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Copper nano-sol loaded woven fabrics: structure and color characterization

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Abstract

Nano technology is an upcoming branch where lot of research work is being done with the intention of improving/changing properties of the materials. In this study polyester, cotton and polyester-cotton blended fabrics were coated with copper nano particles by pad-dry-cure technique. The morphology of nano-composite fabrics characterized using scanning electron microscope reveals that nanoparticles were dispersed on the fabric samples. The elemental analysis for confirming the presence of copper in the fabric samples was done using the Fourier transform infrared spectroscopy and energy dispersive X-ray spectroscopy techniques. The X-ray diffraction analysis of the fabric samples helped to unveil that incorporation of copper nano particles increased the d-spacing value in polyester fabric to a greater extent in comparison to the cotton and polyester-cotton blended fabrics. The nano treated woven fabric samples were dyed using myrobolan; a natural dye. The samples were evaluated in terms of colourimetric parameters and colour fastness. The copper nano-sol treatment was found to improve both K/S and colour fastness.

Keywords: Copper nano, Cotton, Morphology, Dyeing, Natural dye, Polyester, Polymer structure

Introduction

Nano particles have at least one dimension as 100 nm or less. Owing to higher surface areas nano materials can be highly reactive; hence find their application in variety of fields like medicine, electronics, food, fuel cells, batteries, water treatment, chemical sensors, pharmaceuticals, dairy industry, textiles etc. Its application is sought in textiles, to achieve improvement or change in property of the raw material or improvement in its functional properties (Chattopadhyay and Patel 2009, 2010; Patel and Chattopadhyay 2007; Patel and Patel 2012). Currently nano technology is used in textiles with the aim of furthering functional properties like dyeing, resistance to shrinkage, wrinkling, staining and flammability.

Nano application is also opted for improving the ultra violet (UV) protection, water repellency, and in odor and moisture management. They have capability to provide self cleaning and anti-bacterial properties making them popular for intimate as well as non intimate applications. In short the type of effect achieved like self cleaning/antibacterial/UV resistant and so on, is greatly dependent on the choice of nano material selected and

its inherent characteristics (Patel et al. 2014; Bhattacharya and Chaudhari 2014; Shaikh et al. 2015; Chattopadhyay and Patel 2012).

Besides the nano material (filler) chosen, its degree of dispersion greatly influences the enhancement efficiency. To attain excellent properties, it is however very important that the average size of fillers present must be in the nano-meter size along with fine particle distribution in the polymer matrix after its application. Copper nanoparticles have shown a great potential as the nano-filler for hygiene application and in making textiles electrically conductive (Chattopadhyay and Patel 2011, 2012, 2013, 2014a, b).

The present study reports observations when 100% polyester ('p'), 100% cotton ('c') and polyester–cotton blended (p–c; 50:50) fabric samples were treated with copper nano sol. The properties of nano-composite fabrics were evaluated in terms of morphological and chemical changes. The surface morphology has been observed using scanning electron microscopy (SEM). The chemical transformation has been analyzed using Fourier transform infrared spectroscopy (FT-IR), energy dispersive X-ray spectroscopy (EDS) and X-ray diffraction (XRD) technique (<http://www.intechopen.com/books/fourier-transforms-new-analyticalapproaches-and-ftir-strategies/applications-offourier-transform-infrared-spectroscopy-to-study-cotton-fibers>).

Methods

Fabric

Three fabric samples; 100% 'p', 100% 'c' and p–c blend (50:50; intimate), all with plain weave (p.w) were collected from market. These fabrics were then subjected to light scouring before application of any treatment to them [5 g per liter (gpl) of nonionic detergent and 2 gpl of sodium carbonate were mixed in water and boiled for 15 min]. Fabric specifications are mentioned in Table 1.

Dyes and chemicals

Myrobolan (C.I. Natural brown 6) was used as received for this study without further purification. Copper sulphate, sodium borohydride and tri-sodium citrate of analytical grade purity were procured from Sd Fine Chemicals, India.

Preparation of copper nano sol

Copper nano colloidal sol was prepared from copper salt by chemical reduction technique (Chattopadhyay and Patel 2010). "0.3 g CuSO₄ was dissolved in 100 ml of distilled water. It was then kept in the reaction chamber and was slowly reduced by drop wise

Table 1 Polyester, cotton and polyester–cotton fabric specifications

Fabric sample	Fabric specifications					
	Count (English)		Ends/inch	Picks/inch	Weave	Fabric weight g/sq.mt
	Warp	Weft				
Polyester	61.38	37.07	132	96	p.w	120
Cotton	32.63	33.9	88	60	p.w	101
P–C blend	35.48	35.63	100	74	p.w	120

p.w plain woven fabric

addition of very dilute chilled solution of sodium borohydride (NaBH_4 , 0.1 g dissolved in 50 ml distilled water) in nitrogen atmosphere. During the entire reaction process, the solution mixture was stirred vigorously. As the color of the solution turned to light yellow; 5 ml of trisodium citrate ($\text{C}_6\text{H}_5\text{Na}_3\text{O}_7 \cdot 2\text{H}_2\text{O}$, 0.1 g dissolved in 100 ml distilled water), was added drop by drop with vigorous stirring” (Patel and Chattopadhyay 2007). 5 g Lissapol L as surfactant was added to 100 ml of copper nano sol coating solution. The mixture was then stirred using magnetic stirrer at 250 rpm for 30 min at 60 °C temperature.

