1 s	O 1 s	Zn 2p	O/C	Zn/C	(O + Zn)/C
Untreated	65	22	0	0.3	0	0.3
Treated	64	29	4.30	0.4	0.06	0.5
After 10 wash	65	26	2.49	0.4	0.03	0.4

Antibacterial properties

The antibacterial property of untreated, 10% $ZnCl_2$ treated and the same after 10washes was evaluated. As shown in

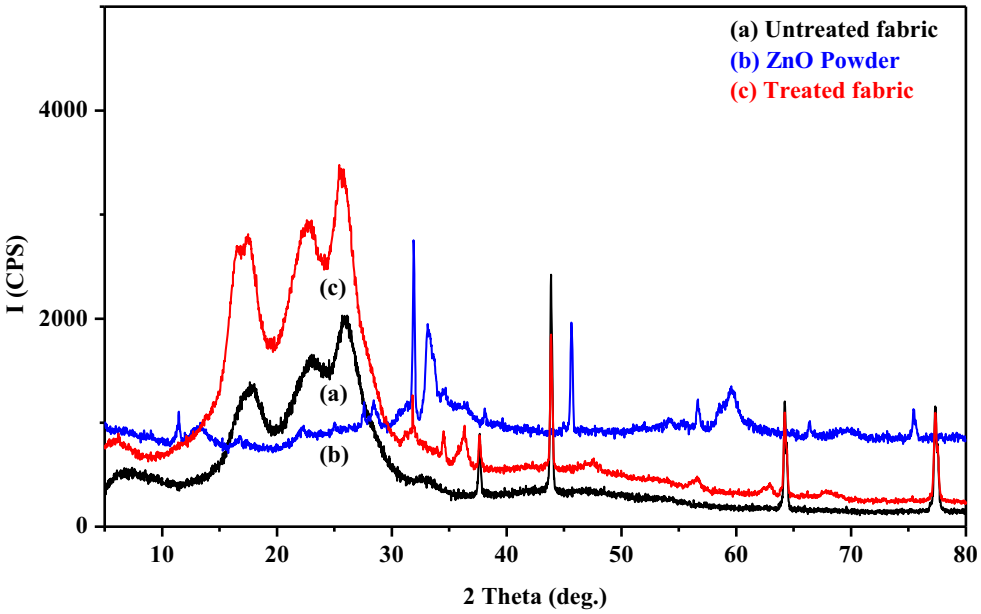

Fig. 10 XRD diffractograms of (a) untreated fabric, (b) ZnO powder and (c) treated fabric

Table 4 and Fig. 7, untreated fabric did not show antibacterial activity while treated sample showed excellent resistance to both the micro-organisms by giving 93% reduction. This might be due the high content of ZnO in the polyester fabric and continuous releasing of Zn^{+} ion into the surrounding medium, which was well supported by the ZnO loading analysis using EDX. For prokaryotic organisms, zinc is an essential micronutrient. However, at super physiological levels, zinc inhibits the growth of many bacteria [28].

UV protection properties

When polymeric materials are exposed to UV light (200 to 400 nm), they undergo photo-chemical degradation. The common aim of using curtains is to obstruct light, but the weak UV obstruction property limits their uses. The UV transmittance (UV-A and UV-B) and UPF values of different polyester samples are reported in Table 5. It can be observed that untreated fabric allowed passage of 4.75% and 0.26% of radiation in the UVA and UVB regions, respectively. As the add-on

