

Functionalization of Cotton Fabric with Metallic Nanoparticles and Madder Root Dye

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Abstract: Cotton is the most abundantly used cellulosic fibers in the clothing industry due to its biodegradability, comfortability, hydrophilicity, smoothness and notable colouration. To fulfil the ever-growing consumer demands of consumers for hygienic, protective and active wear clothing as well as increasing the potential applications and value addition to cotton fabric, numerous studies have been carried out to expand the existing properties and impart functional properties like antibacterial, UV protection, self-cleaning, anti-pilling etc. On the other hand, nanoparticles of metal oxides such as silver, aluminum oxide, copper oxide, iron oxide, silicon dioxide, titanium dioxide, zinc oxide etc. are some of the known agents used for imparting various functional properties such as anti-microbial, water repellency, soil resistance, anti static, anti infrared and flame retardancy, dyeability and strength to the textile materials. The purpose of the current research was to dye cotton fabric with madder root dye in the presence of metallic nanoparticles and evaluate its dyeing performance and functional properties. The scoured cotton fabric was treated separately with the three selected nanoparticles i.e. copper oxide (CuO), titanium dioxide (TiO₃) and zinc oxide (ZnO) and dyed simultaneously with madder root dye by exhaust method in IR beaker dyeing machine using the optimized process variables. The Field Emission-Scanning Electron Microscopy (FE-SEM) analysis was done to analyze the surface morphology of untreated dyed and nanoparticle treated dyed cotton fabrics. The Fourier Transform Infrared Spectroscopy (FTIR) analysis of madder root dye powder as well as nanotreated natural dyed cotton fabric samples was conducted to ascertain the presence of various functional groups. The colour properties of nanotreated dyed cotton fabric samples in terms of colour coordinates, colour strength and colour fastness to wash, light, perspiration and rubbing were assessed to establish the effect of nano treatment on improvement of colour properties. The functional properties of nanotreated dyed cotton fabrics were also tested to evaluate the effect of nanoparticle treatment on these fabric properties. The FE-SEM analysis of nanotreated dyed fabrics confirmed the presence of nano-sized particles embedded into the fibre matrix and were well dispersed on the fibre surface of cotton fabric. FTIR analysis of madder root dye powder exhibited the presence of C-N primary amines, C-O stretch alkyl aryl ether, N-O and stretch nitro C=C/C=O stretch alkene/amide and N-H stretching functional groups indicated by their associated peaks. The FTIR analysis of nanotreated dyed fabric samples showed the presence of different groups that imparted cationic charge to cotton fabric and enhanced dye absorption. The nanoparticle treatments along with natural dyeing of cotton fabric with madder root dye demonstrated enhanced dye uptake, colour properties and functional properties. Hence, the use of metallic nanoparticles in small quantity can be successfully employed for improving the dyeing and functional properties of cotton fabric with natural dyes.

Keywords: Colour properties, cotton, functional properties, metallic nanoparticles, natural dyes

Among all the natural origin fibres, cotton is the most plentifully used cellulosic fibers in the clothing industry and holds the significant market share (over 50 %) among fabric products used for apparel, home textiles and other textile goods. There is substantial demand for cotton

textiles that come into close contact with human skin, including clothing, home textiles, non-implantable medical textile materials and hygiene products. The reason can be found in the characteristic nature of cotton *i.e.* its biodegradability, wearing comfortability,

hydrophilicity, smoothness and air permeability. However, cotton has less affinity toward anionic substances such as dyes, hence cationization of cotton is carried out for its modification to improve its affinity for wet processes. Cationic agents possess two functional features i.e. multiple functional groups that could react with cotton under alkaline conditions and cationic groups that could reduce the negatively charged barrier between fibre and dye. Salts of metal such as alum, ferrous sulphate and inorganic material such as sodium sulphate, sodium carbonate and sodium chloride have commonly been used as mordants in dyeing of cotton with natural dyes to improve the fastness properties and dye uptake of the textile substrates (Zhang et al., 2016; Verma et al., 2021; Tania et al., 2021).

