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Effect of nano TiO_2 pretreatment on functional properties of cotton fabric

S. B. Chaudhari [1], A. A. Mandot [1] and B.H. Patel [2*]
[1]*Department of Textile Engineering, The Maharaja Sayajirao University of Baroda,Vadodara, Gujarat, India.*
[2]*Department of Textile Chemistry, The Maharaja Sayajirao University of Baroda,Vadodara, Gujarat, India.*

Abstract—*In this study cotton fabric was pretreated with nano TiO_2 (<100nm) particles by Pad-Dry-Cure technique. The deposition of nano particles on the fabric was observed by SEM and elementally analyzed by oxford –Inca software using SEM. The treated fabric was analyzed for the change in their physical, biological and functional properties compared to the untreated cotton fabric. Very encouraging results in terms of improvement in antibacterial, UV protection and self-cleaning properties were found. Further the study was extended to evaluate the impact of nano TiO_2 treatment on environment. The result shows that the pretreatment of cotton fabric with nano TiO_2 was not harmful to the environment.*

Keywords—*Antibacterial, Cotton, Tensile strength, Nano TiO_2, Self-cleaning, UV transmission*

I. INTRODUCTION

The research interest for the use of nanotechnology in the textile industry has increased rapidly. This is mainly due to deploying nanotechnology. Fibers provide optimal substrates where a large surface area is present for a given weight or volume of the fabric. The synergy between nanotechnology and textile industry uses this property of large interfacial area and a drastic change in energetic is experienced by macromolecules or supra molecular clusters in the vicinity of a fiber when changing from a wet state to dry state.[1-2]

Textile materials treated with nano materials are aimed at producing finished fabrics with different functional performances. For example, nano silver has been used for imparting antibacterial properties[3-4], nano ZnO for antibacterial[5] and UV blocking properties. [6] Metal oxide nano particles are more preferable than nano silver because of cost consideration. In fact, TiO_2 and nano ZnO are non toxic and chemically stable under exposure to high temperature and are capable of photo-catalytic oxidation. [7-8] Furthermore, nano particles have a large surface area to volume ratio that results in a significant increase of the effectiveness in photocatalytic oxidation activity when compared to bulk materials.[9-11]

Titanium is a low density, highly corrosion resistant, lustrous white transition element and is available in the form of titanium oxide, titanium dioxide and titanium trioxide, titanium pentoxide. About 95% of titanium, production is consumed in the form of titanium dioxide. TiO_2 photocatalyst, find application in the purification of Water, Air, dye degradation and in materials processing functional characteristics such as anti bacterial and self-cleaning.[8] TiO_2 is an n-type semiconductor oxide photocatalyst. Among many photocatalysts TiO_2 is the only material suitable for industrial use because of it's - High photo catalytic activity, Chemical and photo stability, Non –toxicity, Resistance to photo corrosion, Low cost, etc.

Compare to TiO_2, the other semiconductor oxides generally have less photo activity and some have problems associated with stability and reactivity. TiO_2 in its nano crystalline form constitutes an enormously important commercial product, when the crystalline size is less than 100nm.The reflectance of visible light decrease and the material becomes more transparent and exhibits a pronounced absorption of ultraviolet radiation.[12]

The bulk TiO_2 is opaque but nano TiO_2 due to its transparency finds application in textiles that do not blur colors and brightness of these substrates. Since TiO_2 has high UV absorption and concurrent high transparency for visible light, it finds widespread use in diverse areas as sun cosmetics, packaging materials and wood protection coatings.[13-14] With these unique characteristics, TiO_2 could be one of the most important nanomaterials in future research and applications. In the present investigation an attempt has been made to evaluate effectiveness of nano TiO_2 treatment on the functional performance of cotton fabric.

II. MATERIALS AND EXPERIMENTAL METHODS

2.1 Materials

Nano TiO_2 particles supplied from Sigma-Aldrich Chemical Company, USA for this study. The size of supplied nano TiO_2 particles was <100 nm. Surfactant Lissapol L., Nutrient Broth, M 002, Nutrient agar M 001 (all are Microbiology grade) and Staphylococcus Aureus (ATCC-6538 grade) and Escherichia Coli (Lab collection) bacterium colonies were used. Standard potassium dichromate (0.25 N), Sulphuric Acid reagent, Standard ferrous ammonium sulfate, Mercury sulfate crystals and Ferroin indicator solution were used. The 100 % woven cotton fabric of 165 GSM of fabric was provided from Kiran Thread, Vapi, Gujarat.

