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Preparation of multifunctional polyester fabrics using zinc stannate nanoparticles

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A novel self-cleaning, antibacterial and ultraviolet-resistant polyester fabric loaded with zinc stannate nanoparticles has been prepared. Zinc stannate nanoparticles have been incorporated onto the surface of polyester fabrics by an *in situ* sonochemical approach. Synthesis of zinc stannate nanoparticles is carried out along with the alkaline hydrolysis of polyester fabric using sodium hydroxide. The produced novel polyester fabrics are characterized by field emission scanning electron microscope, energy-dispersive X-ray spectroscopy and X-ray diffraction. The self-cleaning property, antibacterial efficiency and ultraviolet protection property of the samples are also studied. The treated polyester fabrics possess significant antibacterial activity and photo-catalytic self-cleaning property by degradation of methylene blue under sunlight irradiation. Also, the coated fabric shows proper UV-blocking activity. Moreover, all properties of the treated fabric with zinc stannate nanoparticle are found superior as compared to the treated sample with zinc oxide nanoparticle.

Keywords*:* Antibacterial fabric, Polyester fabric, Sonochemical synthesis, Self-cleaning fabric, Zinc stannate nanoparticle

1 Introduction

In the recent years, nano-photocatalysts like titanium dioxide (TiO_2), zinc oxide (ZnO), zirconium oxide (ZrO_2) and tungsten trioxide (WO_3) have found a broad range of applications in industrial sectors, including electronics, medical, energy and environmental[1-5]. Also, incorporation of textiles with nano-photocatalysts created many new and promising properties for investigation. For instance, Tudu *et al.*[6] produced self-cleaning, antistain, antibacterial, and anti-water absorption cotton fabrics using titanium dioxide nanoparticles. Aminloo and Montazer[7] treated nylon/cotton blend fabrics with zinc oxide nanoparticles and reported the significant photocatalytic self-cleaning, hydrophilic and antibacterial features on the fabrics. Along the same lines, Javed *et al.*[8] obtained multifunctional cotton fabrics through in situ sonosynthesis of zinc oxide nanoparticles. Nazari *et al.*[9] coated wool fabric with nano-ZnO and proved efficient mothproofing property. Also, superior self-cleaning property on polyester fabric through applying macroporous titanium dioxide was reported by Joomjarearn *et al.*[10].

Nanoparticle of ZnO and TiO_2 are very effective in the UV region between 300 nm and 400 nm, which are mainly UV A and UV B regions. The maximum absorbance is mainly at 290-320 nm for TiO_2 and 370-385 nm for ZnO. Zinc oxide has very low chemical stability and titanium dioxide does not have a broad absorption range[11, 12]. Thus, we need to develop a nanoparticle of a metal oxide kind which could provide protection from UV rays of around 300-400 nm (covering both UV A & B regions), be nontoxic in nature so that its deposition on organisms does not cause any detrimental effect to the ecosystem and also a finishing agent that can impart multifunctional properties like UV protection, antimicrobial, self-cleaning or others. Zinc stannate (Zn_2SnO_4), as a new class of ternary oxide semiconductors with a band gap ranging from 3.8eV to 4.1eV (depending on the structure of zinc stannate), has received much attention because of its high electron mobility, high electrical conductivity, good stability and attractive optical properties. This makes it suitable for widespread applications in solar cells, sensors and photocatalysts[13-16]. Recently, Indian researchers have made attempts to exploit the potential application of zinc stannate in textile field. Paul *et al.*[17] produced functional cotton fabric with superior self-cleaning, antibacterial, flame retardancy and UV-protection properties through applying nano-zinc stannate.

Polyester (polyethylene terephthalate) is a versatile commercial polymer, widely used in textile industry. Due to its high strength, high modulus, abrasion resistance, heat set stability, light fastness and chemical resistance, polyester is the main material for producing apparel, garments, and other finished

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textile goods. However, due to its poor wettability and lack of functional groups, durable functional finishing of polyester fabrics have became concerns of the textile industry. The alkaline treatment hydrolysis of the polyester fabric leads to enhance the hydrophilicity and surface reactivity [18, 19]. This study, for the first time, is focused on *in situ* synthesis of nanocrystalline structure of Zn_2SnO_4 on polyester fabric. Synthesis of zinc stannate nanoparticles and alkaline hydrolysis of the polyester fabric have been conducted in one step using ultrasonic bath. The influence of *p*H and amount of precursors on the self-cleaning property, antibacterial efficiency and ultraviolet (UV) blocking activity of the coated polyester samples are also investigated.

