Development of Antibacterial Polyester Fabric by Growth of ZnO Nanorods

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ABSTRACT

ZnO nanorods were grown on polyester fabric by hydrothermal process. The seeding of fabric to grow ZnO nanorods was necessary because they did not grow without seeding. An air plasma treatment was carried out on polyester fabric to generate polar groups which could attach ZnO seeds. ZnO nanorods were grown on these seeds. The generation of polar groups was confirmed by XPS analysis. The morphology of nanorods was characterized with SEM and TEM. The quantity of ZnO deposited on fabric in the form of nanorods was estimated to be 5.6 % w/w by atomic absorption spectroscopy. Two Gram negative bacteria; *Escherichia coli* and *Pseudomonas aeruginosa* and a Gram positive; Staphyloccocus *aureus* were used for antibacterial activity evaluation by qualitative method. *E. coli* and *S. aureus* were used for quantitative assessment by using NF ISO 20743:2009 Transfer Method. It was noted that the functionalized fabric prevented the growth of bacteria not only on and below the fabric but also in the immediate proximities for all three bacteria. It was also observed that the fabric was more effective against Gram positive as compared to Gram negative bacteria. Moreover, it was shown that UV pre-activation of functionalized fabric enhanced the antibacterial activity.

INTRODUCTION

Microbial contaminations are harmful not only to human beings, but they also cause severe economic loss because of destruction of industrial and process equipment. The attachment of bacteria on any surface is the first step towards its contamination. Therefore, it is important to kill the bacteria as soon as they come in the vicinity of the surface or attach on it. On the basis of the mechanism of bacterial killing, the surfaces can be divided into two main categories; contact active biocidal surfaces and biocide releasing surfaces [1].

The contact active or non leaching surfaces are the ones which kill the bacteria when they attach on the surface. These surfaces are treated with compound such as quaternary ammonium compounds, chitosan, and polyhexamethylene biguanide [2, 3] which are chemically bound to the surface and do not release any active species into the environment around them. The biocide releasing or leaching surfaces are the ones treated with compounds which produce reactive oxygen species like hydroxyl radicals and release them into the environment. These species kill the bacteria before they attach themselves on the surface [4]. Classically, silver and TiO_2 have been used as biocide releasing antibacterial agents [4-6]. For example, textile surfaces were treated with TiO_2 for antibacterial activity [7] and Orthodontic ceramic brackets were coated with nanoparticles of TiO_2 to avoid the growth of *Streptococcus mutans* and *Candida albicans*[8].

Recently, ZnO has been used as an interesting antibacterial agent which represents a good alternative to TiO_2 [9-11]. The possibility to fabricate various types of nanostructures and to functionalize different surfaces also makes it a versatile material. The most commonly fabricated structures of ZnO include nanofilms [12], nano-flower like structures [13-16], nano-dumbbell shaped [17] nanobelts [18], nano-cages [19] and nanorods [20-22]. All these structures have been used for applications such asphotodetection, gas sensing, and dye sensitized.

Various nanostructures of ZnO have been used to functionalize different surfaces for antibacterial purposes. For example, ZnO nano-flower like structures was deposited on cotton fabric with the help of binder to develop an antibacterial textile [23]. The ZnO nanoparticles embedded in polyurethane films were prepared which have excellent antibacterial activity [24]. During recent years, a new method was developed to grow the nanorods of ZnO directly on the solid surfaces. For this purpose, seeds are deposited on the surface and nanorods are grown subsequently by a hydrothermal process. Nanorods were grown on solid substrate by seeding method to study the antibacterial activity against *Escherichia coli* and a Gram positive bacterium *Bacillus atrophaeus* [25]. The ZnO treated surfaces produce hydroxyl free radicals which have the ability to kill the bacteria not only when they attach on the surface but also in the proximity of the surface [26]. ZnO has been used as a food additive and is the most commonly used zinc source in the fortification of cereal-based foods. Because of its antimicrobial properties, ZnO has been incorporated into the linings of food cans in packages for meat, fish, corn, and peas to preserve color and to prevent spoilage [27]. It has recently been shown that ZnO is nontoxic for human cells [28].