2.2 Methods

Before the nano treatment, cotton fabrics were cleaned with 5 gpl soap (Lissapol L) and 2 gpl soda ash at 70^0C for 30 min., rinsed with 1 % H_2SO_4 and water twice at room temperature (35^0C) for 10 min, and equilibrated in a conditioned room (20^0C, 60% RH). The fibers were treated with nano TiO_2 colloid by a pad-dry-cure method. The TiO_2 concentrations in the padding liquor were varied 2, 4, 6, 8 and 10 gpl. The specimens were immersed in a fresh colloidal bath for 10 min and squeezed using a laboratory padder at the constant pressure. The samples were dried at room temperature for prevention of thermo-migration of metal particles for 30 min, and then the curing process of samples was performed at 140^0C, for 3 min.

2.3. Characterization of nano TiO_2 treated cotton

The nano TiO_2 treated cotton sample was observed under scanning electron microscope (SEM) (Model JSM5610LV, version 1.0, Jeol, Japan) and elementally analyzed using oxford –Inca software (U.K.).

2.4. Determination of fabric Properties

2.4.1 Tensile Strength

The breaking load (Tensile strength) of the samples was determined on tensile strength tester instrument (Model-Lloyd, LRX, U.K.). Ten tests were conducted for each sample. For particular specimen the average of ten readings has taken into account.

2.4.2 Wicking

Wicking behavior of treated and untreated samples evaluated by T-PACC standard method and specifications.

2.5 Measurements functional characteristics

2.5.1 Antimicrobial property

Antimicrobial test for nano TiO_2 treated and untreated fabrics were carried out with gram-positive Staphylococcus aureus and gram-negative Escherichia coli bacterium according to the ASTM E-2149 test method.
The percentage reduction in bacterial growth was calculated using the following equation:

$$\text{Percent Reduction of bacterial growth} = \frac{(B - A)}{B} \times 100$$

Where, “A” is the optical density for the inoculated test culture of containing the treated sample.
“B” is the optical density for the inoculated test culture of containing the untreated sample.

2.5.2 UV transmission

The samples were tested on UV transmission instrument working on optic principle, as shown in figure 1. UV radiations were generated through a tungsten halogen lamp ranging from 280-400nm wavelength as shown in figure 1. The light passes through the monochromator input slit of grating and ‘Range of grating’ was 0-1500 nm at 0.2 nm resolutions. The output from diffraction grating of monochromator is a continuous light beam. Optical chopper converts this light into pulses. The AC light passes through the concave mirror to focus the light, the use of concave mirror gives spherical wavefront, and to convert it back into the plane wavefront, plane mirror has placed. Thus, the light is directed towards the sample. From this the light passes on the fabric where the transmitted light detected by a UV diode. A lock in amplifier used to measure the photo current.

The fabric sample was placed at 0.05cm away from the UV diode. The whole set up was programmed on the computer by the Labveiw 7.1 software. The computer screen directly shows the graph between wavelength and transmittance percentage calculated by the following formula:

$$\text{U V Transmission Percent} = \frac{\text{Trans. of UV rays obtained by fabric}}{\text{Trans. of direct UV rays}} \times 100$$

2.5.3 Self-cleaning

The measured quantity of 6% coffee solution was applied on the cotton fabric and allowed to spread. One-half portion of each stain on the fabric was exposed to sunlight for 12 hours while the other half portion was covered with a black paper to prevent its irradiation from sunlight. The exposed part of the stain compared with the unexposed portion for self-cleaning action. The stained samples were assessed for color strength (K/S) values (illuminant D65/100 observer) on Spectra scan 5100 (RT) spectrophotometer (premier colorscan instrument). The self- cleaning action can quantify by comparing the K/S values of the exposed and the unexposed portion of the same stain.

