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Development of Multifunctional Cotton/nylon Blended Fabrics Using Nanoparticles of Different Metal Oxides

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Abstract

Two-bath dyeing is currently used to dye cotton/nylon blended fabrics. The cotton/nylon blended fibers were initially pre-treated independently with metal oxides based nanoparticles (NPs) before being dyed with acid and reactive dyestuffs. Two baths were used for dyeing of NPs metal oxides pre-treated cotton/nylon fabric with reactive and acid dyes. The morphological properties of the treated fabrics were explored by scan electron microscope and energy-dispersive X-ray. The impact of pre-treatment with NPs metal oxides on dye-ability, colorfastness, and physico-chemical features has been studied. Colorimetric strength (*K/S*) and CIE Lab were also explored. *K/S* values, washing, perspiration, and light fastness of the cotton/nylon sample treated with metal oxide nanoparticles were all higher. Antimicrobial activity was also examined against strains of ***K. pneumoniae***, ***S. aureus*** and ***Candida albicans***. The findings indicating that colored fabric had antibacterial potential due to the antibacterial property of NPs metal oxides. Aside from the NPs-treatment colored fabrics with excellent UV protection properties are produced.

Keywords: Cotton/nylon blend; Nanoparticles; Pre-treatment; Antibacterial; UV protection

1. Introduction

Functional clothing can be defined as textile materials with integrated properties, such as antimicrobial, antistatic, electrically conductive, fire-retardant, thermoregulating self-cleaning, and chromic textiles **[1-21]**. Nanotechnology has been regarded as the most significant rising technology in the world **[22-33]**. It is a key factor for creativity in a variety of industries. In textile blends (50:50), such as nylon warp stretch fibers with nylon/cotton or cotton weft for swimwear and narrow textiles for rainwear or work wear, color solidity and depth are more important. Nylon/cotton is still used in a variety of applications **[34-36]**. Because nylon is a polyamide, it has a lot of amide substituents in its polymer strands. It additionally has free amine substituents at the terminal of its polymer strands, in spite of the fact that the number of those free amine substituents is less than the number of carboxyl substituents, and the fiber has a negative charge unless it is in the suitable pH range **[37-51]**.

These amide and amine groups give phenomenal hydrogen bonding sites and are the most important factors in the dyestuff molecules' substance. Acid dyestuffs have an extremely low affinity for cellulose fibers, but cationic cotton can be dyed with them easily. The dye sites are the ammonium groups. Two-bath or one-bath two-step dyeing of cotton/nylon blended fabrics is the traditional dyeing approach (length of 3 hours) **[52-55]**. The goal of this research was to use nanoparticles, particularly metal oxides and nano-metals, to confer cellulose/nylon blended fabrics with multifunctional performances such as improving the hydrophilicity, antibacterial and UV protection properties of a cellulose/nylon blend fabric **[56-62]**.

2. Materials and Methods

2.1. Materials

2.1.1. Chemical reagents

Chemical reagents used for adjusting the pH were glacial acetic acid and anhydrous sodium carbonates.

- An anionic dispersing agent (Setamol WS, BASF) was used during dyeing process.

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- A nonionic detergent (Triton X-100) and sodium sulphate were used in soaping and washing fastness test.

2.1.2. Fabrics

Cotton/ nylon blended Fabrics weighing (per m^2 was supplied from Egypt textile Co. as a gift.

2.1.3. Dyestuffs

Two types of dyestuffs were supplied from the Indian Saya & Friends Co. (Ahmedabad) as follows:

- Reactive red 195 (C.I. Reactive Red 195, CAS 93050-79-4,1136.32, $C_{31}H_{19}ClN_7Na_5O_{19}S_6$) for cotton fabric **(Figure 1)**.
- Acid Red 150 (AR 150) for nylon fabric (**Figure 2**).

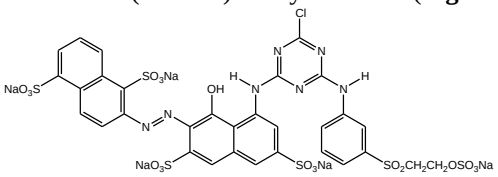

Figure 1. Chemical structure of Reactive Red 195

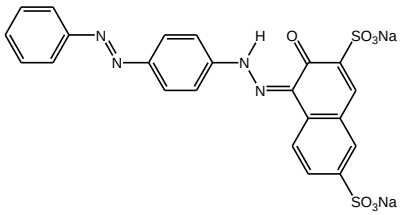

Figure 2. Chemical structure of Acid Red 150 (AR 150).