The objective of the present study was to functionalize polyester fabric by growing zinc oxide nanorods using a hydrothermal method and to study the antibacterial activity of the functionalized fabric by quantitative and qualitative methods. For qualitative assessment, a Gram positive bacterium; *Staphylococcus aureus* and two Gram negative bacteria; *Pseudomonas aeruginosa* and *Escherichia coli* (ATCC 8739) were used whereas, *Staphylococcus aureus* and *Escherichia coli* (ATCC 8739) were used for quantitative assessment by NF ISO 20743:2009 Transfer Method.

MATERIALS AND METHODS

Functionalization of Polyester (PET) Fabric

A plain-woven PET fabric made of 9 -10µm fibers was used. The plasma treatment was carried out on clean and spinning oil free PET fabric to generate polar groups. The atmospheric air plasma device used in this study was from the "Coating Star" Systems (Ahlbrandt system). Dielectric Barrier Discharge (DBD) was created in air at atmospheric pressure. The following machine parameters were used: speed 2 $m.min^{-1}$, electrical power 750 W, and frequency 26 kHz. The sample was treated twice on each side.

To check the effectiveness of plasma treatment, the water contact angle (WCA) was measured by the tensiometric method on balance GBX-3S. The WCA on PET surface before treatment was 95° and decreased to 30° for plasma treated PET surface. This result confirms the creation of polar groups at the surface of PET. In order to check the presence of thin layer of oligomer if any, formed during plasma treatment, a tensiometric test was carried out. The surface tension of water was checked before and after immersion of a piece of plasma-treated PET fabric in water for 5 minutes. It was found that there was no change in surface tension of water before (72.9m $N.m^{-1}$) and after (72.8m $N.m^{-1}$). This showed the absence of oligomers on the surface of fabric, because if any organic impurities (oligomers) had moved from fabric to water, the surface tension of water would have been reduced.

Growth of Nanorods

The growth of nanorods was done with the seeding technique by using a hydrothermal method [21, 29, 30]. In the first step, ZnO seeds were prepared by using zinc acetate dihydrate (ACS reagent ≥ 98 % from Sigma Aldrich) 90 $mM.l^{-1}$ and sodium hydroxide 75 mM l^{-1}. Solutions of both chemicals were prepared in absolute methanol. Then, sodium hydroxide solution was added slowly to zinc nitrate dihydrate solution in round bottom flask. The mixture was refluxed at 60 °C for three hours. The plasma treated PET fabric was immersed in this seed solution for five minutes, then padded at nip pressure of two bars and dried at 120 °C for two minutes. This process was repeated five times to ensure uniform application of seeds on each fiber. Finally, seeded fabric was cured at 170 °C for eight minutes.

To grow the nanorods, 100 mM. l^{-1} zinc nitrate hexahydrate (reagent grade, 98 % Sigma Aldrich) and 100mM. l^{-1} hexamethylenetetramine solutions were used. Seeded PET fabric was placed in 250 ml solution of each reagent. The mixture was stirred at 90 °C for four hours. Then, the sample was washed in distilled water five times and dried at 120 °C for ten minutes.

CHARACTERIZATION

Nanorods

The generation of polar groups on polyester fabric by plasma treatment was characterized by XPS. SSX 100/206 photoelectron spectrometer from Surface Science Instruments (USA) equipped with a monochromatized micro focused Al X-ray source (powered at 20 mA and 10 kV) was used for this purpose. The angle between the surface normal and the axis of the analyser lens was 55°.

The morphologies of ZnO nanorods grown on PET fabric were characterized using a HITACHI S-3500N scanning electron microscope. To get the high resolution of SEM images, the samples were coated with an ultrathin layer of gold. To study the fine details of nanorods grown on PET fabric; morphology, crystal orientation, and lattice structure, a FEI Tecnai G2 20 transmission electron microscope (TEM) was used. The nanorods were scraped from the surface and used for this study.

