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Novel conductive textile fabric based on polyaniline and CuO nanoparticles

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Abstract : In this study, a novel conductive fabric in view of polyaniline/CuO (PANI/ CuO) has been successfully obtained. Different sequence of treatment was applied by aniline to obtain different fabric. The polymerization of aniline on cotton fabric happened in situ to prepare polyaniline (PANI) sample in presence of ammonium persulfate. CuO nanoparticles were formed on cotton fabric via ultrasound-assisted template method. Three cotton fabric samples obtain by different method of treatment .First sample obtained when polyaniline treated sample exposure to CuO nanoparticles on the CuO nanoparticles solution, second one obtained when CuO treated cotton fabric undergo polymerization reaction by aniline monomer .the third samples obtained when cotton samples is treated with PANI/CuO nanocomposite. The morphology the novel garment samples was characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD), and Fourier transform infrared (FT-IR) analyses. .The electric conductivity of the obtained samples was investigated, the most extreme interaction between CuO and polyaniline happens for lower CuO nanoparticles loading, as the electric conductivity reach maximum. The antibacterial activity of the obtained samples was also examined against Gram positive bacteria Staphylococcus aureus (*Staph. aureus*) and Gram negative Escherichia coli (E. coli) as well as fungus (Candida albicans) C. albicans using disk diffusion method. The aforementioned study demonstrated that, all fabric samples have a very good antibacterial activity and ***were found to be effective against two bacteria.***
Key word: CuO nanoparticles, electric conductivity, polyaniline, antibacterial activity.

Introduction

Recently the improvement of fabrics with new properties and applications has become extraordinary thought; the electrical conductivity is considered as one of these properties. Different techniques have been utilized to create leading fabrics, such as treatment of fabrics with conducting polymers[1].

Smart textiles speak to the up and coming era of fibres, fabrics and articles delivered from them[2]. They can be depicted as textile materials that think for themselves, for instance through the incorporation of electronic devices or smart materials. Various smart textiles as of now highlight in cutting edge of garments, primarily for protection and safety and for included fashion or convenience.

One of the primary reasons for the rapid development of smart textiles is the vital venture make by the military industry.

Distinctive works have been concerned with the generation of polypyrrole (PPy) or polyaniline (PAN) treated fabrics to deliver conductive fabrics[3-8]. The utilizations of polyaniline coated fabrics are for instance as: ammonia sensor[3], electromagnetic[4-6] static charge dissipatio[5] electromechanical devices[6] or precious metals recovery[7].

Conducting polymers such as polyaniline (PANI) and polypyrrole are turning out to be progressively vital for their innovative significance due to their optical, electrical properties and their high air, electrical and chemical stability at ambient conditions[8-10]. Another approach to manage enhance process ability is the usage of PANI colloidal dispersions[11]. polarons and bipolarons are considered the charge carriers of the PANI ,stabilized by counter ions incorporated into the polymer during synthesis Therefore, the choice of counter ion which called dopent affected to great extent on physical and conducting properties of PANI. In the recent years, inorganic nanoparticles incorporated polyaniline nanocomposite materials has attracted much interest worldwide because of the improved stability, conductivity and unique optical properties, etc[12-14]. Copper oxide (CuO)is considered as a versatile semiconductor materials and it is pulling in account because of the commercial demand for optoelectronic devices working at blue and ultraviolet regions[15]. CuO is a monoclinic n-type semiconductor with narrow band gap energy of 1.5–1.8 eV, furthermore it has very large excitation binding energy (60 meV) at room temperature[16,17]. Recently CuO has wide applications because of its exotic properties[18,19]. It has reach application in various areas, such as electromagnetic anticorrosion coatings, shielding device, photodetectros, lightweight battery electrode, sensors and solar cells, [20-23]. Because it has environmental stability and good mechanical flexibility, and it's controlled resistivity with acid/base (doping/undoping),

The point of the present study aim to explore the impact of different treatment sequence by polyaniline of cotton fabrics i)polyaniline coated fabric, ii) CuO nanoparticles pretreated fabric coated with aniline, iii) polyaniline pretreated fabric coated with nano CuO and iv) po;lyaniline /CuO nanocopmpsite coated fabric. After the treatment process, the structural properties of the fabrics were determined with XRD, EDX, SEM and FTIR. Moreover, the electrical conductivity as well as antibacterial properties of coated cotton fabric were measured.