2 Materials and Methods

2.1 Materials

A 100% polyester fabric, having fabric weight 150 g/m^2, warp density 36 yarn/cm, and weft density 24 yarn/cm, was used. Tin tetrachloride pentahydrate ($SnCl_4 \cdot 5H_2O$, 98%) was purchased from Sigma-Aldrich Co. (USA). Zinc acetate dihydrate ($ZnAc_2 \cdot 2H_2O$) as a precursor and sodium hydroxide (NaOH) were obtained from Merck Co. (Germany). Methylene blue (CI 52015) was supplied by Tianjin Beilian Fine Chemicals Development Co. (China).

2.2 Apparatus

The surface morphology and elemental composition of the fabrics were analyzed using a field emission scanning electron microscope (FE-SEM, MIRA3-TESCAN, Czech Republic). Gold coating was used on the samples. They were placed in sputter coater (process current 10 mA for 2 min) and then FE-SEM images were taken. The presence and crystalline structure of nanoparticles on the samples were investigated by X-ray Diffractometer (STOE model STADI MP) with Cu Kα radiation (λ=1.540 Å), operating current and voltage of 40 mA and 40 kV, and a step size of 0.04º. The Fourier transform infrared (FT-IR) spectrum was obtained by Bruker FT-IR (Germany). An ultrasonic bath model WiseClean®, 100 W, 40 kHz (Korea) for synthesis processing was used.

2.3 Sonosynthesis of ZnO Nanoparticles on Polyester Fabric

The nano-ZnO synthesis and alkaline hydrolysis of the polyester fabric by sono-chemical method were carried out as follows. Different amount of zinc acetate (1, 2, 3 and 4 % wt.) was added separately to 100 mL distilled water under ultrasonic irradiation. The polyester fabrics were immersed into the solutions, and different amounts of sodium hydroxide (1, 2, 3 and 4 % wt.) were added to the bath. The mixture was irradiated in sonicator bath for 3 h at 80 °C. Thereafter, the treated fabrics were washed with distilled water and dried at 70 °C for 8 h. Also, a control hydrolyzed polyester sample (4 % wt., NaOH) was prepared under the same conditions.

2.4 Sonosynthesis of Zn_2SnO_4 Nanoparticles on Polyester Fabric

Zinc acetate and tin tetrachloride were dissolved in distilled water and sonicated to form transparent solution in the presence of polyester fabric. Different amounts of sodium hydroxide were added to the mixture and the ultrasound irradiation continued for 3 h, during which the temperature of bath was increased from 25 °C to 80 °C. Then, the Zn_2SnO_4 nanoparticles deposited polyester fabrics were washed thoroughly with distilled water. Finally, the fabrics were dried at 65 °C for 8 h in the oven. The exact formation and corresponding number for each sample examined in this study are summarized in Table 1.

2.5 Test Methods

The photocatalytic activities of treated fabrics were estimated by decolorization of the methylene blue solution under sunlight irradiation. The concentration of dye in the solution was calculated by Varian Cary 300 UV–Vis spectrophotometer using calibration curve. The treated polyester fabrics (4 × 6 cm^2) were added into 100 mL of the dye solution (10 mg/L). Firstly, the solution mixture was stirred for 15 min without irradiation in order to get equilibrium of dye adsorption. Then the solution was irradiated with sunlight for 3 consecutive days under continuous stirring (100 rpm). The degradation degree of the methylene blue was calculated according to following equation:

$$\text{Degradation (\%)} = (C_0 - C_e)/\ C_0 \qquad \ldots (1)$$

where C_0 and C_e correspond to the initial and final concentrations of dye before and after sunlight irradiation.