In order to determine the amount of zinc oxide present on fabric in the form of nanorods, Atomic Absorption Spectroscopy PERKIN ELMER 1100 B was carried out. A piece of functionalized textile (about 20 cm^2) was weighed and then stirred in chlorohydric acid (1M. l^{-1}) for an hour. After complete dissolution of Zn^{2+}species, the obtained solution was filtered and diluted. Thanks to the factors of dilution and initial weight of the textile, it was possible to calculate an average of weight percentage of ZnO/PET.

Evaluation of Antibacterial Activity

Two Gram negative bacteria*: Escherichia coli* ATCC 8739 and *Pseudomonas aeruginosa* ATCC 27853, and a Gram positive bacterium: *Staphylococcus aureus* ATCC 6538 were used. Pre-cultures were performed at 37 °C for 24 h by the inoculation of Muller Hinton (MH) broth (5 ml) (Biocar Diagnostic, Beauvais, France) with a colony of the desired bacterium. A culture was then prepared by injecting 0.1 ml of bacterial suspension into 5 ml of MH broth. The overnight cultures performed at 37 °C were used to prepare cell suspensions of (1-3) x 10^6 Colony-Forming Units.ml^{-1} (CFU.ml^{-1}) into a fresh MH broth. These cell suspensions were used for qualitative or quantitative antibacterial experiments to study the antibacterial effect of the plasma treated polyester fabric by growing zinc oxide nanorods.

Qualitative Antibacterial Assessment

The Muller Hinton (Biocar Diagnostic, Beauvais, France) agar plates were inoculated with 1 ml of the dilution described above. Afterward, a swatch of the test sample (1.2 cm in diameter) was placed on the agar surface and incubated at 37 °C for 24 h before observation. At least 3 repetitions of each fabric were performed

Quantitative Antibacterial Assessment

Reduction of bacterial growth on the finished samples was estimated for the Gram negative bacteria *Escherichia coli* (ATCC 8739) and the Gram positive bacteria *Staphyloccocus aureus* (ATCC 6538) using the NF ISO 20743:2009 Transfer Method. The agar plates were inoculated with 1 ml of a Muller Hinton (Biocar Diagnostic, Beauvais, France) nutrient broth culture containing (1-3) x 10^6 CFU ml^{-1}. Afterward, a swatch of the test sample (3.8 cm in diameter) was placed on the agar surface and pressed down with a 200 g cylindrical weight for 60 ± 5 s. For each test, 6 samples were used. Three of them were removed from the agar surface and immediately analyzed for counting; the three other test samples were then removed from the agar surface, placed in a container with the transferred surface face up, and incubated at 37 °C for 24 h in a humidity chamber. For each fabric, 20 ml of neutralizing solution was poured on the test sample in a sterile bag and shaken vigorously using a stomacher shaker for 1 min on each side of the bag.

Serial dilutions were performed with sterilized in physiological saline (0.85 % NaCl) with tryptone (0.1 %) (Biocar Diagnostic, Beauvais, France), and the appropriate dilutions were plated onto agar medium as required by the ISO 20743:2007(F) standard and incubated at 37 °C for 24 h.

The antibacterial activity (A) of the functionalized polyester fabric was calculated as follows:

$$A = (\log C_t - \log C_0) - (\log T_t - \log T_0) \qquad (1)$$

Where

- A is the antibacterial activity value
- $\log C_0$ and $\log C_t$ are the decimal logarithm average corresponding to the number of bacteria obtained from the three untreated samples immediately after inoculation and after an incubating period of 24h respectively.
- $\log T_0$ and $\log T_t$ is the decimal logarithm average corresponding to the number of bacteria obtained from the three functionalized samples immediately after inoculation and after an incubating period of 24 h.

