



## New Multifunctional Properties of Synthetic Fabrics coated by Nanoparticles

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### ABSTRACT

Nanoparticles (<100 nm) of zinc oxide (ZnO), Titanium dioxide (TiO<sub>2</sub>) and aluminum oxide (Al<sub>2</sub>O<sub>3</sub>) are used for finishing and to add new multifunctional properties to synthetic fabrics using pad dry-cure method and a good washing. Polypropylene (PP) and polyester (PET) fabrics are activated by Dielectric-barrier discharge (DBD) plasma (or non-activated) and then dispersed in a colloidal solution of isopropanol and nanoparticles ZnO, TiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> (2 or 4% wt/v, on-weight-fabric (owf)). Scanning electron microscope (SEM) and Fourier transform infrared spectroscopy (FTIR) are used for characterization and confirmation of the presence of nanoparticles onto fabrics. Ultraviolet protection factor (UPF) and antibacterial activity for these fabrics against *Staphylococcus aureus* (*S. aureus*) and *Klebsiella pneumonia* (*K. pneumonia*) are evaluated and are compared to the treated fabrics with or without activation in DBD plasma.

**Keywords:** Nanoparticles, Synthetic fabrics, DBD plasma, SEM, FTIR, UPF factor, antibacterial activity.

### INTRODUCTION

Nowadays, nanotechnology is a promising technique for textile finishing. Loading of nanoparticles is used to add new functionality or change the properties of synthetic fabrics. NanoTiO<sub>2</sub> has UV resistance and self-cleaning properties. Nano Ag and nano ZnO have antimicrobial properties and UV-blocking activities. Zinc oxide is actually one of the best bio-friendly absorber of UV radiation.<sup>1-5</sup>

Literature review revealed that the use of atmospheric pressure electrical discharge corona plasma or else for fibres surface activation facilitates the loading of Ag nanoparticles from colloidal solution onto polyester and polyamide fabrics and thus enhances their antifungal activity against *Candida albicans*.<sup>6</sup>

Antibacterial effect of nano sized silver incorporated onto cellulosic and synthetic fabrics against *S. aureus* and *K. pneumonia* was maintained after many times laundering.<sup>7</sup>

Biological synthesized silver nanoparticles were incorporated on to fabrics, provided them sterile properties. Cotton fabrics bearing silver nanoparticles exhibited antibacterial activity against *S. aureus*.<sup>8</sup>

Antimicrobial polypropylene / nano Ag (PP/Ag) dressing was manufactured by melt extrusion and spinning technique. Application of PP/Ag dressing to wounds enhances complete wound healing.<sup>9,10</sup>

Effective textile treatment for application as wound dressing has been developed. Ease of silver deposition and excellent adhesion of the silver coating to fabric were distinctive characteristics of this technology. Antimicrobial activity of silver treated fabrics was assessed on different microorganisms, such as Gram

positive and Gram negative bacteria and also on fungal strains.<sup>11</sup>

Stain repellency, self-cleaning and excellent antibacterial activity were obtained from deposition of nanoparticles onto natural and synthetic fabrics using ultrasound irradiation.<sup>12-16</sup>

It was reported<sup>17</sup> that nano ZnO powder used in fabrics pretreatment had improved light fastness in sonicated dyes better than in non sonicated ones. Also in case of post treatment, better results were obtained for light fastness which increased UV protection and decreased the rate of fading.

Application of ZnO nanoparticles onto the surface of cotton, PET/cotton and wool fabrics were effective as UV protection.<sup>18,19</sup>

Nanoparticles (NPs) of TiO<sub>2</sub> treatment lead to produce wool fabric having excellent antibacterial and photocatalytic properties. In addition, NPs-treatment had no adverse effects on fastness properties of the functionalized dyed wool fabric.<sup>20,21</sup>

UV-blocking treatment for cotton fabrics was developed using the sol-gel method. A thin layer of TiO<sub>2</sub> was made on the surface of cotton fibers and the treated fabric showed improved protection against UV radiation with UPF factor greater than 50<sup>+</sup>. The treated fabric was also tested for washing fastness. The results showed that excellent UV protection rating of the treated fabrics can be maintained even after 55 launderings, indicating a high level of adhesion between the TiO<sub>2</sub> layer and cotton.<sup>22</sup>