AATCC 100-2004 test method as a quantitative technique was chosen for measuring antibacterial properties of the treated samples against *Escherichia coli* (*E. coli*, ATCC 25922) and *Staphylococcus aureus* (*S. aureus*, ATCC 25923) as ordinary pathogenic bacteria. Antibacterial activity was expressed in terms of the microorganisms reduction percentage (R) and calculated using the following equation:

Table 1 — Experimental conditions and tests results

Sample number	Zinc acetate % wt.	Tin tetrachloride % wt.	Sodium hydroxide % wt.	Degradation %
1	-	-	-	1.12
2	-	-	1	3.87
3	-	-	2	4.21
4	-	-	3	5.35
5	-	-	4	6.51
6	1	-	1	23.43
7	1	-	2	25.82
8	1	-	3	28.67
9	1	-	4	30.08
10	2	-	1	39.21
11	2	-	2	52.95
12	2	-	3	58.94
13	2	-	4	61.24
14	3	-	1	40.00
15	3	-	2	55.34
16	3	-	3	63.57
17	3	-	4	64.26
18	4	-	1	41.98
19	4	-	2	58.50
20	4	-	3	63.11
21	4	-	4	66.84
22	1	1	1	24.88
23	1	1	2	29.56
24	1	1	3	42.11
25	1	1	4	48.85
26	2	1	1	43.79
27	2	1	2	60.49
28	2	1	3	72.66
29	2	1	4	75.21
30	3	1	1	46.91
31	3	1	2	70.95
32	3	1	3	88.10
33	3	1	4	90.21
34	4	1	1	47.57
35	4	1	2	71.79
36	4	1	3	94.01
37	4	1	4	94.13

$$\text{Microorganisms reduction } (R), \% = [(A\text{-}B)/A] \times 100 \quad \ldots (2)$$

where A is the number of microorganism recovered from the inoculated treated test specimen incubated over 24 h; and B, the number of microorganism recovered from the inoculated treated test specimen immediately after inoculation (at '0' contact time).

For cytotoxicity test of the treated polyester fabrics with zinc stannate nanoparticles, normal primary human skin fibroblast isolated from the dermis of neonatal foreskins was used. Cell culture was performed at a 37 ˚C and 5% CO_2 condition using Dulbecco's modified Eagles medium (DMEM, Biochrom, Germany) supplemented with 10% fetal calf serum (FCS, Biochrom, Germany). Cells of the third passage were used and seeded in a 96-well microplate at a density of 20000 cells per well and incubated for 48 h. Then, the polyester fabrics (1 inch ×1 inch) were soaked in 2 mL culture medium for 24 h. The cultured medium with leaching substance was added to the cells and incubated for 24 h. The test samples were removed from the cell cultures, and the cells were re-incubated in fresh medium. After incubation for 24 h, the cell viability was determined by means of the MTT assay. At least three data were averaged. UV-blocking activities of the samples were also evaluated according to AATCC 183-2004 (transmittance or blocking of erythemally weighted UV radiation through fabrics).

3 Results and Discussion

3.1 FTIR Analysis

The treatment of polyester fabric with alkali solution is a resourceful method for imparting certain desirable properties to the polymer chain. Alkali hydrolysis causes a weight loss of the polyester fabric due to the cracks and voids created on the fibre surface [20]. Alkaline hydrolysis degradation in ultrasonic bath causes scission of the surface PET chains due to the ultrasound vibrations. This vibration facilitates alkali solution penetration on the fabric surface and introducing hydrophilic active sites, such as $–COO^-$ and –OH end groups, at the fibre surface by chain scission. This could be explained by degassing effect of ultrasound. A mass of gas entrapped in the inter-yarn and intra-yarn pores is removed by ultrasonic cavitations. Consequently, the boundary-layer thickness between the fabric surface and the alkali solution reduces to support the adsorption of alkaline molecules on the surface of the fiber [21]. The FTIR spectrum of the treated sample with sodium hydroxide (Fig. 1) indicates wide peaks related to hydroxyl and carboxyl groups. The related peaks to OH groups are obtained at around 3400 cm^{-1} and COOH groups at around 1600 cm^{-1}. These polar groups on the fabric surface are used as active centers and binding sites to nano-metal oxides [22].