2. Materials and methods

Mill bleached 100% cotton fabric (230 g/m2) All chemicals were of analytical reagent grade and used without further purification Aniline was purchased from (Sigma–Aldrich) hydrochloric acid (HCl, 37%, Sigma–Aldrich), ammonium peroxydisulfate $(NH4)_2S_2O_8$, other chemicals were used as received.

2.1 Synthesis of CuO nanostructures on cotton fabric

In a typical experiment, 6.040 g $Cu(NO_3)2.3H_2O$ was put into 50 mL distilled water under stirring to form a homogeneous solution. 0.2 g of cotton fabric sample was immersed in the above solution and then the mixture was under ultrasonic treatment (40 kHz, 150 W at 100% efficiency, KQ3200DB model, Kunshan, China) for 20 min at room temperature to make cotton fibers infiltrate fully. Subsequently, 12 mL of 25% $NH_3.H_2O$ was added into the above solution drop by drop under vigorous stirring and keep stirring for 30 min until a stable complex of $Cu(NO_3)_2$ and $NH_3\_H_2O$ was formed (pH = 8). After that, 50 mL of 1.0 mol L_1 NaOH solution was dropped slowly into the mixed solution (pH = 12) under vigorous stirring and then ultrasonic treatment for 1 h at 40°C. A strong blue color was gradually converted into brown after 15 min. The bath temperature was kept at a constant temperature around 40°C.After completing the reaction in desired time the small pieces of cotton fibers was obtained which was collected and washed with distilled water and ethanol several times ***to remove excesses of sodium hydroxide*** and dried in drying oven at 130 ºC for 24 h.

2.2 Synthesis of polyaniline cotton fabric

Washed and dried cotton samples were allowed to soaked in aniline solution(0.02 mol) After that aqueous solution of APS (1 mol)was added drop wise. The temperature was maintained at 0-5 ^oC and keeping under stirring for 4 hr .As the polymerization proceeds, the fabric samples turns green color which is the color of PANI. After completion of the duration of polymerization, the in situ polymerized polyaniline cotton fabric was washed in dilute HCl to remove untreated chemicals and oligomers, the fabric was then dried and weighed.

2.3 Synthesis of aniline-CuO composite on fabric

Equi-volume amount of both polyaniline solution and CuO nanoparticles solution will mix by using a homogenizer**.** Cotton fabric sample was immersed in the above solution for 15 minutes. the treated sample were washed then dried .

2.4 Synthesis of polyaniline-CuO treated fabric

Aniline treated sample was immersed in solution of $Cu(NO_3)_2$. $3H_2O$ under ultrasonic treatment for 20 min at room temperature to make cotton fibers infiltrate fully. Subsequently, 12 mL of 25% $NH_3\_H_2O$ was added into the above solution drop by drop under vigorous stirring and keep stirring for 30 min until a stable complex of $Cu(NO_3)_2$ and NH_3H_2O was formed (pH = 8). After that, 50 mL of 1.0 mol L_1 NaOH solution was dropped slowly into the mixed solution (pH = 12) under vigorous stirring and then ultrasonic treatment for 1 h at 40°C. A strong blue color was gradually converted into brown after 15 min. The bath temperature was kept at a constant temperature around 40°C.After completing the reaction in desired time the cotton fabric sample was obtained , collected and washed with distilled water and ethanol several times ***to remove excesses of sodium hydroxide*** and dried in drying oven at 130 C for 24 h.

2.5 Synthesis of CuO-polyaniline treated fabric

CuO-polyaniline treated fabric was synthesized by in situ chemical oxidation polymerization of aniline monomer in presence of CuO nanoparticles treated cotton fabric sample. The polymerization process occurs as per section 2.2

3. Characterization

3.1 Fourier-transformed infrared spectroscopy (FT-IR)

FTIR spectroscopy has been extensively used in cellulose research, since it presents a relatively easy method of obtaining direct information on chemical changes that occur during various chemical treatments. FT-IR spectra were recorded using a S-100 FT-IR spectrometer (Perkin Elmer) and scanned from 4000 to 400 cm^{-1} in ATR mode using KBr as supporting material.