Natural dyes are having wide applications in the colouration of most of the natural fibres such as cotton, linen, wool and silk and to some extent to few synthetic fibres i.e. nylon and polyester. However, the major issues for natural dyes are reproducibility of shades, non availability of well defined standard procedures for application and poor lasting performance of dye under wash and light exposure. As natural dyes have poor colour fastness and achieving good fastness especially towards washing and light is a challenge for the researchers as well as dyers. Therefore, metallic mordants in the form of metal salts are frequently used for the improvement of dye uptake and retention of natural dyes in the dyed substrate. (Singh and Bharti, 2014).

New technologies and methods are being followed to achieve the goal of improving colour properties. Nano technology in the form of nanomaterial is one such approach to achieve these properties. Nano technology is increasingly attracting worldwide attention because it is widely perceived as offering huge potential in a wide range of end uses. Nano particles of metal oxides such as silver, aluminum oxide, copper oxide, iron oxide, silicon dioxide, titanium

dioxide, zinc oxide etc. are some of the known agents used for imparting various functional properties such as anti microbial, water repellency, soil resistance, anti static, anti infrared and flame retardancy, dyeability and strength to the textile materials. Nano particles have a large surface area to volume ratio and high surface energy, thus presenting better affinity for fabrics and leading to an increase in durability of the given treatment for intended function. (Gopalakrishnan and Jeyanthi, 2013; Gupta *et al.*, 2021).

Therefore, the present study was carried out to ascertain the effect of metallic nano particles on dyeing performance and functional properties of cotton fabric.

MATERIALS AND METHODS

A. Materials:

- 1. **Fabric:** The grey cotton fabric (ends/inch: 54 and picks/inch: 42) employed in this experiment was procured from local market of Hisar, Haryana.
- **2. Nano particles:** Three different metal based nanoparticles (50-100 nm) namely copper oxide (CuO), titanium dioxide (TiO₂) and zinc oxide (ZnO) were used as mordant and were procured from Sigma-Aldrich, India.
- **3. Natural dye:** Madder (*Rubia tinctorum*) root dye material was procured from local market of Hisar. The dye material was washed to remove the impurities, shade dried and then crushed and ground into fine powder.

B. Methods:

- 1. **Preparation of fabric:** To make the cotton fabric more absorbent and free from the extraneous substances, enzymatic desizing followed by enzymatic scouring of selected cotton fabric was carried out using Amylase 543 and Pectinase enzymes respectively by adopting the methods given by Vigneshwaran *et al.*, 2013.
- **2. Dye extraction:** The 10 g dye power was

soaked in 100 ml distilled water overnight, boiled for 60 minutes and filtered using nylon mesh to obtain first filtrate. The next percolation was done by boiling the used dye material of first extraction in 100 ml water for 30 minutes and again filtered to obtain the second filtrate. The last extraction was done by boiling the residue of second extraction in 50 ml distilled water for 20 minutes. All the three dye filtrates were mixed thoroughly and used for dyeing of fabric samples.

- Nano particle treatment and dyeing of 3. cotton fabric: The nano particle treatment and simultaneous dyeing of cotton fabric samples with madder root dye was carried out using Infrared Beaker Dyeing Machine in the treatment solution containing 5 per cent dye extract, 0.75 per cent concentration of copper oxide, 1 per cent concentrations of each titanium oxide and zinc oxide, keeping 1:30 material to liquor ratio at pH 6, at 90°C temperature for 30 minutes. After dyeing, fabric samples were allowed to cool in the solution and then rinsed with hot water followed by soaping with 2 g/l non ionic detergent using ML ratio 1:30 at 60°C temperature for 15-20 minutes. Afterward, the samples were washed thoroughly in cold water, squeezed and dried at ambient temperature.
- 4. **Colour measurements:** The colour strength of nano particle treated dyed fabric samples was analyzed using Computer Colour Matching system (Premier Colour Scan Spectrophotometer SS5100A) and colour coordinates were represented by the CIE L*a*b* colour space system. The K/S values were obtained at 10-degree standard observer in D 65 light source using the Kubelka-Munk equation.