3.2 SEM Images and EDS Analysis

The surfaces of the treated and untreated polyester fabrics are observed with scanning electron microscopy. The surface of the untreated polyester

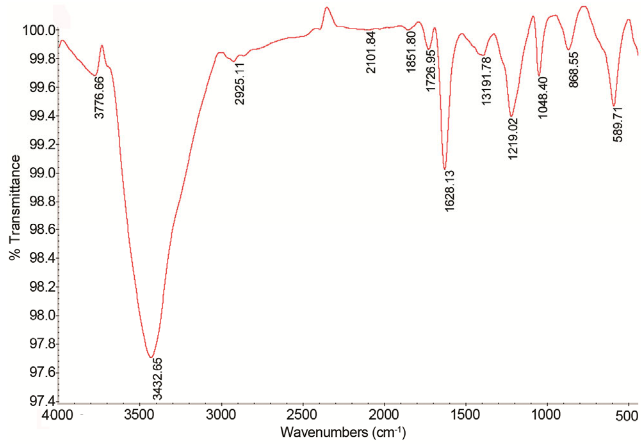

Fig. 1 — FTIR spectrum of treated polyester fabric with NaOH (Sample 4) in ultrasonic bath

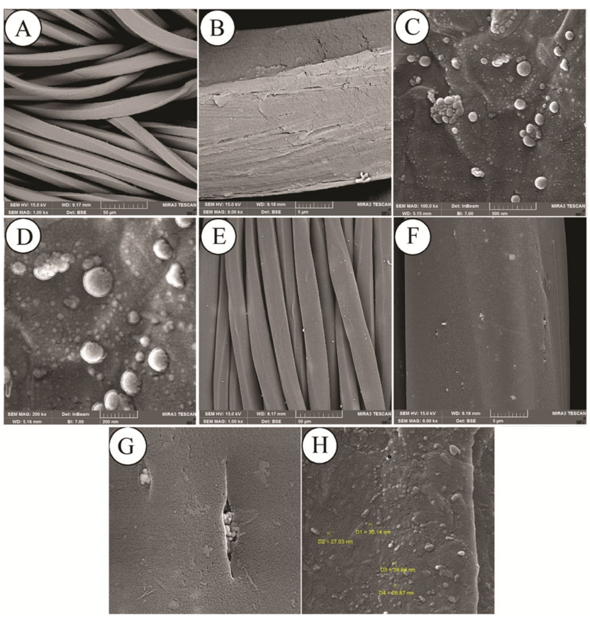

Fig. 2 — FE-SEM images of various polyester fabric samples (A) raw, (B) hydrolyzed polyester (Sample 4), (C and D) treated with ZnO nanoparticles (Sample 20), and (E-F) treated with Zn_2SnO_4 nanoparticles (Sample 36)

fabric is smooth [Fig. 2(A)]. After alkaline hydrolysis, the surface morphology of the fabric shows an obvious change [Fig. 2(B)]. It is well known that the hydrolysis of polyester fabrics with NaOH results in polymer chains cleavage and a decrease in both the fabric weight and fibre diameter, while showing cracks and pits on fibre surfaces [20].

FE-SEM pictures [Figs 2(C) and (D)] demonstrate coating of ZnO nano particles on hydrolyzed polyester fabrics. Possible reaction of synthesis of zinc oxide nanoparticles during alkaline hydrolysis of polyester fabric is shown in following equation:

$$[Zn(CH_3COO)_2 \cdot 2H_2O] + 2NaOH \rightarrow ZnO + 2NaCH_3COO + H_2O \quad \ldots (3)$$

Also, Fig. 2 (E)-(H) show the morphologies of the zinc stannate-treated fabric (Sample 36). It is thoroughly possible to recognize the nano particles on the surface of samples. The cracks which were formed by sodium hydroxide hydrolysis might be the sites for penetrating the nano particles into the fibre. The high-magnification pictures [Figs 2(G) and (H)] recognized the presence of tiny spherical particles on the surface of polyester fabrics. It is obvious that the zinc stannate nanoparticles coated on the polyester fabric are uniform and close-packed, which causes complete surface coverage of the fibres. Also, the average sizes of the particles are in the range of 25–40 nm.