Characterization of samples using FT-IR technique was carried out to follow the change in the functionality

3.2 X-Ray Diffractometry (XRD).

X-ray diffraction (XRD) patterns of finely coated fabrics with metals were recorded on a Philips

PW3040 X-Ray diffractometer system by monitoring the diffraction angle from 5° to 80° (2θ) at 40keV.

3.3 Scanning electron microscopy (SEM)

SEM was studied using a scanning electron – JSM-5400 instrument (Jeol, Japan). The specimens in the form of fabrics were mounted on the specimen stabs and coated with thin film of gold by the sputtering method.

3.4 Energy dispersive X-ray analysis (EDX).

The elemental analysis was performed using EDX, which is an attachment to the scanning electron microscopy. The spectra obtained during EDX studies were used for carrying out the quantitative analysis.

4 Electrical conductivity properties.

The electrical conductivity of the dried fabrics composite were determined at ambient room temperature (25^0C) using a Digital Multi-meter. Electrical measurements were recorded by means of an electrical circuit composed by a Hewlett Packard 6634B System DC Power Supply and a digital Hewlett Packard 34401A Multimeter.

$Conductivity = 1/R_S$;

Where, R_s is the surface resistance. Surface resistance was measured according to the American Association of Textile Chemists and Colourists Test Method 76-1995 [18]. Two rectangular copper electrodes(20 X 30 mm^2) separated by 20 mm were placed on the fabric sample (30 X 60 mm^2) by a 1-kg mass. Surface resistance (R_s) is given by:

$$R_S(\Omega/square) = \frac{W}{D}R;$$

where R is the resistance measured by the multimeter, and W and D are the width of the sample and the distance between the two electrodes, respectively.

5. Antibacterial activity:

The antibacterial activity of the treated samples against Staphyllococcus aureus (G +ve) and Escherichia coli (G−ve) bacteria were determined using agar plate. The plates were incubated at 37◦C for 24 h a growth free zone of inhibition around the fabric appears as antibacterial agent migrates from the fabric onto the agar and diffuses outward. Diameter of inhibition zone was determined according to AATCC test method 100-199.

4. Result &discussion

4.1 Synthesis of CuO nanoparticles coated fabrics

The CuO-coated cotton fabric was gotten by application of CuO nanoparticles onto the cotton fabric via ultrasound irradiation of metal hydroxide according to the reaction in a similar way previously reported[24]

$$Cu^{2+} + 2OH^- \rightarrow Cu(OH)_2$$
$$Cu(OH)_2 \rightarrow CuO + H_2O$$

After the addition of OH−, a blue fresh product Cu $(OH)_2$ is formed promptly, which changed to brown color of copper oxide after a few minutes of sonication. The CuO nanoparticles produced by the reaction were probably physically adsorbed onto the surface of the natural cotton fibers by the sonochemical micro jets coming about because of the collapse of sonochemical bubbles [25].These nanoparticles are strongly physically adsorbed onto the cotton substrate.

4.2 XRD analysis of CuO nanoparticles coated cotton fabric

The XRD pattern of the coated cotton fabric reveals that copper oxide is present in crystalline form on to the cotton fibers. The pattern corresponds to the monoclinic phase of CuO; the diffraction peaks are in great concurrence with the standard JCPDS card 48-1548 with monoclinic crystal structure. The peaks at 23° and 35° indicate the exictance of cellulose in cotton fabrics. The peaks at 34,05 and 39.62 for copper oxide impregnated fabrics is for copper oxide (CuO) that was indexed to (-111) and (111) planes, respectively. All moderately wide in light diffraction peaks is related to copper oxide nanoparticles of smaller size[25].