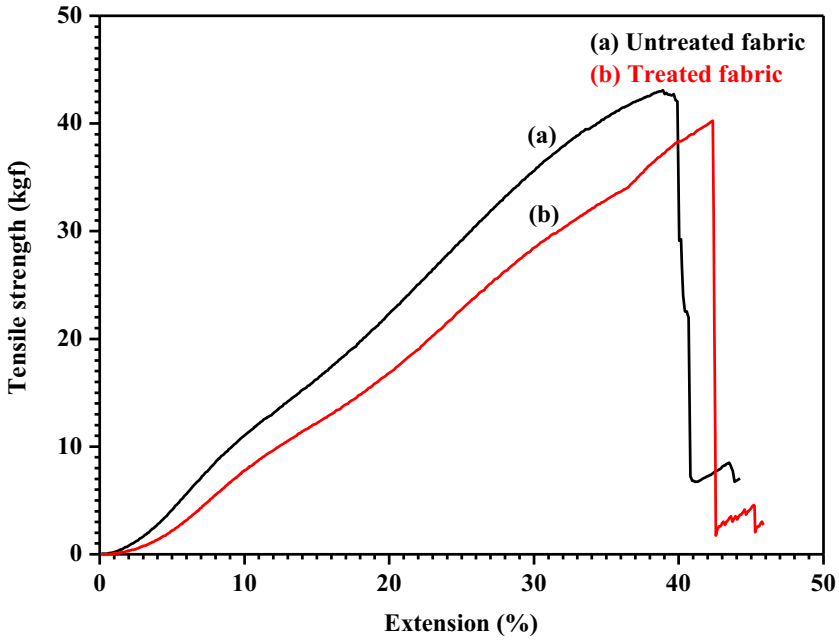

Fig. 11 Tensile strength (Kgf) and extension of untreated and 10% $ZnCl_2$ treated fabric

percentage increased the UVA and UVB radiations were blocked and UPF of the fabric increased indicating uniform dispersion and deposition of ZnO in polyester acting as an effective barrier for the UV radiations. The untreated sample also showed UV protection property as it was a tightly woven fabric having relatively higher GSM. However for curtain fabrics higher the UPF rating better is the product. The UPF factor of treated sample was almost 3 times than the untreated.

Morphological analysis

Figure 8 shows SEM and EXD of different polyester samples. The untreated polyester surface appeared smooth with some small spots which might be oligomers formed during the melt spinning process. The treated and after washed samples showed the deposition of the ZnO on the surface of the fibres. The chemical composition analysed from EDX further confirmed its presence (Fig. 9a and b). Apart from the carbon, zinc and oxygen were the other two elements on the treated (zinc 43%; oxygen 28%) and after 10 wash samples (zinc 36%; oxygen 24%).

XPS analysis

Table 6 shows the elements content and element ratio of the untreated, treated and after 10 wash samples. There was hardly any change in proportion of C-element composition in these samples whereas the amount of O-element has increased for the treated samples. Zn element was not observed in the untreated sample. However its presence was detected in both treated and after 10 washes sample (Fig. 11). The content of Zn was less in washed samples as some of the superficial ZnO was removed during washing. The reason for increase in O content could be the enrichment of samples due to the presence of oxygen in ZnO.

XRD analysis

X-ray diffraction of the untreated, $ZnCl_2$ treated and ZnO powder are shown in Fig. 10. Characteristic peaks of polyester were observed at $2\theta = 44°$, 64°, and 77° corresponding to its crystal structure [1]. Similarly, characteristic peaks of ZnO powder were observed at $2\theta = 32°$, 33° and 46° [29]. The subdued sharpness and intensity of ZnO peaks in the treated samples confirmed the in-situ synthesis of ZnO in the fabric. Presence of ZnO in polyester decreased the crystallinity of polyester, as shown by less intense peaks of the treated polyester sample. The crystallinity values of untreated, treated and ZnO powder were 26%, 24% and 18% respectively. The treated polyester had low crystallinity due to amorphous and self-aggregation of ZnO in polyester. Polyester has both crystalline and amorphous phase organized randomly but due to the uniform dispersion of ZnO in the micro voids of polyester its amorphous region might have increased [27].

Mechanical properties

The solvent crazing directly affect the mechanical properties of fabric. The tensile strength v/s elongation curve of untreated and treated samples is presented in the Fig. 11. The treated sample exhibited lower strength (40 ± 2 Kgf) and stiffness ($42 \pm 2\%$) as compared to untreated sample (43 ± 2 Kgf and $41 \pm 2\%$). However the loss was due to the increment in the amorphous content in treated sample as explained in the XRD section [30].

Conclusion

In this research, the solvent crazing technique was used as a novel tool for obtaining multifunctional finishing effects like flame retardancy, antibacterial activity and UV protection by dispersion and deposition of ZnO in the polyester matrix. Using SEM-EDX analysis, XPS analysis and X-rd patterns, the presence of ZnO was verified. The LOI value and vertical burning test showed excellent flame retardancy. The treatment produced milder thermal effect on Tg, Tc and Tm. The antibacterial test showed excellent resistance to bacteria. The UV protection factor was also improved substantially. About 60% of the ZnO was still present even after 10 washes showing good washing fastness. The fabric modification was accompanied by slight decrease in the tensile strength.

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