Colour Strength (K/S)= $\frac{(1-R)^2}{2R}$

R = Reflectance

K = The adsorption coefficient

S = The scattering coefficient

5. Field emission scanning electron

microscopic analysis: Field Emission-Scanning Electron Microscopy (FE-SEM) was done to elucidate the surface morphology of untreated dyed and nano particle treated dyed cotton samples. FE-SEM was performed on Nova Nano 450 at an acceleration voltage of 15 kV with the magnification ranging from 5000-10000X. Prior to analysis, the fabric samples were sputter coated with gold using a sputter coater (Quorum Q15OTES).

- **6. Fourier transform infrared spectroscopy analysis:** The characterization in terms of interactions and chemical absorption of treated dyed samples were examined using FTIR instrument (Nicolet iS50, Thermo Scientific). FTIR spectrum was recorded for transmittance in the range of 500-10000 cm⁻¹ and 24 scans were performed for each sample.
- **7. Colour fastness:** All the nanoparticle treated dyed samples were examined for colour fastness to washing, sunlight, perspiration and rubbing using the methods prescribed by the Bureau of Indian Standards (BIS). Fastness to washing IS: 3361-1979 method (BIS, 1979), fastness to sunlight IS: 686-1985 method (BIS, 1985), fastness to perspiration IS: 971-1983 method (BIS, 1983) and fastness to rubbing IS: 766-1988 method (BIS, 1988).
- **8. Functional properties:** The untreated dyed and nanoparticle treated dyed fabric samples were tested for the antibacterial property in terms of colony forming units and percent reduction in bacterial count as per AATCC Test Method 100. The Ultra violet protection property was measured using SDL UV penetration and protection measurement system (Compsec M 350 UV Visible Spectrophotometer) according to test method UVR Transmission AATCC-183: 2004.

RESULTS AND DISCUSSION

Field emission scanning electron microscopic analysis of nanoparticles treated dyed cotton fabric: The Scanning Electron Microscopic (SEM) analysis of cotton samples

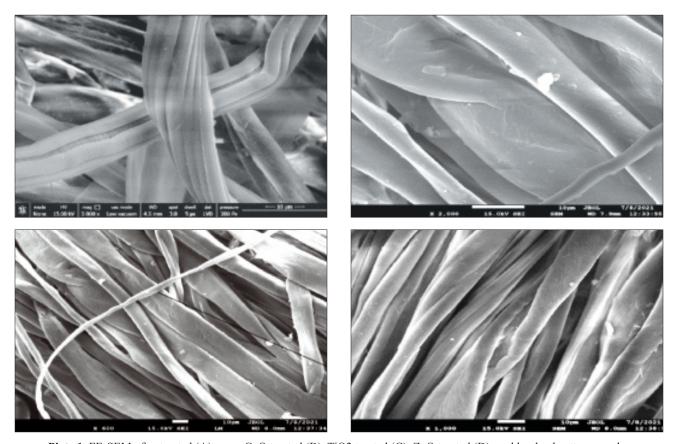


Plate 1. FE-SEM of untreated (A), nano CuO treated (B), TiO2 treated (C), ZnO treated (D) madder dyed cotton samples

treated separately with nano copper oxide, titanium dioxide and zinc oxide and dyed simultaneously with madder root dye was conducted to analyze the morphological changes that occurred due to the nano treatments. It was observed that nano particle treated dyed cotton samples showed the presence of nano particles onto the fibre matrix as the particles were observed to be well dispersed on the fibre surface. The morphological structures of treated dyed cotton fabric are presented in Plate 1. The results are also in line with Chattopadhyay and Patel, (2010); Khandual et al., (2015) that SEM micrograph of nano copper oxide and titanium dioxide treated cotton fabric exhibited the dispersion and distribution of nano particles inside the fibre matrix.