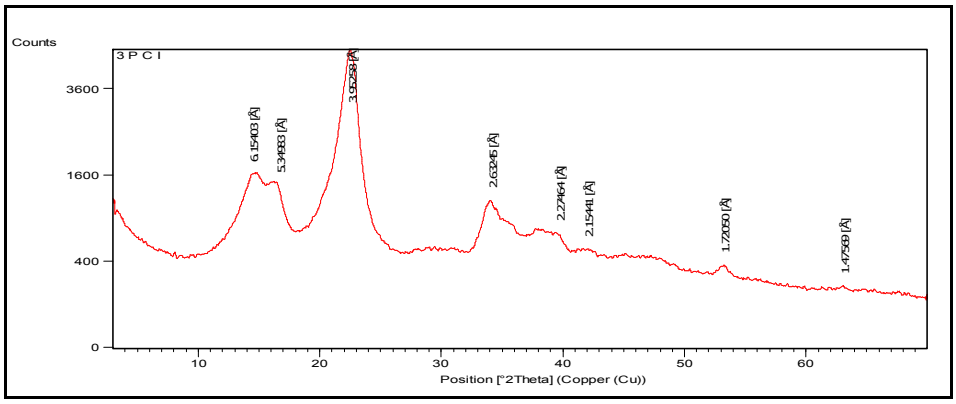
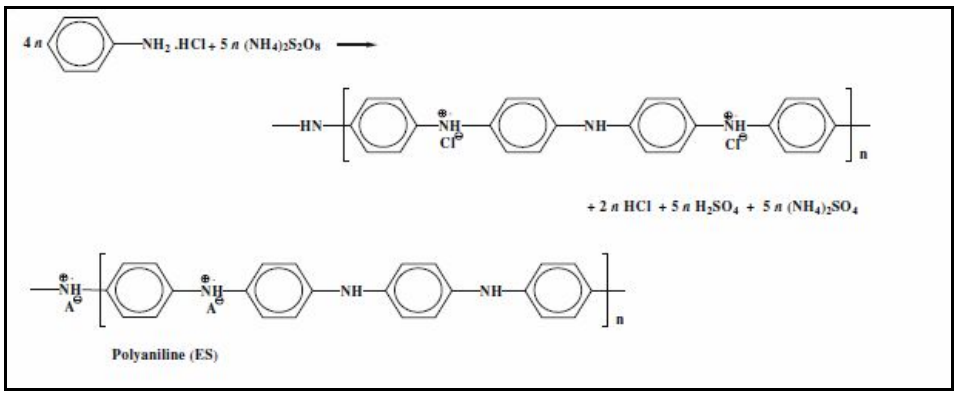


Figure 1 : XRD patterns of coated cotton fibers

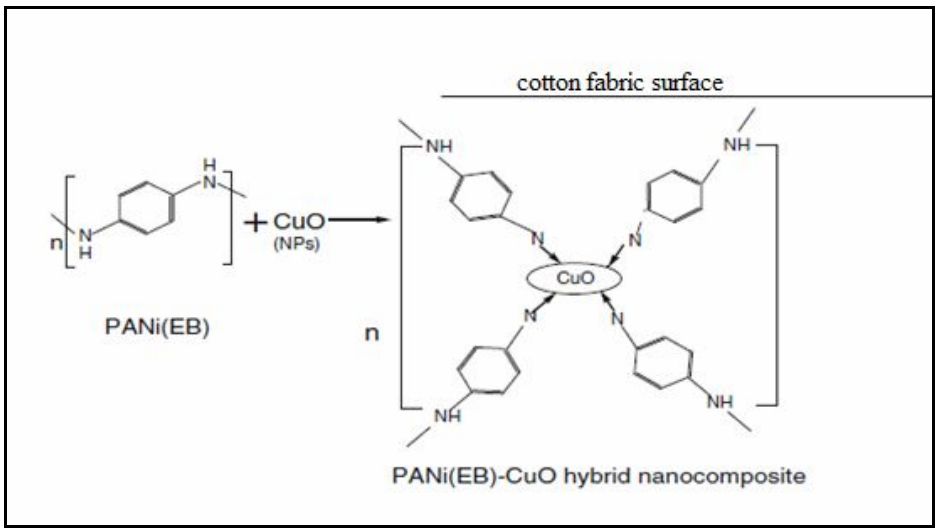
4.3 Synthesis of polyaniline (PANI)