Preparation of nano copper composite fabrics

The padding liquor was applied to the ‘p’, ‘c’ and p–c fabric samples (size: 40 × 30 cm) by dipping them in the dispersion for 10 min. After which they were padded on an automatic padding mangle machine, operating at 15 rpm and 1.75 kg/cm² pressure; using 2-dip-2-nip padding sequence at 70% expression. The padded substrates were dried at 80 °C for 5 min and cured in a preheated curing oven. Curing of ‘p’, ‘c’ and p–c fabric samples was done in oven at temperatures of 140, 120 and 130 °C respectively for 3 min.

Characterization of nanoparticles

The morphology and elemental analysis of the synthesized nanoparticles was performed using SEM (Model JSM5610LV, version 1.0, Jeol, Japan) using Oxford-Inca software (U.K.). The copper nanoparticles were examined after depositing on carbon coated aluminum sheet and further coating with gold (Au).

Characterization of fabric samples

The surface morphology of the copper loaded nano ‘p’, ‘c’ and p–c blended fabric was observed on SEM (Model JSM5610LV, version 1.0, Jeol, Japan). The presence of copper in composite fabric was confirmed by elemental analysis using Oxford-Inca software (U.K.) on the same instrument. The chemical composition of the composite material was further analyzed using FT-IR; recorded using KBr wafer on a Bomem MB series. The initial basal spacing in the fiber matrix is an important parameter for the determination of the potential for polymer intercalation. This was determined by X-ray diffractometer for ‘p’, ‘c’ and p–c blended fabrics treated with nano-copper sol. The scans were performed for each sample and the values are reported for the basal spacing. The X-ray diffraction patterns were obtained using diffractometer D8 advance with $\text{CuK } \alpha$ radiation ($\lambda = 1.54 \text{ \AA}$).

Dyeing of untreated and treated woven fabrics with natural dye

A study on the dyeing behavior using Myrobolan on woven fabrics loaded with copper nano sol was also done, in order to ascertain the change in their dyeing performance. This was executed on laboratory scale instrument, applying exhaust dyeing technique and maintaining constant dye bath temperature (Paramount instruments Pvt. Ltd., India). The dye bath liquor containing natural dye 20% (on weight of fabric-owf) and acetic acid ~2% (owf) was added to maintain dye bath pH of 5.5 in case of ‘p’ fabric whereas 2% (owf) sodium carbonate was added while dyeing ‘c’ fabric; maintaining material-to-liquor ratio of 1:40. Dyeing was started at room temperature and gradually

raised to boil within 15 min; continued till 30 min at same temperature. Finally the samples were thoroughly washed and air-dried.

Dyeing of p–c blend fabric was initially started with 20% (owf) natural dye; the temperature was gradually raised to boil within 15 min and continued for 30 min. 2% (owf) sodium carbonate was then added to the dye bath and the dyeing was continued further for 15 more minutes. The samples were thoroughly washed, neutralized, and dried.

The dyed samples were assessed for *K/S* values using computer colour matching system (Spectra scan 5100 RT Spectrophotometer, Premier Color scan Instrument Pvt. Ltd., Mumbai/India). The light fastness of the dyed samples was tested on Fad-o-meter (FDA-R, Atlas Material Testing Technology LLC, Chicago, Ill/USA) and the wash fastness test of the samples was performed as per ISO-2 test methods in launder-o-meter (Digi.wash, Paramount Scientific Instruments Pvt Ltd., New Delhi/India). The samples were evaluated for the rating in terms of colour change.