Fourier Transform Infrared Spectroscopy (FTIR) analysis of dye powder and nano particles treated dyed fabrics: The Fourier Transform Infrared Spectroscopy (FTIR) analysis of madder

root dye powder, untreated and nano particle treated dyed cotton fabrics was done and presence of various functional groups and their peaks were observed.

The data incorporated in Table 1. depict the presence of various functional groups at different peaks which were responsible for change in different properties. The spectrum of madder root dye exhibited peaks at 1025/cm owing to the presence of C-N primary amines, peaks corresponding to wavelength 1234/cm exhibited the presence of C-O stretch alkyl aryl ether and peak at 1515/cm was attributed to N-O stretch nitro compound. The presence of peaks at 1744 and 2939/cmdepicted the presence of C=C/C=O stretch alkene/amide and N-H stretching respectively. Peaks at 3274 and 3743 are representative of OH/N-H stretch aclohol/amine (Fig. 1). The colour produced by natural dye on fabrics are governed by the nature of chromophores as well as substituent functional

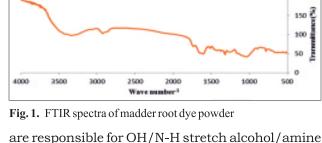
250

200

groups and the auxochromes of molecular species. Yusuf *et al.*, (2015) also reported that madder root is a source of an anthraquinone pigment which is used as colouring matter for imparting colour to textiles.

The data in Table 2. demonstrates the FTIR spectrum for untreated, nano treated and madder root dyed cotton fabric samples. The data in Table reveal that the peaks at 1030, 1104 and 1155/cm were present in untreated and madder root dyed cotton fabric and all the three nano treated dyed cotton fabric samples which indicated the presence C-N stretch primary amines, while peak at 1155/cm in untreated dyed cotton fabric slightly intensified to 1159/cm in nano copper oxide and zinc oxide treated and madder root dyed sample, 1158/cm in titanium dioxide treated dyed sample.

The appearance of a strong broad band at 1314/cm in nano copper oxide treated dyed, 1320/cm in nano titanium dioxide treated dyed and 1316/cm in nano zinc oxide treated dyed samples confirmed the characteristics of C-O stretch alkyl aryl ether groups which was not present in untreated dyed cotton fabric. Peaks at 3334, 3342, 3348 and 3354/cm wave numbers



in untreated dyed, nano copper oxide, titanium dioxide and zinc oxide treated dyed samples respectively. Characteristics of -OH stretching were found in untreated dyed cotton fabric as peak appeared at 3750/cm which further intensified after treatment with nano copper oxide (3853 cm), titanium dioxide (3820/cm) and zinc oxide (3856/cm) and dyeing simultaneously with madder root dye (Fig. 2). Nithya et al., (2015) stated that increase in the carbonyl group on the fabric surface enhanced the hydrophilic property of the fabric in addition to the physical changes. Gupta et al., (2021) noted the peak at 3226/cm region and peak got broadened at 3743/cm after dyeing with madder dye indicating the addition of -OH group after dyeing.