The proposed mechanism of polymerization of aniline on fabric as follow .Polyaniline (PANI) was formed via oxidative polymerization using ammonium persulphate $(NH_4)_2S_2O_8$ as initiator. The ammonium persulphate was added to the acidified aniline solution, the reaction temperature was kept at 0 -5ºC with constant stirring for 5 h .After that time, cotton fabric sample with green colored was obtained[26]



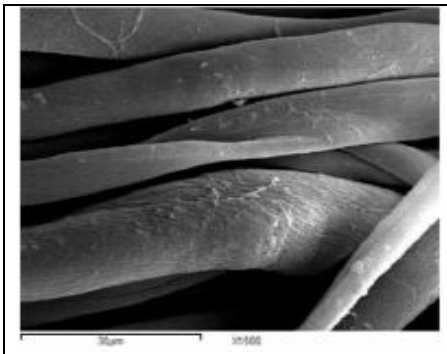
4.4. Synthesis of PANI–CuO on fabric

It was proposed that incorporation of PANI–CuO hybrid nanocomposite on fabric undergo the following reaction mechanism [8]

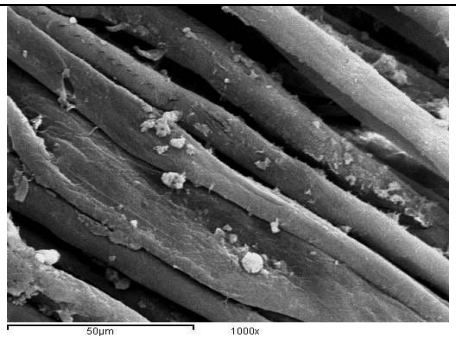


4.5. SEM &EDS analysis

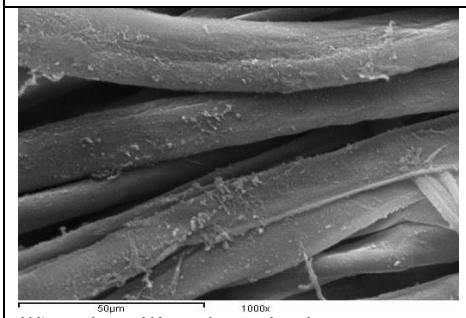
The morphology of the fiber surface area before and after treatments was investigated by SEM and is presented in figure 2. On the SEM image of the original cotton fiber image 2i) grooves and fibrils could be easily noted on the surface of the fiber. Image 2ii) shows the SEM photographs of CuO nanoparticles coated onto cotton fibers. It's clear to see that aggregates of nano particles on the fibers and we can see also distribution of nanoparticles over the fabric threads. For 2iii) image, cotton fabric seems clearly coated by polyaniline . The coated layer is quite smooth and with lower grains size. The morphology of the coating in 2iv) is totally not quite the same as the former. For this type of coating more flaky with higher aggregation of CuO nanoparticles were covered the fibers .It can be further seen for last image 2vi) the fabric is regular and uniform morphology with prevention of agglomeration. The high surface area of CuO nanoparticles is seemed to be the resoan for enhancement the uniformity of polyaniline on the surface of PANI/CuO nanocopmosite functionalized cotton fabric.