Moreover, X-ray mapping images confirm the zinc stannate nanoparticles well distributed on the fabric surface (Fig. 3). It is marked that a uniform layer of nano Zn_2SnO_4 particles is located on the surface of fibres, resulting from ultrasonic irradiation. The presence of zinc and tin elements on the fabric sample in EDX spectrum [Fig. 3(a)] indicates well synthesis and loading of Zn_2SnO_4 nanoparticles on the polyester fabric. The preparation of zinc stannate nanoparticle is based on the following reaction [23, 24]:

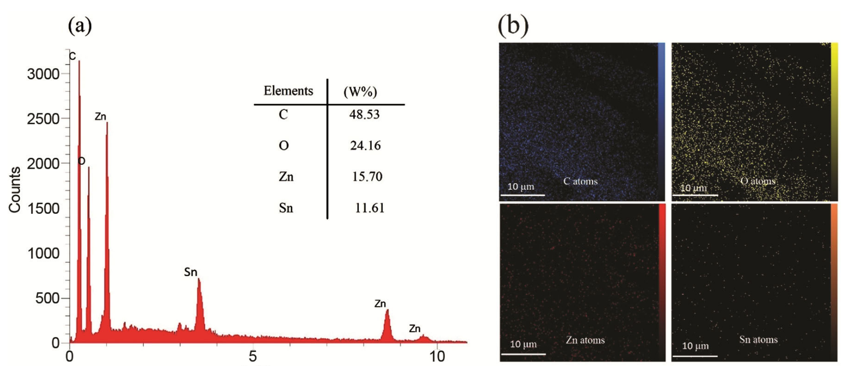

Fig. 3 — EDS spectrum (a) and X-ray mapping images (b) of polyester fabric treated with Zn_2SnO_4 nanoparticles (Sample 36)

$$2(Zn(CH_3COO)_2 \cdot 2H_2O) + SnCl_4 + 8NaOH \rightarrow Zn_2SnO_4 + 4NaCl + 4NaCH_3COO + 8H_2O \quad \ldots (4)$$

The presence of sodium hydroxide in the processing bath leads to hydrolysis of polyester. Zinc acetate in alkali media along with tin tetrachloride in presence of polyester fabric leads to *in-situ* sonosynthesis of Zn_2SnO_4 nanoparticles on the fabric surface. In effect of alkaline hydrolysis of polyester with sodium hydroxide, the polymer chains are shortened and the negative charge is created on the polyester surface. The zinc and tin cations can be attached to the negative parts of polyester chains obtained through hydrolysis [25]. The zinc and tin ions connected to the polyester chains working as nuclei then grow. Progressively produced Zn_2SnO_4 nanoparticles linked to the fabric surface. The strong driving force that is generated during the ultrasonic process causes the nanoparticles incidence to the surface of the fabric with more intensity and absorption on its surface [26].

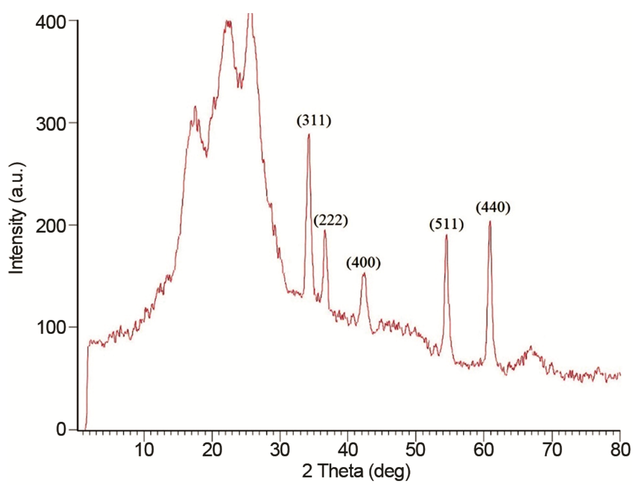

Fig. 4 — XRD pattern of treated polyester fabric with Zn_2SnO_4 nanoparticles (Sample 36)