Better bactericidal photo catalytic activity and excellent UV-protection were achieved by applying rutile nanorods onto cotton fabric.<sup>23</sup> Nano titanium dioxide/nano silver



composite emulsions gave antibacterial activity and improved odor elimination from fabric.<sup>24</sup>

In this investigation, synthetic fabrics of polypropylene (PP) and polyester (PET) fabrics were activated by DBD plasma (or non activated) and then dispersed in a colloidal solution of ZnO, TiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> nanoparticles at 80°C for one hour, padded and cured at 140°C for 10 min, well washed several times in water and dried at 100°C. Ultraviolet protection and antibacterial activity for these fabrics were measured and compared to non-activated ones.

## MATERIALS AND METHODS

### Materials

#### Fabrics

Nonwoven polypropylene (PP, 70 g/m<sup>2</sup>, thickness 0.58 mm) and woven polyester fabrics (PET, 155g/m<sup>2</sup>, thickness 0.33 mm) were used.

#### Nanoparticles and chemicals

Titanium dioxide, zinc oxide and aluminum oxide (< 100 nm) and nonionic wetting agent (Triton X-100) were purchased from Aldrich Co. and have been used as received. Isopropanol was bought from local market.

### Methods

#### Scouring of fabrics

Woven PET fabric was washed with 2gm/L nonionic detergent (Triton X-100), at 50°C for 1hr., liquor ratio 1:50 with continuous stirring. Nonwoven PP fabric was extracted with acetone for 30 min before plasma exposure to remove lubricant from the fabric.

#### Finishing of fabrics with nanoparticles

Nanoparticles (<100 nm) of ZnO, TiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>, inorganic compounds were used for the finishing of PP and PET fabrics.

Nanoparticles of a specific concentration (2 or 4% owf) and liquor ratio 1:30, in presence of 2 g/L nonionic wetting agent (Triton X-100) were dispersed under reflux in isopropanol with a magnetic stirrer. Then, the fabric was added and refluxed at 80°C for one hour, to assure the wettability of synthetic fabric. The fabric was padded to 100% (wt/wt) pick up followed by drying in air and then cured at 140°C for 10 min. The fabric was well washed at 60°C for 30 min in a Launder Ometer and dried at 100°C; the new added properties of the fabric were measured such as UPF factor and antibacterial properties.

#### Activation of fabrics by DBD plasma

Pretreatment of PP and PET fabrics (10 cm diameter) in DBD –plasma cavity at constant discharge conditions [time 20 min: (10 min on each side), electrode gap distance 2 mm, input voltage 10 KV, electric current 12 mA] and in oxygen gas. The plasma activated fabrics were

then treated with nanoparticles colloidal solution as described before.

### Analysis methods

#### Scanning electron microscope (SEM)

The fabrics were well washed with distilled water in a sonicator to remove the unattached particles of TiO<sub>2</sub> and then dried before SEM determination. The fabrics were measured on SEM Quanta FEG 250 (FEI Co.) working at 20 KV. Fabrics were coated with Carbon double face and fixed with stubs of Quanta holder and examined in low vacuum.

#### Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectrometer (Nicolet Magna-IR 560) was used to analyze the spectrum of the untreated and treated PP and PET fabrics with nanoparticles. The transmittance between 400 and 4000 cm<sup>-1</sup> were recorded.

#### Antimicrobial activity test

The antibacterial and antifungal studies of treated fabrics with Ag nanoparticles were accomplished in triplicates using standard methods (AATCC TM100).

The treated fabric was introduced into 20 ml nutrient broth and inoculated with the respective bacterial strains followed by overnight (24 hrs) incubation at 37°C. Growth of the bacterial strains was determined spectrophotometrically by measuring the optical density at 660 nm (OD<sub>660</sub>) in presence of the treated fabric against a blank of un-inoculated sterile medium. Similarly, the fungal strains inoculated into potato dextrose broth and incubated for 48 hours at 28°C in a shaker incubator followed by measurement of optical density at 450 nm (OD<sub>450</sub>) against a blank of un-inoculated sterile medium. Before recording the OD of the respective media after incubation, the culture tubes were shaken thoroughly in order to bring microorganisms into suspension.