2.2. Methods

2.2.1. Preparation of metal oxide nanoparticles

2.2.1.1. Preparation of ZnO nanoparticles

Nanoparticles of zinc oxide were developed **[25, 26, 63-66]**. To accomplish a nucleation rate far higher than the growth rate, synthesis was carried out at a high level of super saturation (5.5 g), 98% of $ZnCl_2$ was dissolved in 200 mL of water at 90ºC in an oil bath. Then, over a duration of 10 minutes at 90ºC, 16 mL of 5 M NaOH (pellet min.99%) aqueous solution was applied drop-wise to the zinc chloride solution with delicate stirring. The particles were isolated from the supernatant scattering by sedimentation. To lower the concentration of NaCl below 6-10M, the supernatant solution was discarded and the resulting suspension was washed five times with purified water. The condensed suspension and the washing solution were diluted at a 1:10 ratio per time. A solution of $AgNO_3$ was used to ensure that all of the NaCl had been removed from the suspension. The sanitized particles were then peptized in an ultrasonic bath for 10 minutes at room temperature with 2-propanol (98 %). The peptization process is needed to break up the micro-agglomerates and release the zinc oxide nanoparticles. After that, the particles were gathered by centrifugation for 15 minutes at 6,000 rpm. Three times the washing process was carried out. The formation of ZnO is caused by thermal treatment of the particles at 250°C for 5 hours.

2.2.1.2. Preparation of TiO_2 nanoparticles:

Under the fume hood, 3.5 mL titanium tetra chloride ($TiCl_4$) was applied to 50 mL deionized water in an ice bath, supplemented by 35 mL ethanol and intense stirring for 30 minutes at room temperature. Drops of ammonium hydroxide were carefully applied, and a precipitate was obtained, To neutralise the titanium tetra chloride (TiC_{l4}) solution, ethanol, and deionized acid were added. The solution was allowed to settle for twelve hours after intense stirring. The precipitate was centrifuged after that. The collected precipitate was centrifugally isolated after being treated with deionized water before the chloride ion was extracted. The precipitate was then dried in an oven at 200°C for 4 hours to clear some of the absorbed water, yielding amorphous TiO_2. The collected amorphous TiO_2 was calcined at 400°C for four hours bit by bit. Finally, TiO2 nanomaterial powder was obtained. **[32, 51]**

2.2.1.3. Preparation of MgO nanoparticles:

Hydrothermal or sol–gel processes are commonly used to make MgO nanoparticles. The sol–gel process was used to make nano-MgO in this experiment. To make MgO nanoparticles, 100 g of $MgCl.6H_2O$ was dissolved in 500 ml distilled water in a 1L beaker, followed by adding 50 ml of 1N NaOH solution. After that, the solution was vigorously stirring for 4 hours to produce the magnesium hydroxide precipitates. To acquire the Mg $(OH)_2$ gel, the suspension was centrifuged at 3000rpm for 5 minutes, washed several times with purified water, and dried at 60ºC for 24 hours. MgO nanoparticles were formed by calcining the dried powder in air for two hours at 450ºC. **[67, 68]**

2.2.2 Pre-Treatment methods

2.2.2.1. Treatment of cotton/ nylon blended fabrics with ZnO nano particles

The samples were impregnated in a ZnO treatment bath (conc. 0.5 - 2 % wof), with 1:30 a liquor ratio and a dispersing agent, to suspend the ZnO in water and produce an uniform mixture. After padding, the samples were squeezed to 80% pick up and dried at 60°C. The pre-treated fabrics were cured for 10 minutes at 140 °C. Finally, the treated fabrics were washed at 60°C for 20 minutes then drying at room temperature.

2.2.2.2. Treatment of cotton/ nylon blended fabrics with TiO_2 nano particles

Fabrics pre-treated with TiO_2 nanoparticles by exhaustion process. The blended fabrics were pre-treated with four different concentrations of TiO_2 nanoparticles (0.5% - 2% wof) by using a wetting agent in the dyeing machine for 20 minutes at 80ºC. The treatment bath had a 1-10 liquor ratio. Following 20 minutes the treated cotton/nylon blended fabrics were cured for 10 minutes at 140ºC. After that, the

treated cotton/nylon blended fabrics were washed at 60°C for 20 minutes then drying at room temperature.