The K/S value of the unexposed part of the stain taken as 100 and the relative decrease in the K/S value of the exposed part can calculate using the following relation.

$$\%\ \text{Decrease in } K/S \text{ value} = \frac{(K/S)\text{unexposed} - (K/S)\text{exposed}}{(K/S)\text{unexposed}} \times 100$$

2.6 COD-Chemical oxygen demand

COD represents the oxygen required to chemically decompose the pollutant. The COD test is used exclusively in the analysis of industrial wastes. In addition, it is helpful in indicating toxic conditions and the presence of biologically resistant organic substances. In experiment, the organic matter is completely decomposed to CO_2, H_2O and oxides. Organic matters are destroyed by a boiling mixture of chromic and sulfuric acids. A sample is refluxed with known amounts of potassium dichromate and sulfuric acid. The excess dichromate is measured as oxygen equivalent. In this way toxicity of nano TiO_2 effluent tested.

III. RESULT AND DISCUSSION

3.1 Characterization of nano TiO_2 coated fabrics

The treated nano substrates were observed by scanning electron microscopy. The SEM images of cotton fiber treated with 10 gpl nano TiO_2 particles and untreated cotton fabric is presented in Fig. 2 a and b respectively. An uniform dispersion of nano particles is observed on the fabric.

3.2 Effect of nano TiO_2 on fabric properties

3.2.1 Tensile strength

It has been observed from the Table 1 that there is no significant change in the tensile behavior of cotton fabric treated with nano TiO_2 at different concentration level. Even at higher level of pretreatment concentration there is a minor increase in tensile property of cotton fabric.

3.2.2 Wicking behavior

The results given in Table 2, indicates that there is no major effect of nano TiO_2 pretreatment on wicking behavior of cotton fiber, however there is minor decrease in wicking behavior at higher concentration level. It may be because of the nano particles remain on the surface of fiber in agglomerated form at higher concentration. This may hinder the flow of water through the fabric matrix.

3.3 Effect of nano TiO_2 on antimicrobial property of cotton

Antimicrobial property tested against S.Aureus and E.Coli bacterium. From Table 3, Cotton fabric treated with all concentrations of nano TiO_2 is tested for antimicrobial property. There is consistent improvement in antimicrobial property of cotton fabric found with S.aureus bacterium. Antimicrobial property is in increasing order with the increase in concentration of nano TiO_2 pretreatment but there is no significant improvement found in antimicrobial property with E.Coli bacterium[12].

3.4 Effect of nano TiO_2 on UV transmission percent of cotton

Table 4 shows, UV transmission percent through fabric decreases as the concentration of nano TiO_2 increases i.e. better UV protection is obtained as compared to untreated fabric. At 1gpl, 3gpl there is no significant improvement in UV protection of fabric. From the results it can be safely interpreted that higher the concentration of nano better is the UV protection property of the fabric. It is also evident from the table that the application of nano sized particles increases UV absorbance in the region between 300nm and 400nm.There is around 50% UV shielding as compare to untreated cotton fabric samples.

3.5 Effect of nano TiO_2 on Self-cleaning property of cotton

Table 5 revels that the K/S values of cotton fiber treated with nano TiO_2 exposed to sunlight is decreased compared to the untreated sample. The results also reveal that as the concentration of nano increases the K/S values of the samples decreases, indicates improvement in self-cleaning action. The K/S values of nano TiO_2 pretreated cotton sample with 6% coffee stain has been decreased to 40 % at 10 gpl concentration level.

3.6 Environmental impact of Nano TiO_2

COD value of 5 gpl concentration effluent solution found to be 44 which is less than standard value for COD. This result shows that nano TiO_2 applied on the fabric is non-toxic and not harmful to the environment, it does not require any effluent treatment.

IV. CONCLUSIONS

Nano size TiO_2 particles can successfully applied on cotton fabric by Pad-Dry-Cure technique. The dispersion of nano on fabric surface is observed by SEM. The pretreatment of cotton fabric with nano TiO_2 improves antimicrobial property with gram-positive bacterium i.e. S.aureus only there is no reaction of antimicrobial property with gram negative bacterium E.coli. It has been also found that the nano treatment at higher concentration improves UV transmission percentage of cotton fabric without affecting the tensile strength and wicking behavior of fabric. The self cleaning action of nano TiO_2 pretreatment is higher at the higher concentration level.

Finally, it can be concluded that the pretreatment of cotton with nano TiO_2 particles is found not harmful to the environment and can successfully be utilize for improving functional performance of the fiber.