Optical density is directly proportional to the number of microorganisms (bacteria or fungi) in the medium. The percentage of reduction of the microorganisms was expressed as follows.

$$R = (B - A)/B \times 100 \quad (\text{Eq.1})$$

R is the percentage of reduction of microbial population; B is the absorbance of the media inoculated with microbes and A is the absorbance of the media inoculated with microbes and treated fabric.<sup>25</sup>

#### Ultraviolet protection factor of fabrics

The ability of a fabric treated with nanoparticles to block UV light is given by the ultraviolet protection factor value (UPF). The measurement of UPF value was performed in UV/Visible Spectrophotometer 3101 PC with a software version, using an integrating sphere loaded with the fabric sample from 290 nm at an interval of 10 nm.

The measurements of the UV- penetration characteristics of the compressed fabric were carried out in the range of



290-400 nm using the UV penetration and protection measurements system. Before measurements, the fabric was conditioned at NTP for 24 hours. During the measurements, four scans were obtained by rotating the sample 90° each time and the spectral data were recorded as the average of these four scans.

The equation used by the software to calculate the UPF value of a flat tensionless dry fabric is as follows<sup>26</sup>:

$$UPF = \frac{\sum_{290}^{400} E(\lambda) \times \epsilon(\lambda) \times \Delta(\lambda)}{\sum_{290}^{400} E(\lambda) \times T(\lambda) \times \epsilon(\lambda) \times \Delta(\lambda)} \quad (\text{Eq. 2})$$

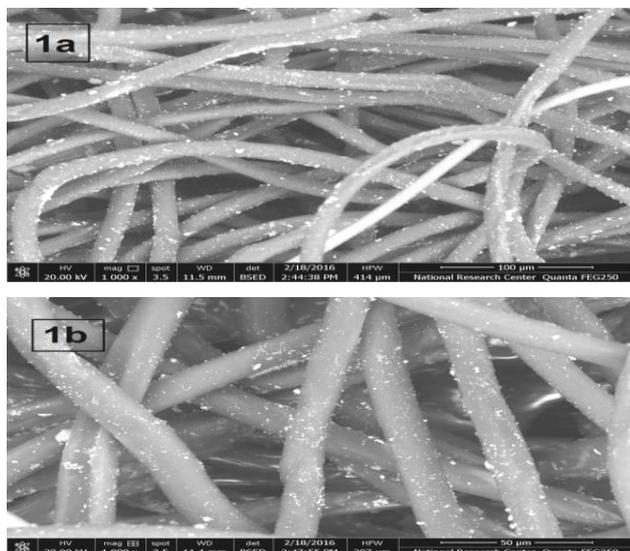
Where  $E(\lambda)$  is the solar irradiance ( $\text{Wm}^{-2}\text{nm}^{-1}$ ) measured;  $\epsilon(\lambda)$  is the erythema action spectrum;  $\Delta(\lambda)$  is the wavelength interval of the measurements; and  $T(\lambda)$  is the spectral transmittance at wavelength  $\lambda$ <sup>290</sup>. The percentage blocking of UVA (315-400nm) and UVB(290-315nm) was calculated from the transmittance data.