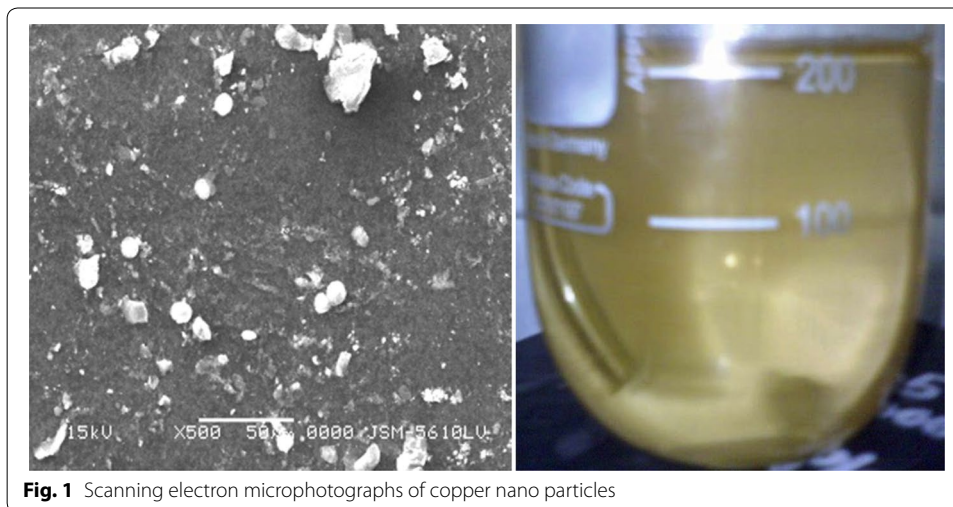
Results and discussion

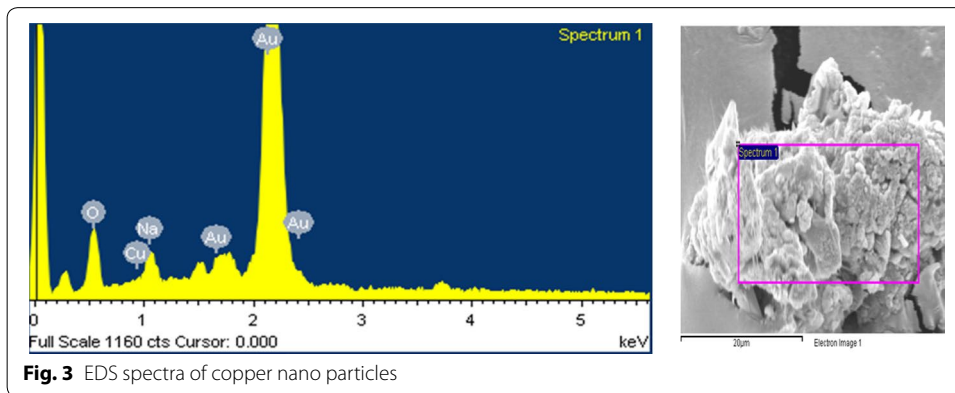
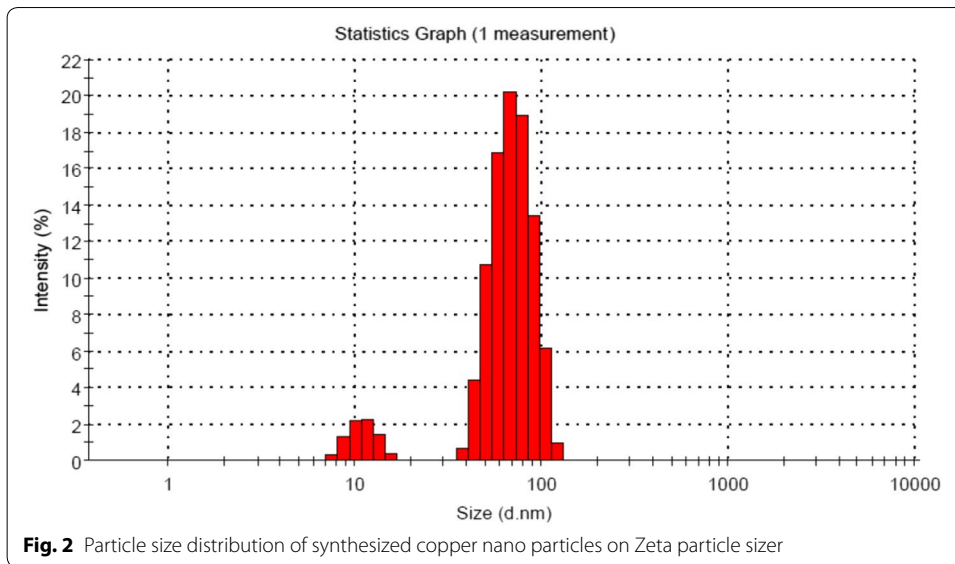
Morphological analysis of nanoparticles

Figure 1 shows the scanning electron micrographs of nanoparticles sample. The average size of nanoparticles as seen from the scale of the photograph, is approximately in the range of 90–150 nm which is further confirmed by particle size analysis on Zeta particle size analyzer as shown in Fig. 2. It can also be seen from the same figure that the shape of synthesized particles is spherical.