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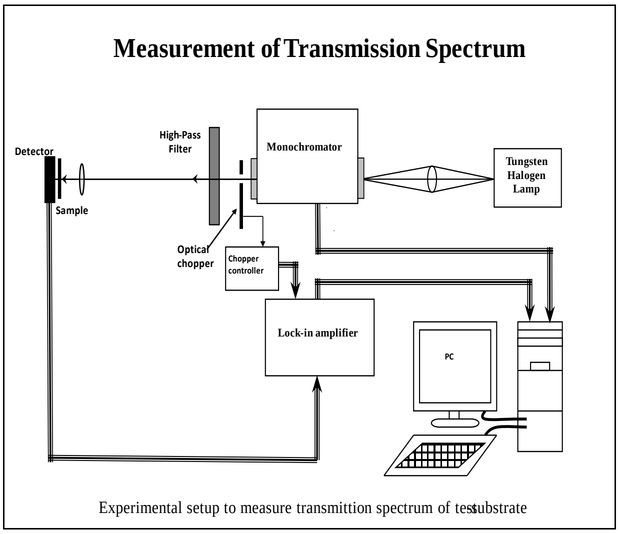

Figure 1 Laboratory set-up to determine the UV transmission percentage.

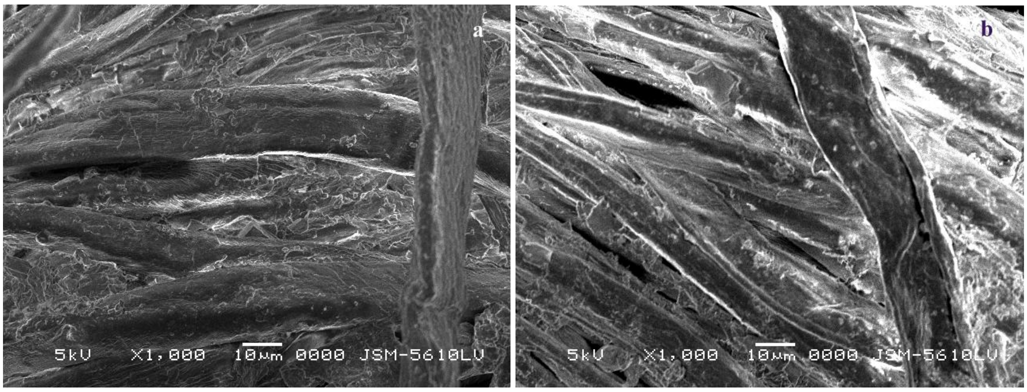

Figure 2 SEM images of cotton fabric (a) untreated and (b) treated with 10gpl nano TiO_2

Table 1: Effect of nano TiO_2 treatment on Tensile Strength property

Sample	**Tensile strength(Kgf)**
Untreated	20.8
Nano TiO_2 Treated (GPL)	
2	21.5
4	20.7
6	22.6
8	22.8
10	21.9

Table 2: Effect of nano TiO_2 treatment on Wicking property

Sample	**Wicking Height (mm)**		
	1min	**5min**	**10min**
Untreated	60.7	101.2	152
Nano TiO_2 Treated (GPL)			
2	61.6	103.5	148
4	60.4	102.6	151
6	61.6	98.6	149
8	60.9	97.8	145
10	61.5	98.3	151

Table 3: Effect of nano TiO_2 treatment on Antimicrobial property

Sample	**Optical density of samples treated with S.aureus bacteria**	**Reduction of bacterial growth (%)**
Untreated sample	1.45	No reaction
Nano TiO_2 Treated (GPL)		
2	0.073	94.96552
4	0.066	95.44828
6	0.042	97.10345
8	0.032	97.7931
10	0.012	99.17241

Table 4: Effect of nano TiO_2 treatment on UV transmission property

Sample	**UV-A**	**Improvement %**	**UV-B**	**Improvement %**
Untreated sample	21.50		21.60	
Nano TiO_2 Treated (GPL)				
2	16.4	17.26852	17.05	16.82927
4	15.8	18.65741	16.43	19.85366
6	15.2	25	15.4	24.87805
8	13.6	41.2037	12.68	38.14634
10	11.04	48.7037	11.16	45.56098

Table 5: Effect of nano TiO_2 treatment on Self cleaning property

Sample	K/S Value Unexposed	K/S Value Exposed	Decrease in K/S (%)
Untreated	0.66	0.56	15.15
Nano TiO_2 Treated (GPL)			
2	1.05	0.8	23.80
4	1.08	0.75	30.55
6	1.12	0.71	36.60
8	1.14	0.69	39.47
10	1.14	0.68	40.35