## RESULTS AND DISCUSSION

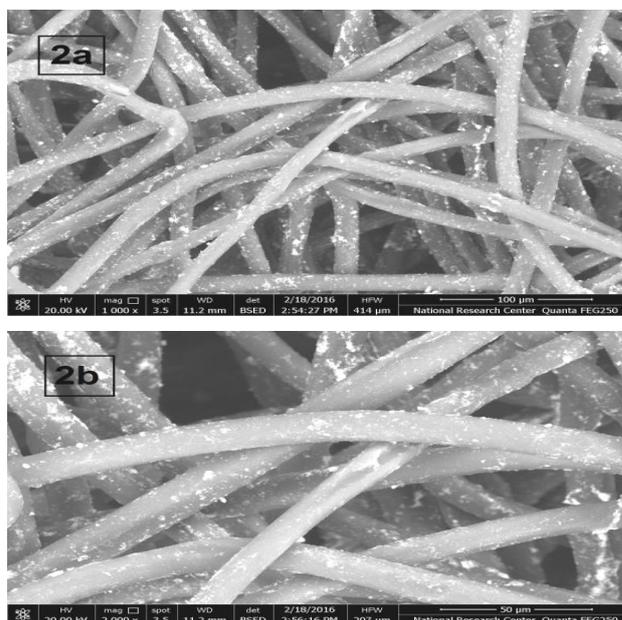
The fabrics treated with nanoparticles ( $\text{ZnO}$ ,  $\text{TiO}_2$  and  $\text{Al}_2\text{O}_3$ ) acquire new properties. These important properties include namely, UV blocking which are measured by UV protection factor (UPF) of the fabric spectrophotometrically. Fabrics with UPF greater than (40-50, 50+) are rated as excellent protective fabrics according to the cited specifications.<sup>27, 28</sup> At a given concentration of nanoparticles onto the fabric greater than 1 % owf, they provide antibacterial function to the fabric. The presence of hydroxyl and carboxylic acid groups content of fabrics are important for incorporation of nanoparticles onto the fabrics. DBD plasma is used to activate the surface of synthetic fabrics, especially polypropylene which has no active groups, therefore, facilitates loading of nanoparticles from colloidal solution onto polyester and polypropylene fabrics.

### Scanning Electron Microscope (SEM)

Activated fabrics using DBD plasma were characterized and confirmed by SEM and FTIR analysis.



**Figure 1:** SEM of PP treated with  $\text{TiO}_2$ (2% owf) with plasma, (1a): mag.1000x, (1b): mag.2000x



**Figure 2:** SEM of PP treated with  $\text{ZnO}$  (2% owf) with plasma, (2a): mag.1000x, (2b): mag.2000x

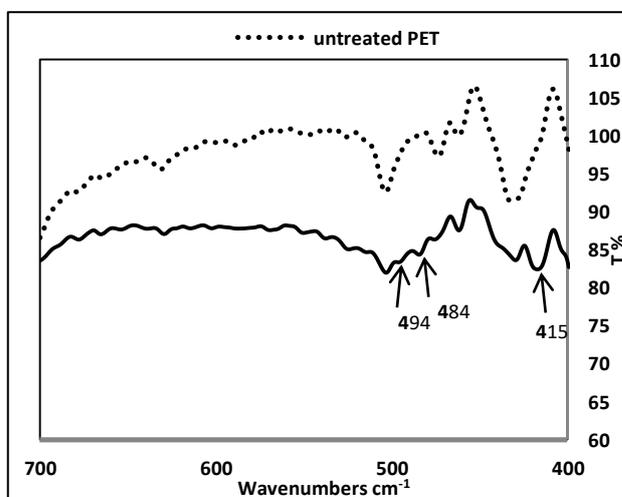
SEM images (Figures 1 and 2) confirmed the presence of nano  $\text{TiO}_2$  and nano  $\text{ZnO}$  on the surface of PP fabric with uniform and non coagulate dispersion.

### Fourier Transform Infrared Spectroscopy (FTIR)

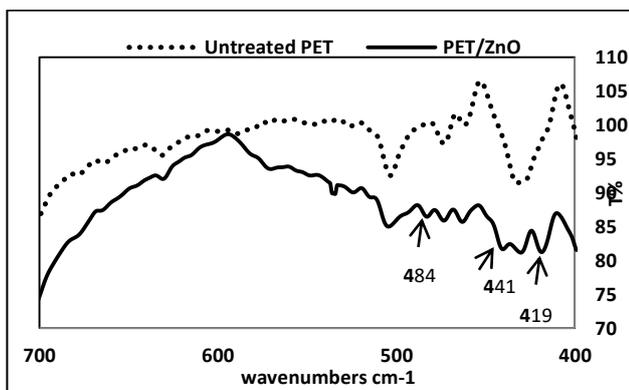
Figures 3a and 3b showed infrared analysis of untreated PET and that treated with nano  $\text{TiO}_2$  and nano  $\text{ZnO}$  at 400-700  $\text{cm}^{-1}$  respectively.

PET coated with nano  $\text{TiO}_2$  revealed new bands at 415, 484 and 494  $\text{cm}^{-1}$  compared to untreated PET. PET treated with nano  $\text{ZnO}$  has showed new bands at 419, 441 and 484  $\text{cm}^{-1}$ .