Elemental analysis of nanoparticles

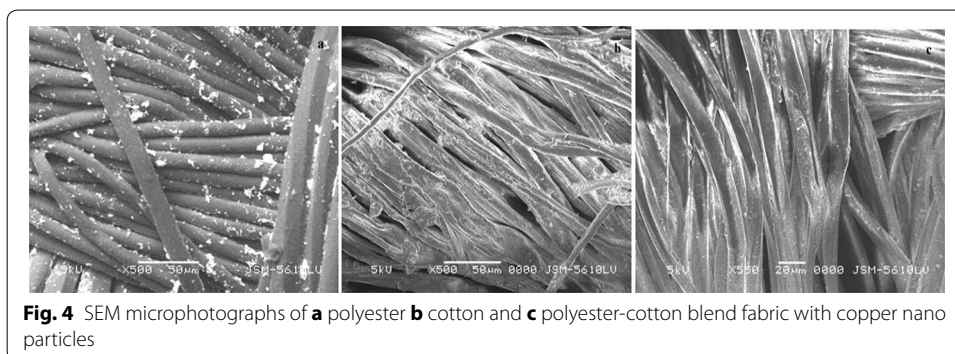
Elemental analysis of nanoparticles was performed using the scanning electron microscope with oxford-Inca software. The spectrum recorded for the particle is shown in Fig. 3, which confirms the presence of copper along with oxygen; giving indication that the nano particles are in their oxide form (CuO_2). The peaks for Au, carbon (C) and Sodium (Na) are visible.





Surface morphology of nano copper loaded fabric

The surface morphology of copper loaded ‘p’, ‘c’ and p–c fabrics is shown in Fig. 4 a, b and c respectively. The nano scale copper particles can be seen distributed on the entire fabric surface for all the fabric samples. However in case of polyester minor agglomeration is seen from the microphotographs. The particle size plays a primary role in



determining their adhesion to the fabric. However the nano particles in agglomerate form remain smaller than the polymer matrix. It is reasonable to expect and assume that the particles whether in agglomerated form or individual can penetrate deeper and adhere strongly on/into the fabric matrix.

Fourier Transform Infrared analysis

Figures 5a, b, c and 6a, b, c shows FT-IR spectra of untreated and treated 'p', 'c' and p-c blend fabrics respectively. The 'p' fabric (Fig. 5a) shows typical band characteristics due to the presence of different functional groups like: CH₂-anti-symmetric stretching at wavelength of 2945 cm⁻¹, carbonyl bond stretching at wavelength of 1725 cm⁻¹, standard absorption can be seen at wavelength of 1410 cm⁻¹ due to OH bond (Ibrahim et al. 2010). Besides these, CH-stretching second overtone at wavelength of 1143 cm⁻¹, O-CH₂ stretching at wavelength of 973 cm⁻¹, and benzenoid group at wavelength of 868 cm⁻¹ are also visible. After treatment no significant change can be observed in the absorption spectrum (Fig. 6a). However minor changes in the peak values recorded can be attributed to the given copper nano treatment.

Figure 5b shows the FT-IR spectra of the untreated 'c' fabric samples. The fabric samples show a strong absorption band between the wavelengths of 3000–3500 cm⁻¹. The vibrations located at 1161 cm⁻¹ can be attributed to the anti symmetric bridge C–O–C stretching vibration, 1105 cm⁻¹ can be attributed to the anti symmetric in-plane ring stretching band and 1056 cm⁻¹ to C–O stretching mode.

After treatment the absorption band at wavelength of 2918 cm⁻¹ becomes more prominent (Fig. 6b) in case of 'c' fabrics. Most of the peaks match each other almost exactly except the peak created at 2918.82 cm⁻¹. It thus confirms the presence of hydrogen bond due to cellulosic nature of the parent fabric. This absorption band can be attributed to the asymmetric vibrations of –CH₂ bonds. The broad absorption peak at around 3430 cm⁻¹ is caused by the absorbed water molecules since the nano crystalline material exhibits a high surface to volume ratio and thus absorbs more amount of moisture. It is known that the usual absorption pattern in this region can be attributed to the intermolecular and intra-molecular hydrogen bonds (Ibrahim et al. 2010) of which absorption peak at 3335 cm⁻¹ is due to intermolecular hydrogen bond.

Figure 5c shows the FT-IR spectra of untreated p–c blended fabrics. The OH and C=O absorption band at 3500 and 1600 cm⁻¹ is respectively due to OH bond stretching and anti-symmetric stretching of carbonyl group present; attributed to the presence of cotton component. The anti-symmetric stretching band at 2945 cm⁻¹ is due to CH₂ bond, carbonyl bond stretching at 1725 cm⁻¹, the standard stretching bond of OH at 1410 cm⁻¹, O-CH₂ stretching at 973 cm⁻¹ and benzenoid group (Das et al. 2009) at 868 cm⁻¹ are present by default. It will be seen that the absorption spectrum of treated p–c blended fabric samples does not show much of change (Fig. 6c). However on treatment with copper nanoparticles, additional absorption bands can be noticed at 2922.50, 1408.84, 1339.09, 894.39 and 871.81 cm⁻¹ while other absorption spectrum remains unchanged. From results obtained for 'p' and 'c' fabrics, it becomes clear that the copper nano treatment was able to influence the cotton component while the polyester component was least affected. The changes thus observed in the absorption spectrum after treatment for p–c blended fabrics (Fig. 6c) may be due to the changes that the cotton component may have undergone.