2.2.2.3. Treatment of cotton/ nylon blended fabrics with MgO nano particles

The exhaustion process was used to treat the fabrics with MgO nanoparticles. The fabrics were treated with four different concentrations of MgO nanoparticles (0.5 % - 2 % wof) for 20 minutes by using a wetting agent in the dyeing machine. The exhaustion bath's liquor ratio was 1:20. After 20 minutes, the treated fabrics were cured for 3 minutes at 120°C. Eventually, treated fabrics were washed at 60°C for 10 minutes then drying at room temperature.

2.2.3. Dyeing Method

Dyeing was performed at 100/130ºC in an infrared Dyeing machine (Roaches Co., England). After pre-treatment of cotton/nylon blended fabrics with ZnO, TiO_2 and MgO nanoparticles (0.5-2%), a specific concentration of dyeing bath (1-4%) was set up at pH 8 for cotton and pH 4 for nylon fabrics utilizing a liquor ratio of 1:40 at 100 ºC for cotton and 130 ºC for nylon fabrics. The dyeing procedure began at 40°C and eventually increased to 100/130°C and proceeded for 60 minutes. At last, fabrics were soaped in 2g/l non-ionic detergent (Triton X-100) at 50ºC for 30 minutes, then rinsed with warm water, and let it air dry.

2.2.4. Colorimetric results

Color strength (K/S) and CIE Lab were calculated according to Kubelkae-Munk equation using Hunter Lab DP9000 **[69-72]**.

2.2.5. Antibacterial testing

The antimicrobial effects of nanoparticle-coated fibers were tested utilizing standard AATCC TM-100 procedure **[73-80]**.

2.2.6. Colorfastness properties

Washing was assessed as stated in ISO105-C01(1989) using ATLAS launder-o-meter (Germany) at 50°C for 45 minutes utilizing 5g/L non-ionic detergent and a liquor ratio of 1:50. The composite specimen was taken out, rinsed under tap water, subjected to squeezing, and dried in the open air. It consisted of the test specimen as well as 2 adjacent fabrics that in contact of the primary sample. The colorimetric changes were evaluated utilizing a grey scale (wool and cotton) **[81-84]**. Light fastness was assessed by ISO105-B02(1988) using xenon light. To assess the degree of color resistance to light photo-degradation, samples were exposed to continuous light for 35 hours **[81-84]** .

2.2.7. Evaluation of ultraviolet protection

The ultraviolet protection factor (UPF) was studied using 3101 PC spectrophotometer according to previously reported procedure **[50, 85-89]**.

2.2.8. Surface morphology

Quanta FEG-250 SEM was applied at 20 kV to study the surface morphology of blank and treated fabrics, while their chemical contents were studied by TEAM-EDS spectral diagrams. The bending length was explored b Shirley stiffness teser under British 3356-1961 standard process.

3. Results and Discussions

3.1. Pretreatment and dyeing cotton/nylon blend fabrics

The major target of the current study is to investigate the pretreatment effects of cotton/nylon blend fibers with metal oxides nanoparticles (NPs) to improve their dyeing performance as well as generating multifunctional properties, such as ultraviolet protection and antimicrobial activity. Thus, TiO_2, ZnO and MgO NPs were synthesized according to previously reported procedures **[56, 90]**. The average diameter of the generated nanoparticles was determined by Image J software attached to scanning electron microscopy (SEM). **Figures 2-4** display SEM and EDX micrographs of both blank and treated cotton/nylon blend fabrics. Close testing of those SEM micrographs signifies average diameters of 8-20, 10-18 and 15-26 nm for TiO_2, ZnO and MgO, respectively. The cotton/nylon blend fabrics were pretreated with the synthesized meal oxide nanoparticles at different total contents, including 0.5, 1, 1.5 and 2% wof. The pretreated cotton/nylon blend fabrics were then subjected to dyeing with a mixed coloration system of dyestuffs, including Reactive Red 195 (RR 195) for cotton fibers and Acid Red 150 (AR 150) for nylon fibers. The dyed cotton/nylon blend fabrics subjected to fixation, washing and drying.