RESULTS AND DISCUSSIONS

Nanorods

Figure 1-a shows the SEM micrograph of a sample on which nanorods were grown without seeding. They did not grow on the surface. There were few nanorods deposited on fibers which grew in the solution. Then, the seeding method was adopted. In this method, ZnO seeds were deposited on the surface which provided the sites for the uniform growth of nanorods [29, 31]. For this, the fabric was seeded and nanorods were grown which grew only on scattered places. Polyester is hydrophobic and does not have

any polar groups which can attach the seeds. Therefore, the seeds were removed from fabric during the growth process. To attach these seeds on fabric, polar groups were generated by plasma treatment. The *Figure 1-b* shows the micrograph of nanorods grown on seeded fabric after plasma treatment. Highly oriented and perpendicular to fiber surface nanorods grew all over the surface. This uniform growth is attributed to the attachment of seeds. The XPS analysis of fabric surface before and after treatment confirmed the generation of polar groups *Table I*. It shows that O/C ratio was increased after plasma treatment due to the generation of oxidized carbons (C-O and -(C=O)-O).

The plasma treatment had very minor effect on mechanical properties of fabric and also there was no weight loss of fabric which happened if the fabric was treated with caustic soda [30]. No cavitation was observed and the fabric retained its tensile strength after plasma treatment (strength retention > 95 %).

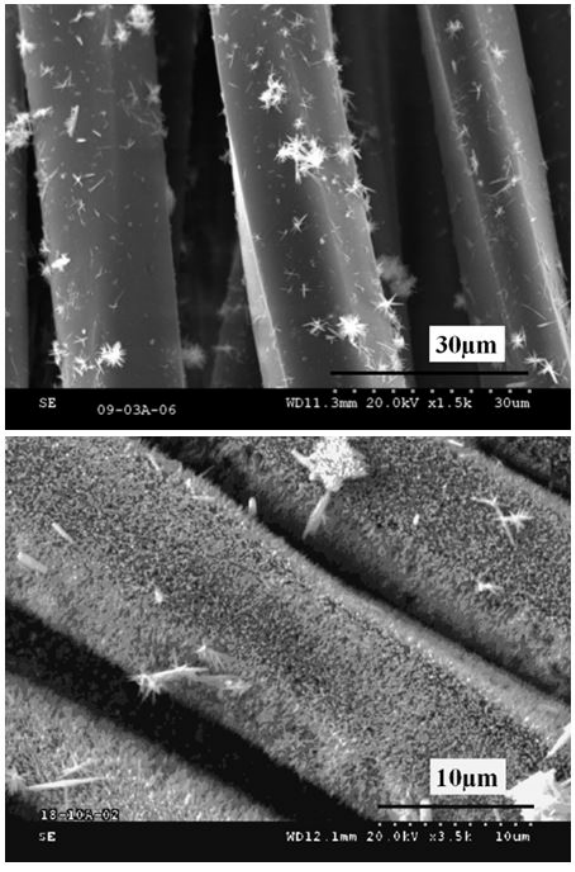

FIGURE 1. SEM image of nanorods grown; (a) on PET fabric without seeds. (b) on seeded PET fabric after plasma treatment.

TABLE I. Chemical composition of PET fabric before and after plasma and after plasma treatment determined by XPS.

Sample	-(C=O)-O-	-C-O-	-C-C-
	288.8eV	286.3eV	284.8eV
PET	14.68%	25.71%	59.60%
Plasma treated PET	21.70%	28.64%	49.66%

The TEM images are presented in *Figure 2*. These images were made after scraping the nanorods from the fabric. The *Figure 2-a* shows that nanorod grows in taper shape; its diameter at the bottom is about 100nm which decreases to 50nm at the top. It also confirms that the nanorods are highly crystalline *Figure 2-b*. The selected area electron diffraction SAED shows that the nanorods grow as mono crystals with hexagonal unit cell (wurtzite).