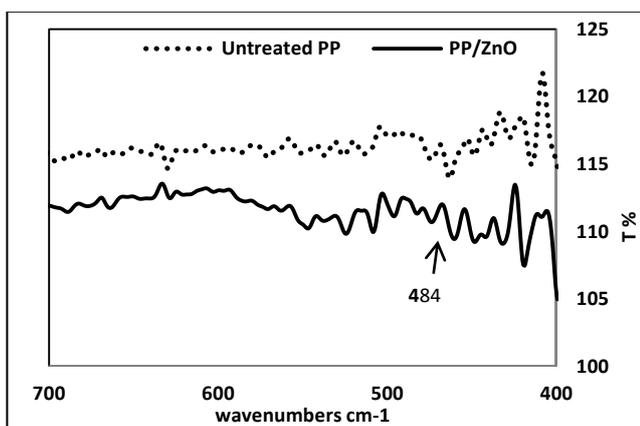
Also, IR spectra of PP coated with  $\text{ZnO}$  (Figure 3c) showed absorption band at 484  $\text{cm}^{-1}$ .



**Figure 3a:** FTIR of untreated PET and PET/ nano  $\text{TiO}_2$  (2% owf), with plasma



**Figure 3b:** FTIR of untreated PET and PET/ nanoZnO(2% owf), with plasma



**Figure 3c:** FTIR of untreated PP and PP/ nanoZnO(2% owf), with plasma

**Antimicrobial activity test**

Literature review revealed that antibacterial activity is an important test for textile.<sup>3-5</sup>

a) Disc diffusion method

Disc diffusion method was done for the three fabrics (PET / nanoTiO<sub>2</sub>, PET /nanoZnO and PET / nano Al<sub>2</sub>O<sub>3</sub>) against *S. aureus* and *K. pneumonia*:

- Only (PET/nanoZnO) gave inhibition zone (minimal 8-10 mm) against both *S. aureus* and *K. pneumonia*. The other two (PET/ nanoTiO<sub>2</sub>) and (PET / nano Al<sub>2</sub>O<sub>3</sub>) had no activity.

b) Optical density measurement

**Table 1:** Optical density measurement for PET/nanoTiO<sub>2</sub>, PET/nano ZnO and PET/nano Al<sub>2</sub>O<sub>3</sub>

Fabrics	Percentage reduction of microbial count (OD; as compared to control growth)	
	<i>Staphylococcus aureus</i> (G+)	<i>Klebsiella pneumonia</i> (G-)
PET/nanoTiO <sub>2</sub> (4%) owf	No effect	No effect
PET/nanoZnO (4%) owf	86.3%	91%
PET/nanoAl <sub>2</sub> O <sub>3</sub> (4%) owf	No effect	6.5%

Table 1 showed that only (PET / nanoZnO) gave excellent reduction of bacteria against *S. aureus* and *K. pneumonia* respectively. (PET /nano Al<sub>2</sub>O<sub>3</sub>) had no effect against *S. aureus* and a low bacteria reduction 6.5% on *K. pneumonia*

**Ultraviolet protection factor (UPF)**

Table 2 illustrated the UV protection level (UPF rates) according to the Australian/New Zealand specifications.<sup>27</sup>

**Table 2:** UPF rates, percentage relative transmittance and UV protective level

UPF range	Protection rates	UV-B Transmittance (%)
< 15	insufficient protection	> 6.7
15-24	Good protection	6.7-4.2
25-39	Very good protection	4.1-2.6
40-50, 50+	Excellent protection	≤ 2.5

Table 3 showed that PP fabric activated by plasma followed by treatment in colloidal solution 2% owf of ZnO and TiO<sub>2</sub> had increased the UPF to 38.1 (very good) and 17.9 (good protection) compared to 12.69 and 10.56 for PP fabric treated without plasma. While the activated PET fabric treated with ZnO and TiO<sub>2</sub> had excellent protection with UPF of 56.76 and 60.55, respectively.