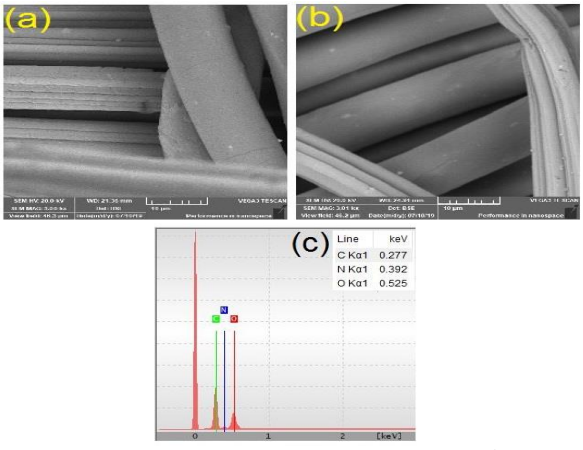

Figure 2. SEM images (a, b) and EDX diangram (c) of blank cotton/nylon blend fabric**.**

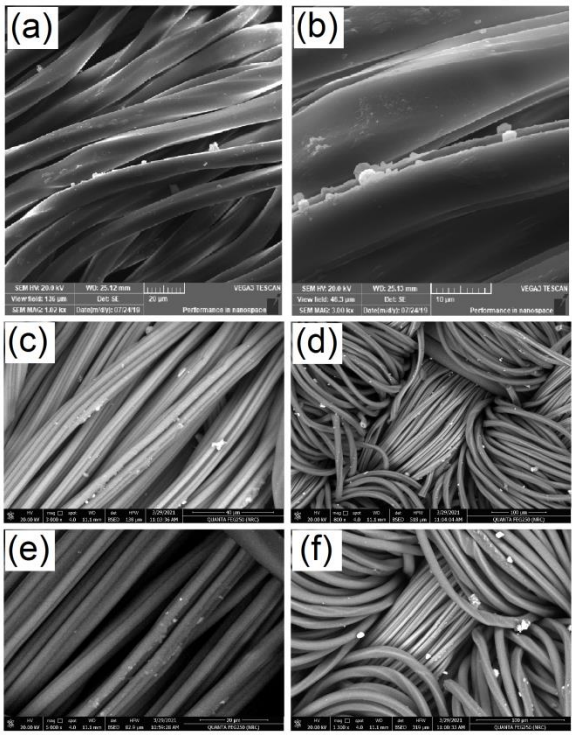

Figure 3. SEM images of dyed cotton/nylon blend fabrics pretreated with TiO_2 (a, b), MgO (c, e) and ZnO (d, f) NPs

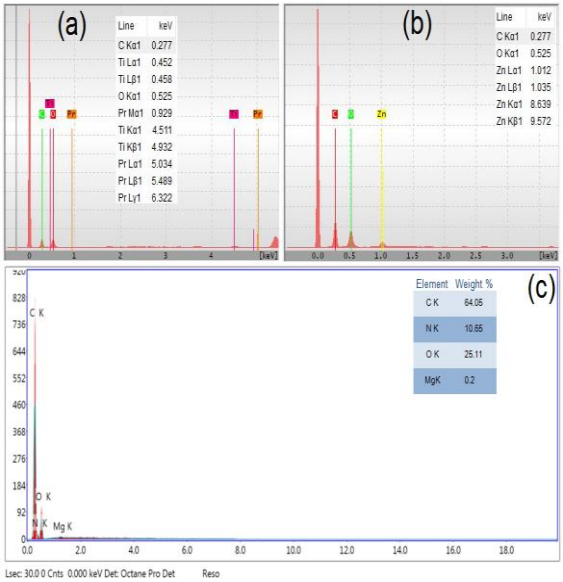

Figure 4. SEM images of dyed cotton/nylon blend fabrics pretreated with TiO_2 (a), ZnO (b) and MgO (c) NPs.