3.3 XRD Spectroscopy

The X-Ray diffraction pattern of coated fabric with Zn_2SnO_4 nanoparticles (Fig. 4) confirms the successful synthesis and fabrication of zinc stannate nanoparticles on the hydrolyzed polyester substrate. The well-defined diffraction peaks at 2θ=34.2°, 35.8°, 41.6°, 51.6° and 60.4° indexing at (311), (222), (400), (511) and (440) planes of Zn_2SnO_4 with the cubic spinel structure relate to standard data with JCPDS file no. 24-1470. Also, the peaks at 2θ values of 17°, 23.1°, and 26.4° are attributed to polyester substrate.

3.4 Self-cleaning Effect

The self-cleaning property of polyester samples has been evaluated using the rate of decomposition of the methylene blue dye solution under sunlight irradiation. The results for polyester fabrics are presented in Table 1 and Fig. 5. For raw and hydrolyzed polyester fabrics, the methylene blue concentration change is negligible. Therefore, the raw and hydrolyzed polyester samples show no photocatalytic activity. Use of zinc acetate in impregnated bath leads to a more significant decrease in methylene blue concentration than polyester fabric, which demonstrates the successful deposition of the nano materials on the polyester fabrics. As seen in Fig. 5, the increase in concentration of sodium hydroxide in the impregnating bath increases the photo-degradation of methylene blue. This could be a result of the increase of polar groups, such as

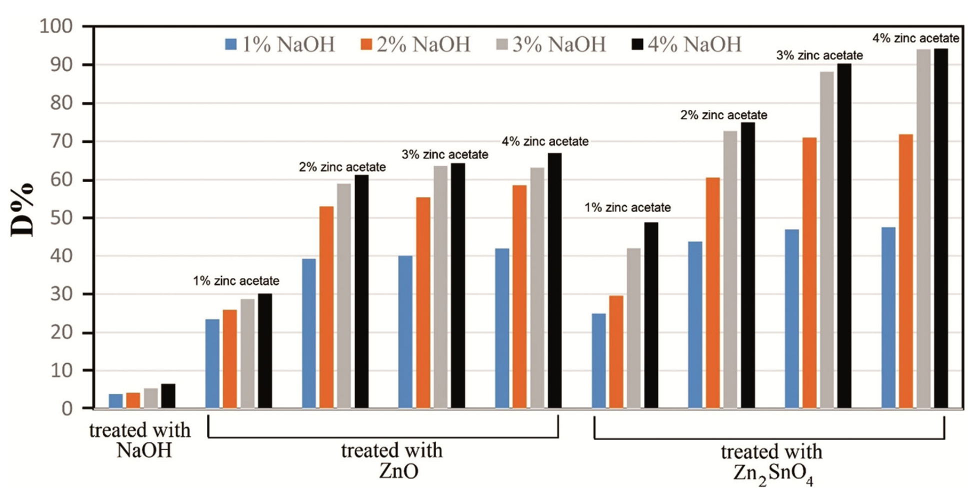

Fig. 5 — Comparative diagram of self-cleaning performance results of the polyester fabrics

carboxyl and hydroxyl groups on the surface of the fibres, which may favor the adsorption of nanoparticles on the polyester fabrics.

When the photocatalyst is illuminated by a light with energy higher than its bandgap energy, electron-hole pairs diffuse out to the surface of the photocatalyst. The created negative electrons and oxygen combine into $O_2^{\cdot -}$, and the positive electric holes and water generate hydroxyl radicals. This highly active oxygen species can oxidize pollutants [27]. The general mechanism of photocatalysis using semiconductors can be summarized in the following equations [28]:

$$ZnO \text{ or } Zn_2SnO_4 + h\nu \rightarrow e^- + h^+ \quad \ldots (5)$$

$$h^+ + H_2O \rightarrow H^+ + OH^\cdot \quad \ldots (6)$$

$$h^+ + OH^- \rightarrow OH^\cdot \quad \ldots (7)$$

$$e^- + O_2 \rightarrow O_2^{\cdot -} \quad \ldots (8)$$

$$O_2^{\cdot -} + H^+ \rightarrow HO_2^\cdot \quad \ldots (9)$$

$$(OH^\cdot, O_2^{\cdot -}, HO_2^\cdot) + \text{dye molecule} \rightarrow \text{Degradation products} \quad \ldots (10)$$