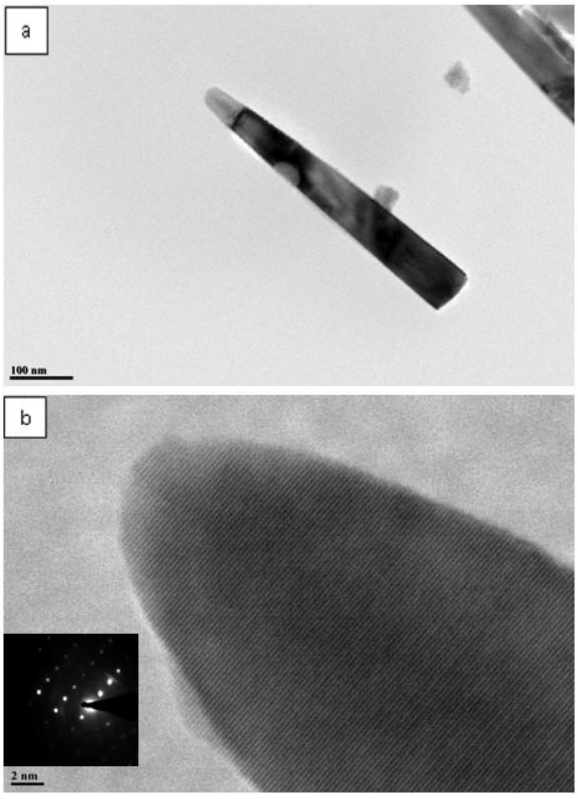

FIGURE 2. (a). TEM images of nano rod (b). high resolution of TEM image and saed in inset.

The atomic absorption spectroscopy gave mass concentration of Zn species present in acidic aqueous solution used to dissolve ZnO deposited on fabric. An average of 5.6 % w/w Zn was calculated.

ANTIBACTERIAL ACTIVITY

Qualitative Assessment

The qualitative antibacterial activity of functionalized fabric was evaluated by using one Gram positive; *S. aureus* and, two Gram negative bacteria; *E. coli* and *P. aeruginosa*. The functionalized and as-received samples were placed on agar plates containing these bacteria. After 24 hours of incubation in the dark, it was observed that the bacteria grew below, on, and in the immediate proximities of untreated samples but the functionalized samples inhibited their growth not only on the top and below but also in their vicinities. This phenomenon was observed more remarkably for *S. aureus* than other two bacteria. The antibacterial activity was approximately same for both Gram negative bacteria; *E. coli* and *P. aeruginosa*.

The inhibition of bacteria above and below the functionalized samples can be due to both physical and chemical reasons. ZnO has + 24 mV zeta potential at 7.2 pH whereas the surfaces of bacteria are negatively charged. The bacteria attach on ZnO surface due to electrostatic interaction [32]. If the size of ZnO is very small like in our case, the tips of the nanorods are around 50nm, they can penetrate the cells membranes leading to their disintegration and malfunctioning of the permeability barrier [33] which cause the death of bacteria. The second important reason for bacterial growth inhibition by ZnO is the production of reactive oxygen species (ROS) which includes $^{\cdot}OH$, H_2O_2 and O_2^{2-}. As the bacteria carry negative charge on the surface, therefore, the penetration of O_2^{2-} seems impossible but the hydroxyl radical and hydrogen peroxide can penetrate into the cell membrane which leads to the death of bacteria.

The functionalized samples inhibit the bacterial growth in their immediate proximities as well. To understand whether this inhibition is due to leaching of Zn^{2+}, the neutralizing solutions in contact with textile were collected after the bacterial extraction of the samples and the atomic absorption spectroscopy was carried out to determine the presence of Zn. It was found that it contained 0.0473 ± 0.0042 $g.l^{-1}$ of Zn. This shows that in bacterial culture, ZnO dissolve and generate Zn^{2+} ions which attach on the bacterial surface and prevent the growth of bacteria in the vicinities of the functionalized samples.