Table 1: FTIR analysis of madder root dye powders

Wave numbers (cm ⁻¹)	Functional groups	Peak positions
1000–1200	C-N stretch primary amine	1025
1200-1400	C-O stretch alkyl aryl ether	1234
1400-1600	N-O stretch nitro compound	1515
1600–1800	C=C/C=O stretch alkene/amide	1744
2800–3000	N-H stretching	2939
3200–3800	OH/N-H stretch aclohol/amine	32743743

Table 2. Ginning performance of pink bollworm infested cotton

Parameter	Un infested cotton	Pink bollworm infested cotton	Change (%)	
Ginning Percentage (%)	35	29	(-) 17.1	
Fibre length (mm)	31.0	26.7	(-) 13.9	
Uniformity Index (%)	83	79	(-) 4.8	
Fineness (mic)	4.0	3.0	(-) 25.0	
Tenacity (g/tex)	29.5	26.1	(-) 11.5	
Elongation (%)	5.6	5.3	(-) 5.4	
SFC (%)	5.2	8.7	(-) 67.3	
Rd (%)	76.8	64.3	(-) 16.3	
+b (%)	8.5	12.1	(-) 42.4	
Colour grade	31-1	41-1	-	

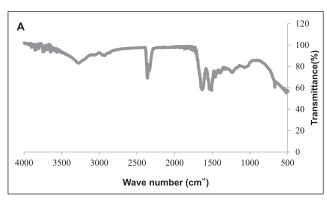
Effect of nano particle treatments and dyeing on colour properties of cotton fabric: The colour properties of untreated cotton fabric, copper oxide, titanium dioxide and zinc oxide nano particles treated and dyed simultaneously with madder root dyes was assessed to ascertain the effect of nano particle treatments on L*, a*, b*, C*, H* values of dyed cotton fabric.

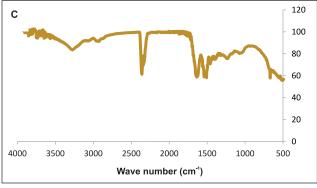
The Table 3. exhibit colour coordinates and colour strength of untreated, nano copper oxide, titanium dioxide and zinc oxide treated samples and dyed simultaneously with madder root dye. The L* values of untreated dyed cotton fabric was 63.16 which indicated lighter shade as compared to all the nano particle treated dyed samples exhibiting the L* values 58.66, 56.88 and 58.02 in respect of copper oxide, titanium dioxide and zinc oxide nano particles respectively.

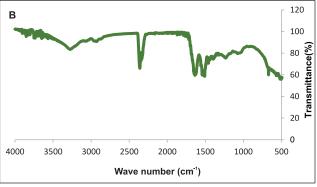
All the three nano particle treated dyed cotton fabric samples exhibited positive a* and b* values with hue angle less than 90° indicating maroon to brownish maroon tone which were noted to be enhanced in comparison with untreated dyed cotton fabrics. Similarly, the

chroma (C*) values of nano particle treated dyed cotton fabric samples were also found to be increased to 28.36, 22.82 and 28.00 when treated with copper oxide, titanium dioxide and zinc oxide nano particles respectively in comparison to chroma values of untreated dyed (18.90) cotton fabric.

The colour strength (K/S) of untreated madder root dyed cotton fabric was 14.17, the addition of nano particles during dyeing of cotton fabric with madder root dye resulted in increased colour strength and highest increase was noted for zinc oxide treated dyed sample (23.62) followed by nano copper oxide (22.48) and nano titanium dioxide (19.80). The outcomes were also confirmed by the results of Chatopadhyay and Patel (2011) that the treatment of cotton fabric with zinc oxide nano particles and dyeing with direct dyes improved the colour strength of the fabric. The findings of Gupta et al., (2021) also support the results of the present study that meta mordanting with nano TiO2 and SiO2 enhanced the colour properties of madder dyed cotton fabric.