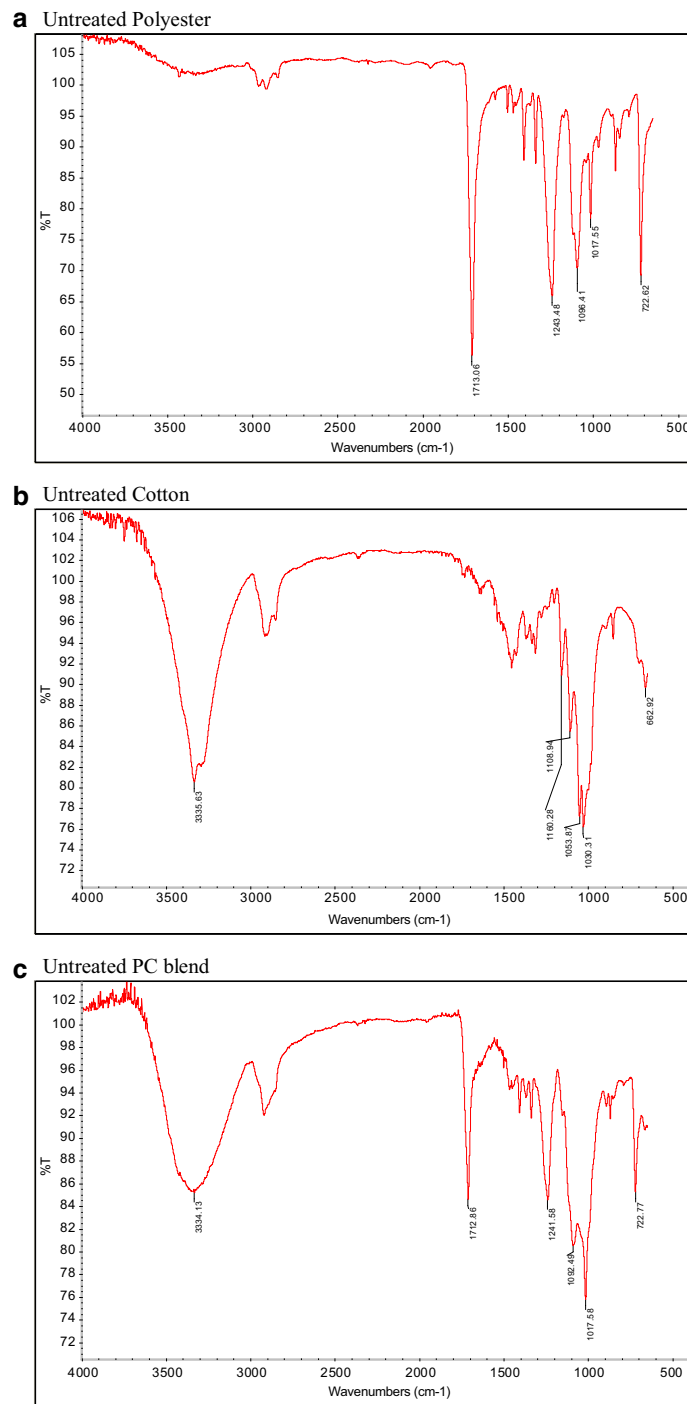
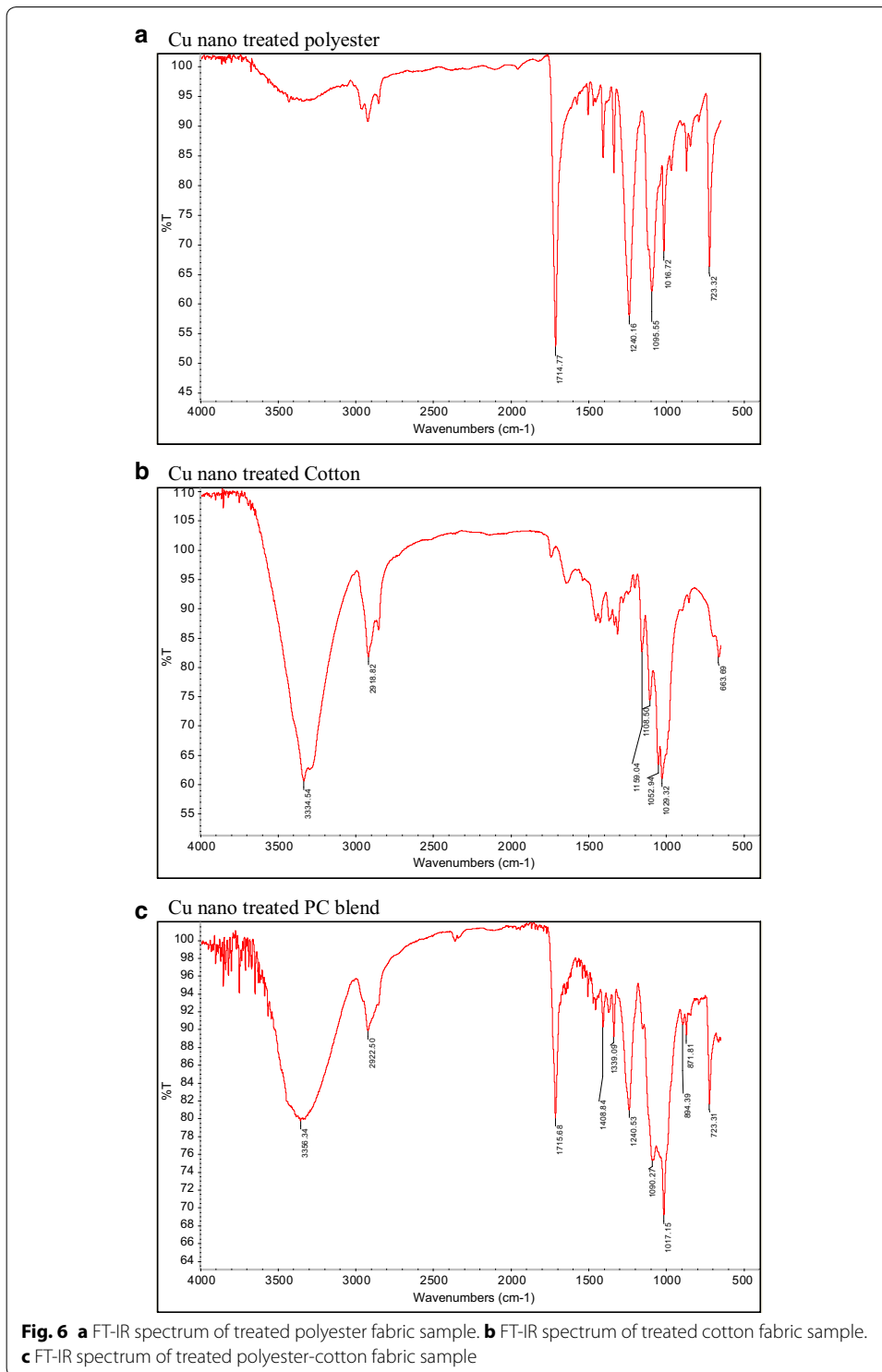