To study the effect of UV pre-activation on antibacterial activity of functionalized fabric, the samples were exposed to UV light for 24 hours. Then antibacterial activity was tested for *S. aureus*. It was noted that the area of immediate proximity of the sample where the growth of bacteria was prevented, had increased. In order to estimate the mortality of bacteria by the functionalized fabric and the effect of UV pre-activation on this mortality, quantitative evaluation of antibacterial activity was carried out.

Quantitative Assessment Under Laboratory Conditions

The quantitative assessment of the antibacterial activity of the functionalized fabric was performed on *Staphyloccocus aureus* and *Escherichia coli*. As both the Gram negative bacteria showed similar antibacterial activity in qualitative test. Therefore, a second Gram negative bacteria (*P. aeruginosa*) was not used for quantitative assessment. *Figure 3* shows the cell count of *S. aureus* and *E. coli* obtained from control and the functionalized fabrics after their contamination (t = 0) and after their incubation period of 24 h (t = 24 h).

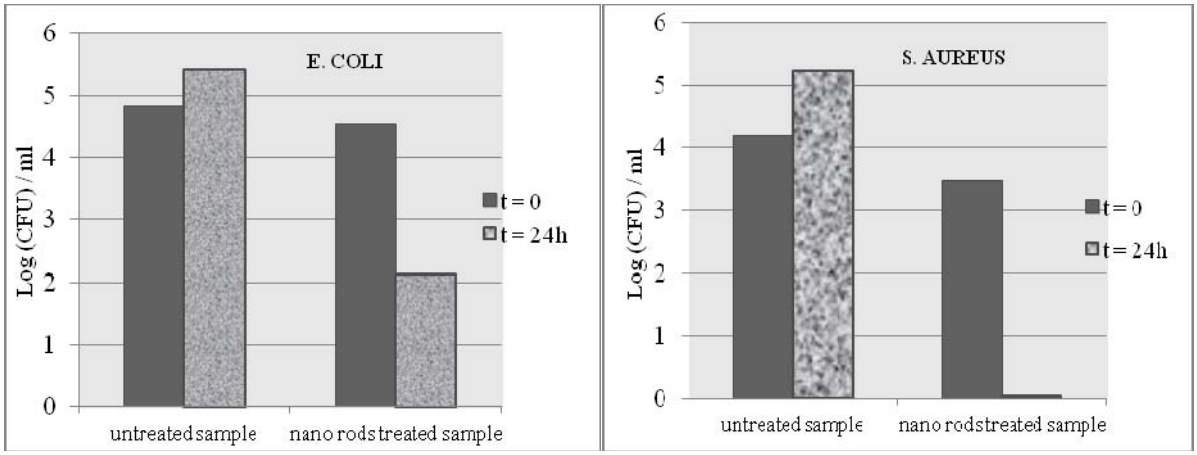

FIGURE 3. Counting of *S. aureus* and *E. coli* population directly after inoculation (t = 0) and after an incubating period of 24 h (t = 24 h) on control and functionalized fabrics.

Our findings show that the antibacterial activity of functionalized fabric is much higher on *S. aureus* as compared to *E. coli.* The cell enumeration of *S. aureus* from the contaminated functionalized sample shows a decrease of the initial cell count from 3.5 $CFU.ml^{-1}$ to up *ca* 0 $CFU.ml^{-1}$ after 24 h of incubation, under dark conditions. However, *S. aureus* enumeration carried out on the control fabric shows an increase of the cell count of *ca* 1 log $CFU.ml^{-1}$ after 24 h of incubation, under dark conditions. The results obtained for *E. coli* show a decrease of the initial cell count from 4.5 log $CFU.ml^{-1}$ to 2 log $CFU.ml^{-1}$ after 24 h of incubation, under dark conditions. The cell count performed on control fabric show an increase of *E. coli* cell count from 4.7 log $CFU.ml^{-1}$ to 5.3 log $CFU.ml^{-1}$. The calculation of the antibacterial activity of the functionalized fabric on the basis of the NF ISO 20743:2009 (Eq. 1) gave a value of 4.5 against *S. aureus* and 3 against *E.coli* The percentage cell death of the bacteria was calculated by using the following formula;

$$\text{Percentage mortality} = (\log T_t - \log C_t) \times 100 / \log T_t \qquad (2)$$

48 hours UV pre-activation respectively. The percentage cell death increased from 96 % to 99 % after 24 hours of UV activation.