Based on the findings, the nano-Zn_2SnO_4 treated fabrics confirm more self-cleaning activity in comparison with nano-ZnO treated samples. Along the same lines, Zeng *et al.*[29] reported the photocatalytic degradation of methylene blue using hydrothermally synthesized Zn_2SnO_4 nanoparticles. The degradation rates were higher for the zinc stannate nanoparticle than for the commercially available Degussa P25 TiO_2 nanoparticles. The superior self-cleaning property of nano-Zn_2SnO_4 coated fabrics could be attributed to the advanced adsorption efficiency of molecules, the improved light absorption, and the unique electronic structure of the of zinc stannate nanoparticles [30, 31].

3.5 Antibacterial Property

Due to the resistance of many bacterial strains to various antibiotics, such as penicillin and ampicillin, antibacterial finishing of fabrics have become concerns of the textile industry. The antibacterial fabrics can be used in medical, healthcare, and hygiene materials, which provide adequate barriers against highly communicable bacteria. Antibacterial activities of raw and treated polyester fabrics have been assessed quantitatively by the AATCC agar diffusion test method 100 (Fig. 6). The raw polyester sample provides a suitable media for growth of bacteria, which is due to the fact that raw polyester does not have any antibacterial agent in its surface. For treated fabric with nano ZnO (Sample 20), R% against *S. aureus* and *E. coli* is more than 62% and 71% respectively. The ZnO treated polyester can inhibit the growth of bacteria possibly by two mechanisms. The reactive oxygen species, generated from zinc oxide excitation, damage the membrane of bacteria cell through oxidizing lipids and proteins. Another suggested mechanism is, Zn^{2+} ions can be released from the nanoparticles and may attach to the bacterial cell wall with negatively charged, resulting in rupture and death of cell [32].

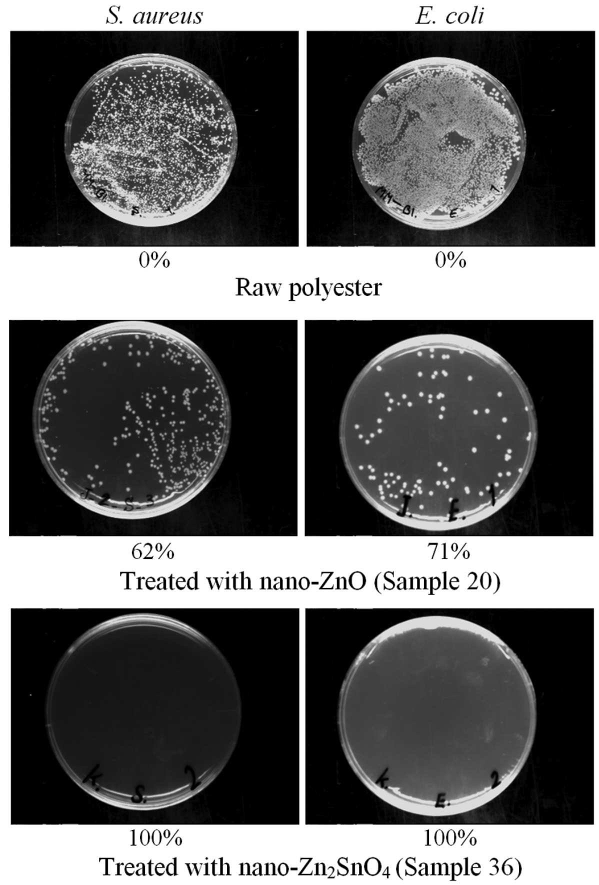

Fig. 6 — Antibacterial activity measurement of raw and treated polyester samples

Also, the treated fabrics with nano-Zn_2SnO_4 show very high percentage reduction in bacterial colony against both *S. aureus* and *E. coli*. The higher antibacterial activity of nano-Zn_2SnO_4 treated polyester than nano-ZnO treated sample is attributed to the effect of composite ions (Zn and Sn ions). This creates oxygen defects to damage the microbe synthesis activity, leading to the cell losing their ability of cell division, which may lead to the cell death of the bacteria [33-35].