Fig. 5 **a** FT-IR spectrum of untreated polyester fabric sample. **b** FT-IR spectrum of untreated cotton fabric sample. **c** FT-IR spectrum of untreated polyester-cotton fabric sample



Elemental analysis of copper nano coated fabrics

Elemental analysis of treated woven fabrics on SEM is in form of spectra as shown in Fig. 7a, b and c. Presence of copper was confirmed by the elemental analysis curve for individual fabric samples. The inserted data in corresponding figures is the overall percentage of copper added as a result of the treatment given to the fabrics.

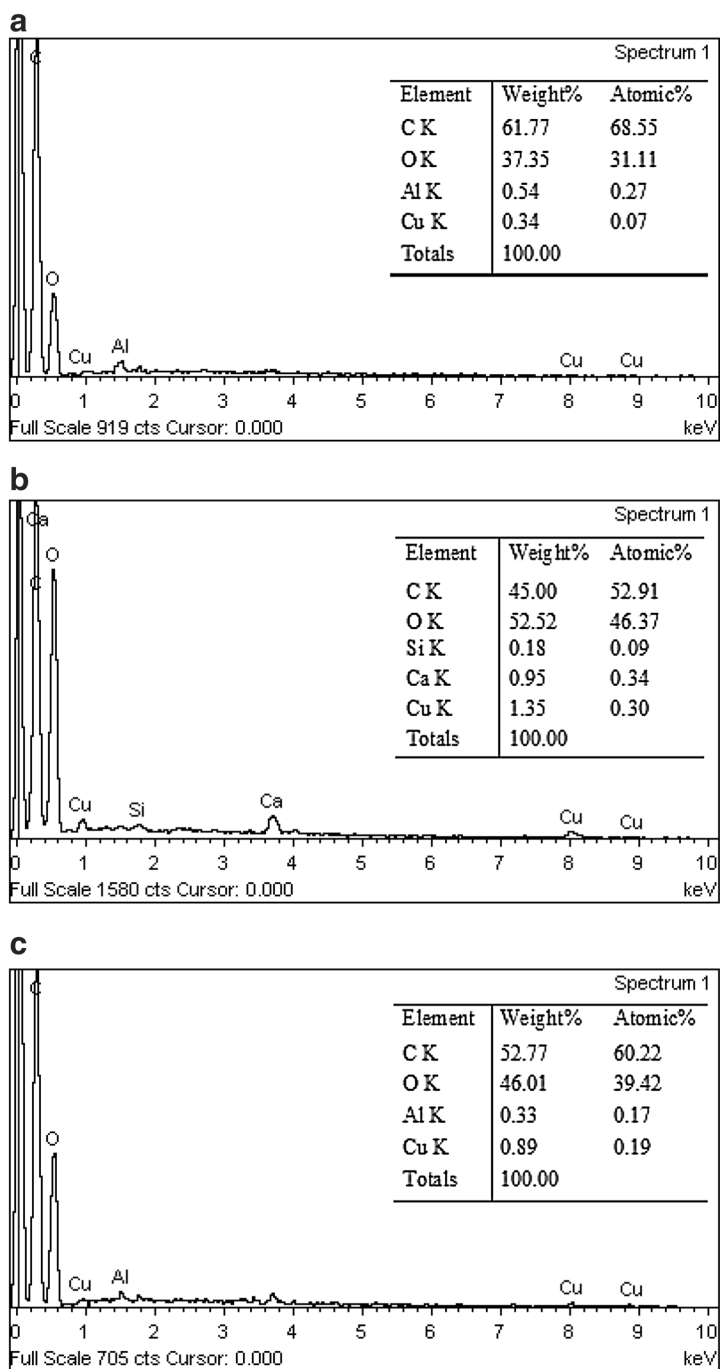


Fig. 7 **a** Elemental analysis of copper nano sol coated polyester fabric. **b** Elemental analysis of copper nano sol coated cotton fabric. **c** Elemental analysis of copper nano sol coated polyester cotton blended fabric

XRD analysis of copper nano loaded fabrics

The nature of copper nano coated woven fabrics was studied with the aid of X-ray diffraction as shown in Fig. 8a, b and c. Three peaks at 2θ values of 42.91, 51.02, and 73.96 degrees corresponding to (111), (200) and (220) planes of copper is observed. The XRD study confirms/indicates that the resultant particles are copper nanoparticles (Chattopadhyay and Patel 2013) and also indicates that they are crystalline in nature.