3.2. Evaluation of color strength

Nanotechnology has been concerned with nano-scaled materials with structures exhibiting considerably new and enhanced chemical, physical and biological properties, which can be attributed to their nano-scaled size **[57, 91-98]**. To optimize the most appropriate total content, different amounts of each metal oxide were applied in the pretreatment process of the cotton/nylon blend fabrics. The screening results of *K/S* are shown in **Table 1** and **Figure 5**. It is obvious that the color strength of the cotton/nylon blend fabrics depends on both of the type and nature of the metal oxide nanoparticles, as well as the applied concentration. The numerical values of *K/S* were monitored to follow the order of TiO2 > MgO > ZnO. It is also monitored that the higher *K/S* was established upon using TiO_2 NPs. Even at low concentration (0.5 % wof) of TiO_2 NPs, *K/S* was found to increase. Increasing the total content of TiO_2 NPs to values higher than 0.5% led to a slight decrease in color strength. This proved that the pretreatment of cotton/nylon blend fabrics with the lowest concentration of TiO_2 at 0.5% is even adequate to accomplish a significant increase in *K/S*. This could be attributed to Ti^{+4} ions, which results in an increase in the positive charges on the fabric surface leading to more ionic attraction with the reactive/acid anionic dyestuffs. However, increasing the total content of TiO_2 NPs higher than 0.5% results in a slight increase in *K/S* due to the increased repulsion forces between the increased concentration of Ti^{+4} ions. Hence, the optimized conditions for the pretreatment process of cotton/nylon blend fabrics with TiO_2 NPs were found to be at the lowest concentration of 0.5%. Moreover, the numerical values of *K/S* were monitored to follow the order of TiO2 > MgO > ZnO. Thus, the pretreatment of cotton/nylon blend fabrics with MgO NPs was found to accomplish higher *K/S* compared to ZnO NPs. The higher values of *K/S* for the fabrics pretreated with MgO NPs were observed at 1%. Increasing the total content of MgO NPs higher than 1% led to a slight decrease in *K/S*. This may be due to the negatively charged fabric surface which was deduced for ZnO NPs. Upon increasing the concentration of ZnO NPs, the *K/S* was found to increase demonstrating that the highest *K/S* was achieved at a total content of 1.5 %. All the pretreated samples were observed to accomplish higher *K/S* values compared to the untreated fabric.

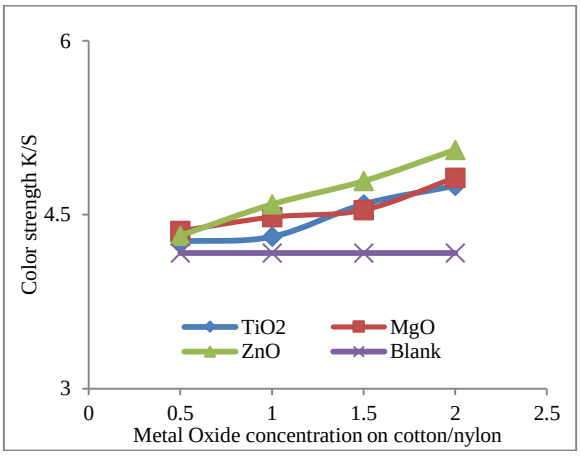

Figure 5. Effect of pre-treatment of cotton/nylon blended fabrics with different NPs metal oxides on K/S of dyed blended fabrics.

Table 1. Effect of pretreatment of ***cotton/nylon blended*** fabrics with different NPs metal oxides on *K/S* of blended fabrics.

Sample	Conc. (%)	*K/S* (Front)	*K/S* (Back)	*K/S* (average)
Blank	0	4.27	4.51	4.17
ZnO	0.5	4.32	4.57	4.32
	1	4.20	4.59	4.59
	1.5	4.79	5.47	4.79
	2	5.06	4.52	5.06
TiO_2	0.5	4.7	4.59	4.27
	1	4.31	4.21	4.31
	1.5	4.75	5.23	4.59
	2	4.28	4.42	4.75
MgO	0.5	4.81	4.36	4.36
	1	4.81	4.16	4.48
	1.5	4.02	4.54	4.54
	2	4.82	4.72	4.82

3.3. Antimicrobial properties

The antibacterial activity can be imparted to the pretreated cotton/nylon blend fabrics by incorporating functional agents into the surface of fibers **[58]**. As shown in **Table 2** and **Figure 6**, the antibacterial performance of the pretreated and dyed cotton/nylon blend fabrics depends on the type of the applied metal oxide NPs oxide. The numerical values of the antimicrobial properties were monitored to follow the order of TiO_2 > MgO > ZnO, while the dyed and untreated cotton/nylon blend fabric displayed the lowest antibacterial activity. Thus, the highest antibacterial activity was for the dyed cotton/nylon blend fabrics pretreated with TiO_2 NPs, which is preferable compared to other metal oxides as a result of its higher efficiency to prevent infection. This can be attributed to the photocatalytic activity of TiO_2 when exposed to light photons with energy equal to or higher than its bond-gap to excite electrons to the conductive band. Those excited electrons inside the crystal structure interact with the air-oxygen species leading to generating free-radical oxygen species to function as strong oxidizing agents. These free-radical oxidizing agents can then break down the cell-wall of the microorganism via oxidation-reduction process **[99-105]**. On the other side, ZnO is known by its ability to function through the induction of oxidation stress owing to generating reactive oxygen species leading to degradation of the cell membrane structure of the microorganism.

Table 2. Antimicrobial activity of nano-particle metal oxides impregnated fabrics.