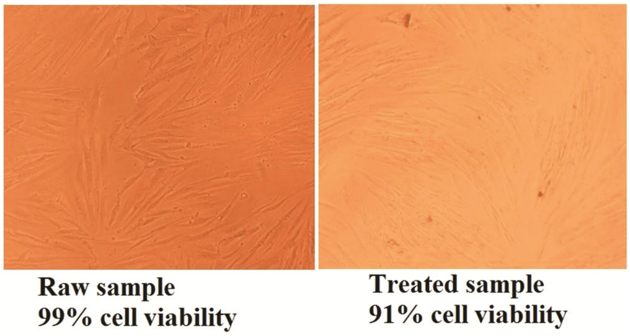

Fig. 7 — Viability and morphology of human fibroblasts cells incubation with the untreated and nano zinc stanate treated fabrics

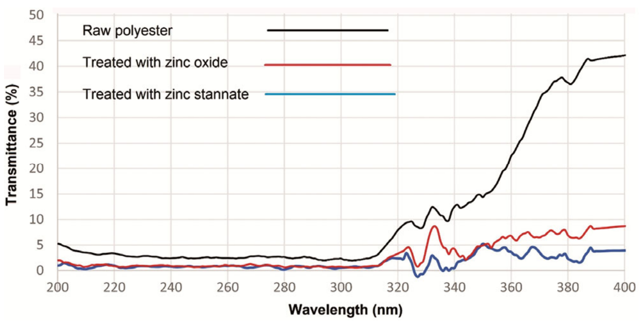

Fig. 8 — UV transmittance spectra of raw and treated polyester samples

3.6 Cytotoxicity

Cytotoxicity test of polyester fabric coated with Zn_2SnO_4 nanoparticles (Sample 36) and blank polyester fabric is performed by MTT assay. The viability and the morphology of human fibroblasts cells are shown in Fig. 7. Based on the obtained results, the treated fabrics with nano zinc stannate have no cytotoxicity, similar to the blank polyester fabrics. Moreover, the morphology of fibroblast cells in presence of the coated polyester fabric is normal, which is similar to the morphology of the blank fabric.

3.7 UV Protection

The UV transmittance spectra of raw polyester fabric, nano-Zno treated polyester and nano- Zn_2SnO_4 treated polyester samples (in the range of 200-400 nm) are shown in Fig. 8. It is observed that the transmission per cent of raw polyester is higher than that of treated fabrics. Within the two treated samples, nano-Zn_2SnO_4 treated sample has a little better UV blocking, but both of them have excellent UV blocking property due to the UV absorption ability of the nano-photocatalysts. The electronic structure of metal oxide semiconductors plays main role in mechanism of UV protection of treated sample. The high UV absorption of metal oxide semiconductors nanoparticles can be explained based on the solid band theory. Under UV radiation with a wavelength longer than photocatalyst band gap, photocatalyst can generate pairs of electrons and positive holes. These can either be recombined and release their absorbed energy as heat or trigger series of redox reactions. The former mechanism has been used in developing UV-protective coatings and films and later one has been employed as photocatalytic activity for different applications [36-38].

4 Conclusion

Novel zinc stannate multifunctional nanoparticles have been successfully fabricated on the surface of polyester fabrics using a sustainable sono-chemical approach, which can be easily implemented into industrial processes of textile finishing. The effect of alkaline hydrolysis of polyester fabric with sodium hydroxide in the sono-synthesis of Zn_2SnO_4 nanoparticles has been explored. FE-SEM and X-ray images, XRD and EDS patterns establish the formation of the zinc stannate nanoparticles on the polyester fabric surface. Functional characteristics of the nano-Zn_2SnO_4 treated polyester including self-cleaning, antibacterial and ultraviolet (UV) protection properties are found superior as compared to the treated polyester with nano zinc oxide. It is expected that the nano zinc stannate-treated fabrics can be used in medical textiles, protective garments, sportswear and smart textiles for healthcare benefits.

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