Due to UV illumination, the electron present in the valence band jumps to the conduction band which causes generation of positive hole and electron pair. Some of the positive holes react with the lattice oxygen leading to the formation of surface oxygen vacancies. Similarly, the electrons generated react with surface metal (Zn) to reduce it from Zn^{++} to Zn^{+} to form defective sites [37]. The water and oxygen may compete to adsorb on them. The defective sites are kinetically more favorable for hydroxyl adsorption than oxygen adsorption [38]. After UV illumination, the surface of ZnO will be rich in hydroxyl which is one of the reactive oxygen species and has ability to kill bacteria.

The antibacterial activity on *S. aureus* presented in *Figure 3* is higher (A=4.47) than the one presented in *Figure 4* for the sample (A=4.29) placed in complete dark. The difference is 34 % if represented without log. According to norm, the acceptable tolerance is 20 %. Therefore, this difference is really significant. Although the samples used were the same, the one used to study the UV effect was first put in complete darkness before carrying out the test; whereas, the other was used as prepared without putting it in darkness. It must have been activated by the sun light and the normal tube light because both contain some amount of UV light.

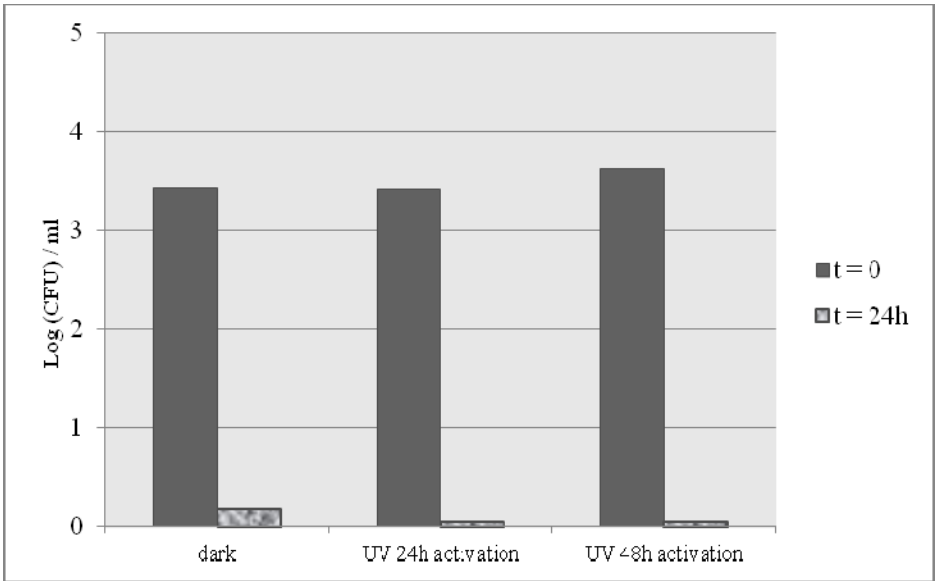

FIGURE 4. Counting of *S. aureus atcc 6538* population (in decimal logarithm average per milliliter) directly after inoculation (t=0) or after an incubating period of 24h (t= 24 h) on functionalized fabrics conserved in dark, after 24 h of UV activation, and after 48 h of UV activation.

CONCLUSION

Polyester fabric was successfully functionalized with ZnO by growing nanorods on plasma treated polyester fabric. This fabric showed the ability to kill the bacteria and to prevent its growth. The antibacterial activity of functionalized fabric for *S. aureus* was much higher than for *E. coli*. The mortality of *S. aureus* and *E. coli* after 24 hours incubation was 99 % and 53 % respectively. The effect of UV pre-activation was studied and it was found that the antibacterial activity was increased after 24 and 48 hours of UV activation.

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