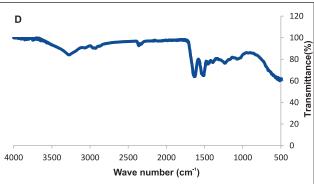


Table 3: Colour coordinates and colour strength of madder root dyed cotton fabric

Treated dyed samples		Сс		Colour strength (K/S)		
	L*	a*	b*	C*	H*	
Untreated	63.16	14.58	12.30	18.90	38.14	14.17
Copper oxide	58.66	24.31	24.33	28.36	30.96	22.48
Titanium dioxide	56.88	20.94	24.03	22.82	23.38	19.80
Zinc oxide	58.02	25.14	26.14	28.00	36.11	23.62

Table 4: Colour fastness grades of nanoparticle treated and madder root dyed cotton fabric

Treated dyed		Colour fastness grades										
Samples		Perspiration					Rubbing					
	Wa	ash	Light	Alkaline Acidic			Dry		We	et		
	CC	CS		CC	CS	CC	CS	·	CC	CS	CC	CS
Untreated	3/4	3/4	3/4	3/4	3/4	3/4	3/4	;	3/4	3/4	3/4	3/4
Copper oxide	4/5	4/5	4/5	4/5	4/5	4	4		4/5	4/5	4/5	4/5
Titanium dioxide	4/5	4/5	4/5	4/5	4/5	4	4		4/5	4/5	4/5	4/5
Zinc oxide	4/5	4/5	4/5	4/5	4/5	4	4		4/5	4/5	4/5	4/5

CC: Colour change; CS: Colour staining

Table 5: Effect of nanoparticle treatments on antimicrobial property of madder root dyed cotton fabric

Treated	Staphylo	coccus aureus	Escherichia coli			
dyed samples	Bacterial count (CFU/ml)	Percent reduction in bacterial growth	Image of bacterial growth on treated dyed fabrics	Bacterial count (CFU/ml)	Percent reduction in bacterial growth	Image of bacterial growth on treated dyed fabrics
Untreated	7.34×108	-		6.60×108	-	
Copper oxide	4.45×108	39.37	\bigcirc	3.85×108	41.66	
Titanium dioxide	4.26×108	41.96		3.44 ×108	47.87	
Zinc oxide	4.10×108	44.14		2.67 ×108	59.54	

Effect of nanoparticle treatments and dyeing on colour fastness properties cotton fabric:

The colour fastness properties of untreated and nano particle treated and madder root dyed samples was evaluated to establish the effect of nano particle treatment on wash, light, perspiration and rubbing fastness of treated dyed cotton fabrics. The results pertaining to colour fastness of the different treated dyed cotton fabric samples are presented in Table 4.

It was found that the untreated madder root dyed cotton fabric had fairly good (3/4)

fastness to washing, light, perspiration and rubbing. The treatment of cotton fabric with nano copper oxide, titanium dioxide and zinc oxide and simultaneous dyeing with madder root dye, resulted in very good wash fastness (4/5) in terms of colour change and colour staining, sunlight, dry and wet rubbing and alkaline perspiration whereas good fastness (4) was noticed in case of acidic perspiration. Chattopadhyay and Patel (2011) mentioned that the metal nano particle treatment resulted in better bonding between the dye and fibre

Table 6: Effect of nanoparticle treatments on ultra-violet protection property of madder root dyed cotton fabric

Treated dyed Samples	UVA Transmission (%)	UVB Transmission (%)	UPF	Protection Category
Untreated	6.74	8.40	11.40	No category
Copper oxide	2.55	1.50	51.03	Excellent
Titanium dioxide	2.64	1.67	51.99	Excellent
Zinc oxide	2.83	1.77	51.36	Excellent

UPF Rating: Good protection: 15, 20; Very good protection: 25, 30, 35; Excellent protection: 40, 45, 50, 50+

reflecting by improvement in colour fastness properties of dyed fabrics. Alebeid *et al.*, (2015) reported that durability of colour of dyed fabrics to washing could be attributed to the formation of covalent linkages between hydroxyl groups of TiO_2 and cotton fabric.

Effect of nanoparticle treatments and dyeing on functional properties of cotton fabric: Functional properties *viz.* antimicrobial and ultraviolet protection factor of untreated and nanoparticle treated cotton fabric were assessed to know the effect of nano treatments with copper oxide, titanium dioxide and zinc oxide and dyeing with madder root dyes on these functional properties.