**Table 3:** Activation of PP and PET fabrics with DBD plasma followed by treatment with colloidal suspension of ZnO and TiO<sub>2</sub> nanoparticles and UPF measurement

Fabrics	UPF level	Transmittance (%)	
		UV-A (315-400 nm)	UV-B (290-315 nm)
PP(untreated)	2.4 (insufficient)	49.03	40.86
PP/ nanoZnO (2%) owf	38.1 (very good)	6.93	2.38
PP/ nanoTiO <sub>2</sub> (2%)owf	17.90 (good)	8.12	5.34
PET (untreated)	32.43 (very good)	15.69	1.77
PET/ nanoZnO (2%) owf	56.76 (excellent)	5.43	1.56
PET/ nanoTiO <sub>2</sub> (2%)owf	60.55 (excellent)	5.05	1.36

Table 4 revealed that the untreated PET has a very good UPF protection 32.43. Treatment of PET with 2% owf ZnO and TiO<sub>2</sub> gave 56.07 and 76.22 excellent UPF grades while 4% owf ZnO and TiO<sub>2</sub> increased the UPF to 82.98 and 157 high excellent grades, respectively.

Treatment of PET fabric with nano Al<sub>2</sub>O<sub>3</sub> has a slight effect (35.20 and 36.76) UPF grades compared to (32.43) for the untreated fabric.

**Table 4:** Treatment of synthetic fabrics (PP and PET) with colloidal suspension of nanoparticles of ZnO, TiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>

Fabrics	UPF	Transmittance (%)	
		UV-A (315-400 nm)	UV-B (290-315 nm)
PP (untreated)	2.37 (insufficient)	49.03	40.86
PP/ nanoZnO (2%) owf	12.69 (insufficient)	15.08	7.35
PP/ nanoTiO <sub>2</sub> (2%)owf	10.56 (insufficient)	13.34	9.13
PP/nanoAl <sub>2</sub> O <sub>3</sub> (2%) owf	5.10 (insufficient)	29.03	17.73
PET (untreated)	32.43 (very good)	15.69	1.77
PET/ nanoZnO (2%) owf	56.07 (excellent)	7.03	1.40
PET/ nanoZnO (4%) owf	82.98 (excellent)	4.36	1.06
PET/ nanoTiO <sub>2</sub> (2%)owf	76.22(excellent)	2.81	1.18
PET/ nanoTiO <sub>2</sub> (4%) owf	156.90 (excellent)	0.77	0.64
PET/nanoAl <sub>2</sub> O <sub>3</sub> (2%) owf	35.20 (very good)	13.36	1.86
PET/nanoAl <sub>2</sub> O <sub>3</sub> (4%)owf	36.76 (very good)	14.50	1.63

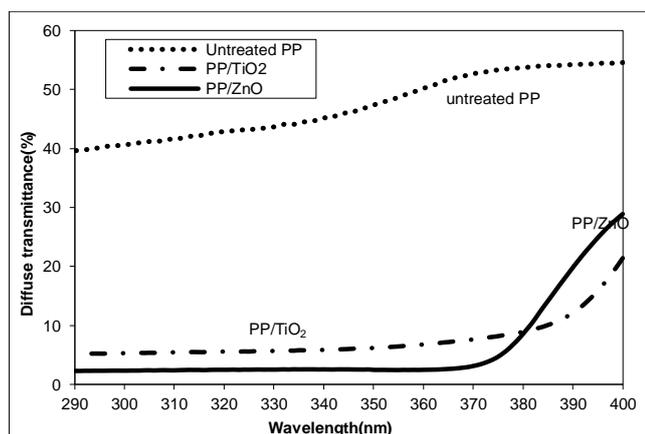
**Ultraviolet protection activity of PP and PET fabrics****Figure 4a:** % UV transmittance- wavelength of treated PP fabric with DBD plasma and (nanoTiO<sub>2</sub> and nanoZnO, 2%owf)

Figure 4a showed the percentage UV transmittance of PP/nanoZnO (2% owf) and PP/nanoTiO<sub>2</sub> (2% owf) in 290-380 nm is low 2-7%, which increased to 20% and 27% respectively in 380-400 nm. The transmittance of untreated PP is high, 40% at 290 nm and increased to 54% at 400 nm.

Figure 4c illustrated the percentage UV transmittance for PET/nanoTiO<sub>2</sub> at (4% owf) is low and does not exceed 3% at 290-400 nm. The percentage UV transmittance for PET/

nanoZnO at (4% owf) is low as 4% at 380 nm and increased to 20% at 400 nm.