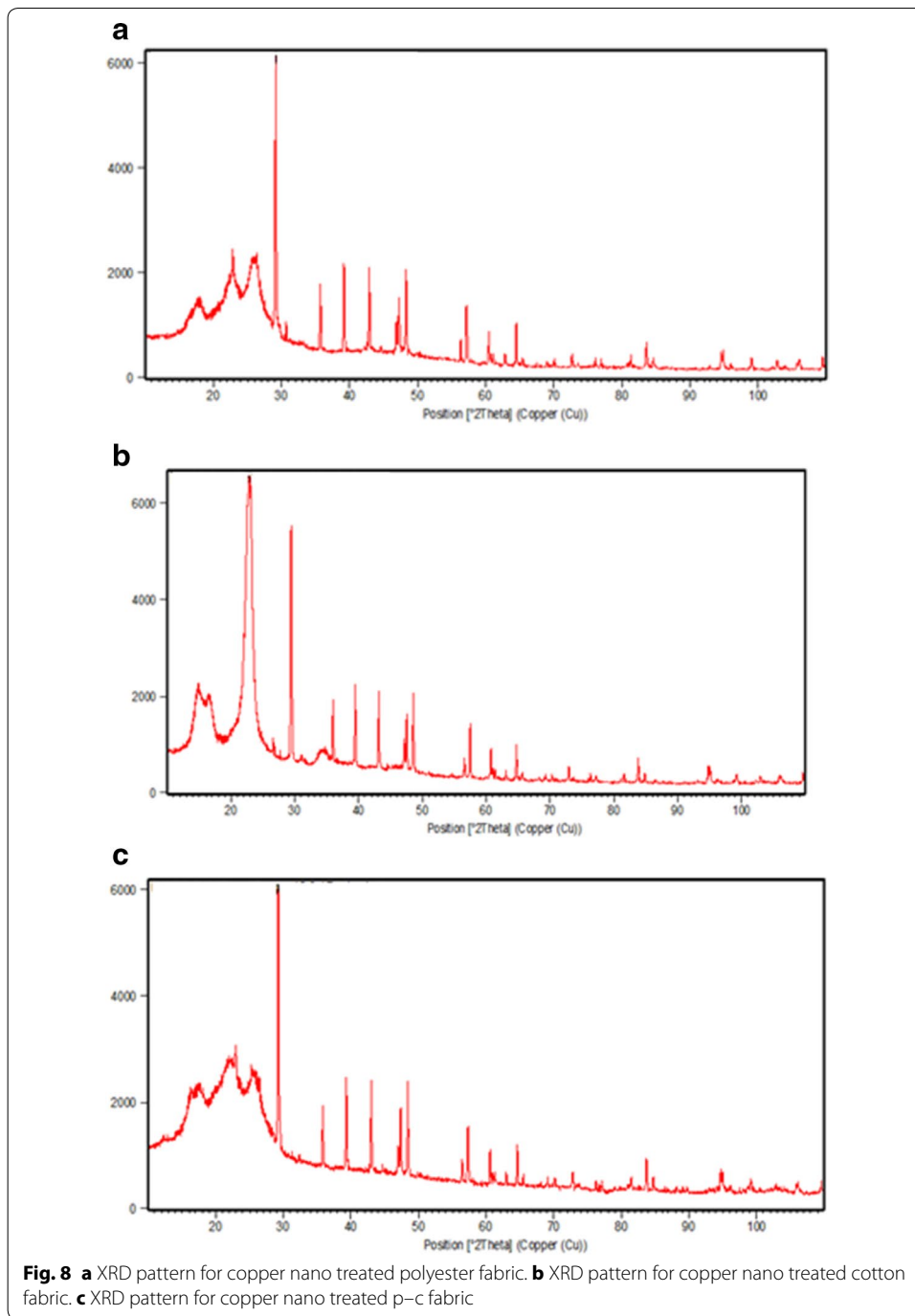


Table 2 shows results obtained from the XRD spectrum for the treated woven fabrics. The 2θ values for all of treated fabrics are similar; ‘p’ and p–c blended fabrics show the maximum relative intensity.

It has also been observed that the flex width and d-spacing value in polyester-nano copper structure was higher compared to the cotton-nano copper structure or p–c blended-nano copper structure. The d-spacing value of cotton-nano copper structure is lower than polyester- nano copper structure. It may be due to the trapping of copper

Table 2 XRD analysis of Cu loaded polyester, cotton and P-C blend fabric

Sample	2θ	D-spacing	Flex width	Height	Rel. intensity
Polyester	29.12	3.06	1065.43	4782.67	100.00
Cotton	29.43	3.03	1032.92	4636.75	83.38
P-C blend	29.29	3.05	1200.38	4849.64	100.00

Table 3 Colour strength values of woven fabrics dyed with natural colourant

Fabric sample	Colour strength (K/S) values	Fastness ratings	
		LF	WF
Untreated polyester	1.24	7	4–5
Polyester treated with copper nano	1.96	6–7	4
Polyester treated with CuSO ₄	2.33	6–7	4–5
Untreated cotton	3.45	6–7	4
Cotton treated with copper nano	5.35	6–7	4–5
Cotton treated with CuSO ₄	5.87	6–7	4
Untreated P-C blend	2.47	6	4
P-C blend treated with copper nano	2.51	6–7	4
P-C blend treated with CuSO ₄	4.26	6–7	4

K/S Colour Strength value, *LF* Light Fastness, *WF* Washing Fastness

nanoparticles inside the ‘p’ polymer matrix which results in increased intermolecular spacing while in ‘c’ and p-c blended fabrics, the d-spacing value was found to be comparatively lower. The amorphous nature of cotton makes it incapable of trapping the nano sized copper particles.

Effect of copper nano treatment on dyeing

The copper nano-loaded woven fabrics treated with natural dye (Myrobolan 20% owf depth of shade) were compared with the untreated samples. The results in Table 3 show that the *K/S* values of the nano copper pretreated samples are higher than those of the corresponding untreated samples of woven fabrics. The higher *K/S* values (Table 3) of nano-treated samples