Effect of nano particle treatments and dyeing on anti microbial properties of cotton fabrics: The data incorporated in Tables 3 presents the anti bacterial activity against *Staphylococcus aureus* (gram positive) and *Escherichia coli* (gram negative) bacteria of untreated and nano particle treated cotton fabric dyed with madder root dye.

The data in Table 5. narrates that the untreated madder root dyed cotton fabric had 7.34×10⁸ CFU/ml for *Staphylococcus aureus* bacteria and 6.60×10⁸ CFU/ml for *Escherichia coli* bacteria. When copper oxide nano particle treatment and simultaneous dyeing of cotton fabric with madder root dye was done, treated dyed sample displayed bacterial count of 4.45×10⁸CFU/ml for *Staphylococcus aureus* with 39.37 per cent reduction in its growth whereas for *Escherichia coli* the bacterial count was 3.85×10⁸ CFU/ml showing 41.66 per cent reduction in its growth.

The nano titanium dioxide treated dyed

fabric exhibited bacterial count of 4.26×108 CFU/ml for *Staphylococcus aureus* and 3.44×10⁸ CFU/ml for Escherichia coli with 41.96 and 47.87 per cent reduction in bacterial growth of Staphylococcus aureus and Escherichia coli respectively. With the nano zinc oxide treatment and dyeing of cotton fabric with madder root dye, the bacterial count of 4.10×10⁸ and 2.67×10⁸ CFU/ml with 44.14 and 59.54 per cent, respective reduction in growth of Staphylococcus aureus and Escherichia coli was witnessed. Montazer and Seifollahzadeh (2011) reported the antibacterial activity of nano TiO2 on cotton fabric against E. coli bacteria and found that the reduction of bacterial count reached to 100 per cent with 0.75 per cent concentration of nano TiO₂ and 99 per cent with 0.25 per cent concentration of nano TiO2. Harifi and Montazer (2014) mentioned that iron nano particles possess unique characteristics that make them promising agents for antibacterial applications as these are capable of binding bacterial cell walls causing membrane disruption.

Effect of nano particle treatments and dyeing on ultra violet protection property of cotton fabric: The data presented in Table 6 describe the ultra violet protection property of untreated and nano particle treated madder root dyed cotton fabric samples.

It is noticed from the data in Table 6. that UPF value of untreated madder root dyed cotton fabric was 11.40 which came under no protection category. After treatment of cotton fabric with nano copper oxide, titanium dioxide and zinc oxide and dyeing with madder root dye, the UPF values of treated dyed fabric samples were observed to be increased to 51.03, 51.99 and 51.36, respectively which represented the

excellent protection category. It was also noticed increased UPF value of cotton fabric treated with ZnO nanoparticles.

CONCLUSIONS

Thus the above work concludes the following:

- It is thus concluded that the FTIR spectra
 of madder root dye powder confirmed the
 presence of different functional groups
 responsible for imparting colour to textile
 substrates.
- Nano particle treated dyed samples displayed the presence of nano particles onto the fibre matrix as the particles were well dispersed on the fibre surface.
- Brighter shades were obtained on cotton fabric when treated with copper oxide, titanium dioxide and zinc oxide nano particles and dyed with madder root dye as compared to the untreated dyed cotton fabric samples.
- All the nano particles enhanced the colour strength of the treated dyed cotton fabric, but the zinc oxide nano treated cotton fabric obtained darker as indicated by higher colour strength value.
- Nano particle treatment enhanced the wash, light, perspiration and rubbing fastness of treated dyed samples in comparison to untreated dyed cotton samples.
- Nano particle treated and madder root dyed cotton fabrics had lower bacterial count as compared to untreated dyed cotton fabrics and highest bacterial resistance was obtained with nano zinc oxide treatment.
- Nano particle treatments and dyeing with madder root dye improved the UPF value of cotton fabric as compared to untreated dyed cotton fabrics.

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