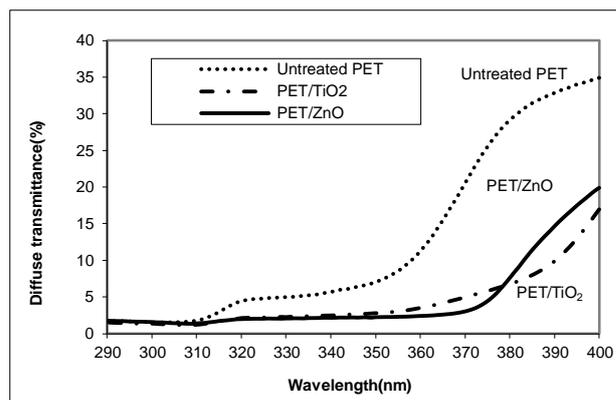
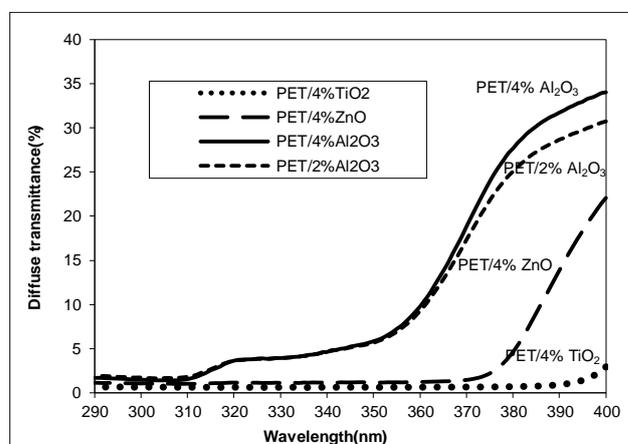
**Figure 4b:** % UV transmittance - wavelength of treated PET fabric with DBD plasma and (nanoTiO<sub>2</sub> and nanoZnO, 2%owf)

Figure 4b demonstrated the percentage UV transmittance of PET/nanoTiO<sub>2</sub> (2% owf) and PET/ nanoZnO (2% owf) in 290-380 nm is low 2-5% and increased to 15% from 380-400 nm. For untreated PET, UV transmittance is low 7% at 345 nm then increased to 20 % at 365 nm and 35% at 400 nm.

**Figure 4c:** % UV transmittance - wavelength of PET fabric treated with nanoparticles of TiO<sub>2</sub>, ZnO and Al<sub>2</sub>O<sub>3</sub>, 4%owf, without plasma

In case of PET/ nano Al<sub>2</sub>O<sub>3</sub> at (2% owf) and PET/nano Al<sub>2</sub>O<sub>3</sub> at (4% owf) they have the same transmittance 3-5% till 365 nm and increased then to 30% for PET/ nano Al<sub>2</sub>O<sub>3</sub> at (2% owf) and 34% for PET/ nano Al<sub>2</sub>O<sub>3</sub> at (4% owf).

**CONCLUSION**

Incorporation of nanoparticles onto PP and PET fabrics is an important and promising finishing process. These fabrics have acquired new multifunctional properties such as UV blocking and antibacterial properties.

Polypropylene and polyester fabrics were activated with DBD plasma (or without) and then dispersed in a colloidal solution of nano ZnO, nano TiO<sub>2</sub> and nano Al<sub>2</sub>O<sub>3</sub> at 80°C for one hour, padded, cured at 140°C for 10 min, well washed several times in water and finally dried at 100°C.

SEM and FTIR analysis were used for characterization and confirmation of the presence of nanoparticles onto fabrics. Ultraviolet protection factor and antibacterial activity of these fabrics were measured and were compared to the treated fabrics with nanoparticles and without activation in plasma.

PET fabric treated with nano ZnO had excellent antibacterial activity against *S. aureus* and *K. pneumonia*. DBD plasma treatment improved the binding of nanoparticles to the surface of PET and PP fabrics due to increase of the active groups on fabric surface. PP activated fabrics had better UV protection compared to nonactivated PP. In case of PET fabrics, activated and nonactivated fabrics, had almost the same UV-protection effect.

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