indicate that the presence of nano metal particles increases the dye affinity towards the material. The copper nanoparticles in the fabric thus act as mordant. The negatively charged dye anions get attracted towards the fiber probably due to the polarity developed in the metal particles by induction which results in better bonding between the dye and the fiber. The better coupling of the dye and the fiber is also reflected in the improvement in the colour fastness properties (Table 3). Thus copper nano pre-treatment not only improves the colour strength but also improves the colour fastness which is a major drawback of most of natural dyes.

Conclusion

Plain woven ‘p’, ‘c’ and p-c blended fabrics were coated with nano sized copper by pad-dry-cure method. The surface morphology of woven fabrics was characterized by SEM technique and shows uniform distribution of nanoparticles throughout the fabric surface. The coated woven fabrics were further characterized by EDS and FT-IR technique;

confirming the presence of copper nanoparticles on/in coated woven fabric samples. The structure of the prepared composite woven fabrics was analyzed by X-ray diffraction technique, which indicates that the d-spacing value of polyester polymeric structure was higher than the highly amorphous structure of cotton and p–c blended fabrics. In case of dyeing the improvement could not exceed that obtained using bulk conventional mordanting agent but introduction of nano copper in the system enhanced the wash fastness of dyed fabric samples. Dyeing of the nano copper loaded woven fabrics with natural dye (Myrobolan) also proved to be beneficial.

Authors' contributions

PK acquired the fabric samples and carried out the structural and elemental testing of fabric samples. BP gave copper nano treatment to the fabric samples and analysed the test results. The manuscript was jointly drafted by both the authors. Both authors read and approved the final manuscript.

Competing interests

The authors declare that they have no competing interests

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