Sample	Staph. (%)	Klebsiella (%)	Candida (%)
Blank	0	0	0
ZnO	45	90	50
TiO_2	98	99	60
MgO	63	91	55

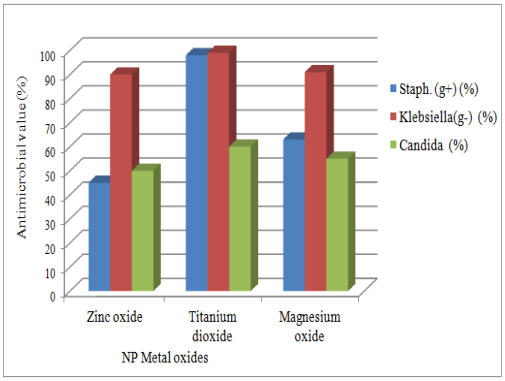

Figure 6. Antimicrobial activity of nanoparticle metal oxides impregnated fabrics.

3.4. Ultraviolet shielding

Table 3 represents the screening results of UPF values for both of untreated and pretreated cotton/nylon blend fabrics. The numerical values of the UPF properties were monitored to follow the order of MgO > ZnO > TiO_2, while the dyed and untreated cotton/nylon blend fabric displayed the lowest UV protection activity.

Table 3. UPF for wool and cotton fabrics treated with ZnO, TiO_2 and MgO NPs.

NPs	Conc. (5)	UPF (AS)	UPF (AATCC)	UVA	UVB
Blank	Without	4.6	4.6	2.6	1.1
ZnO	1.5	67.3	67.6	1.9	1.6
TiO_2	1.5	67.5	67.8	2.0	1.6
MgO	1.5	72.8	73.1	1.9	1.4

3.2. Colorfastness properties

The ultraviolet radiation shielding activity was explored by absorption spectroscopy. The transmission results were used to measure the ultraviolet protection factor (UPF). The cotton/nylon blend fabrics pretreated with TiO_2 , ZnO or MgO NPs and dyed with reactive/acid dyes acquired the highest *K/S*. The overall colorfastness properties are summarized in **Table 4**. For comparison, the properties of the dyed untreated cotton/nylon blend fabrics were also reported under the same conditions. In general, the overall colorfastness measurements were monitored between good to very good relying on both of nature, type and concentration of the applied metal oxides NPs utilized in pretreatment process. Nonetheless, the colorfastness for the pretreated cotton/nylon blend fabrics is almost equal or slightly better compared to untreated fabric. Both mechanical properties and durability were also explored to demonstrate that no adverse effects occurred to the treated fabrics compared to blank sample. The antimicrobial activity was maintained stable for treated samples after 10 washing cycles. Also, the bend length of the TiO_2 treated fabric was monitored at 2.83 and 2.95 cm for both of wrap and weft directions compared to 2.65 and 2.78 cm, respectively, for the blank fabric.

Table 4. Fastness properties of ***pre-treated cotton/nylon blended fabrics*** with NP metal oxides and dyed with reactive and acid dyes.

Metal salt concentration	Washing (with detergent)			Washing (with water)			Perspiration						Light
							Acidic			Alkali			
	St	St	Alt	St	St	Alt	St	St	Alt	St	St	Alt	
Blank (Without salt)	4	4-5	4	4	4	4	4	4-5	4	4	4-5	4	4-5
ZnO Conc. (%) owf	4	4-5	4	4	4-5	4	4	4-5	4	4	4-5	4	4
TiO_2 Conc. (%) owf	4	4-5	4	4	4-5	4	4	4-5	4	4	4-5	4	4-5
MgO Conc. (%) owf	4	4-5	4	4	4-5	4	4	4-5	4	4	4-5	4	4

4. Conclusions

In summary, we describe a novel technique for the fabrication of dyed cotton technical textiles with multiple functions. Some selected metal oxide nanoparticles, including TiO_2, ZnO and MgO, were synthesized and utilized to pre-treating cotton/nylon blend fabrics at different total contents before dyeing. Both scan electron microscopy (SEM) and energy-dispersion X-ray spectra (EDX) of the dyed fibers were investigated. The dyed cotton/nylon blend samples exhibited UV protection, antimicrobial activity and good fastness performance. Moreover, the colorimetric strength was monitored to reflect better enhancements on the treated fabric surface. In comparison to previously reported results on antimicrobial and UV-protection properties, the current method displayed a good method for an efficient production of functional textiles with antimicrobial and UV-protection properties.

5. Acknowledgements

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6. Conflicts of Interest

The authors declare no conflict of interest.

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