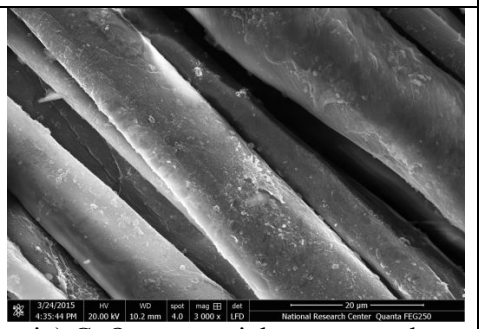
i)Blank cotton fabric



ii) CuO nanoparticles coated cotton fabric

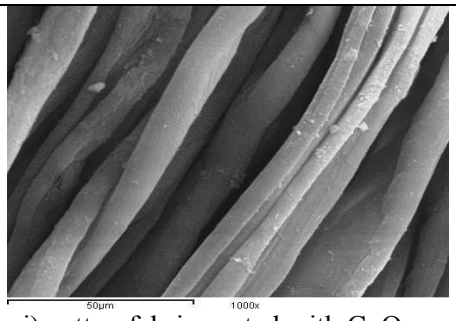


iii) polyaniline deposited cotton fabric



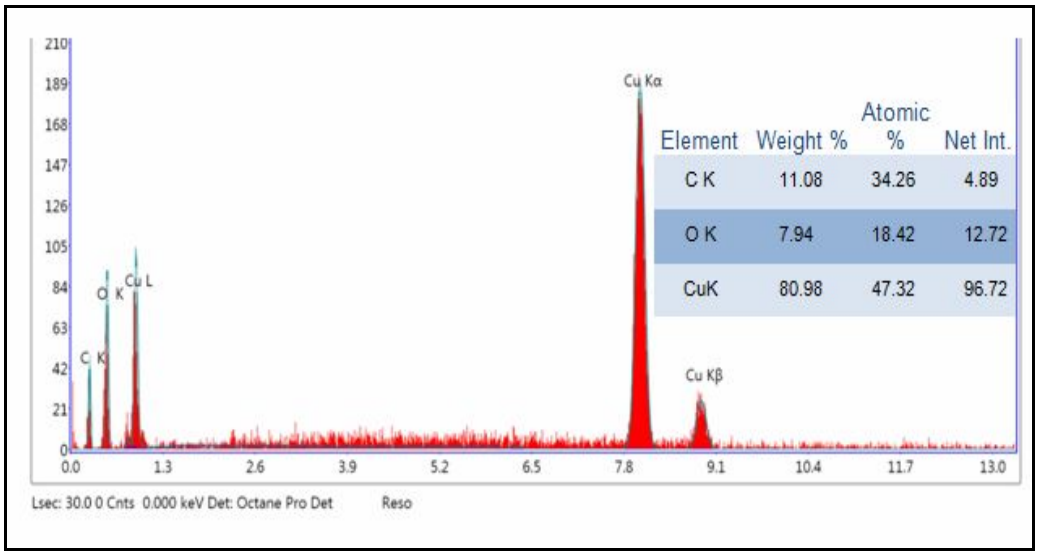
iv) CuO nanoparticles pretreated cotton fabric coated with polyaniline

v) Polyaniline pretreated cotton fabric coated with CuO nanoparticles

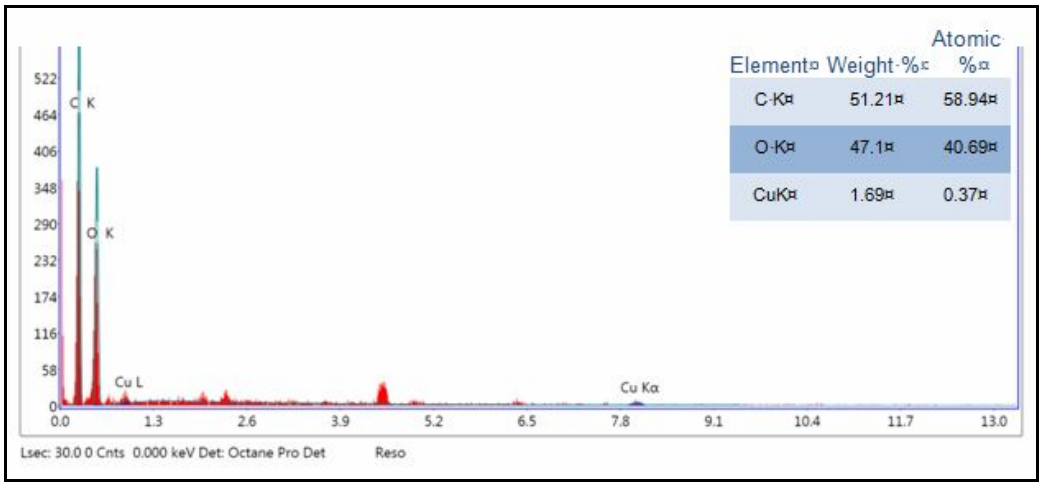


vi) cotton fabric coated with CuO-aniline composite

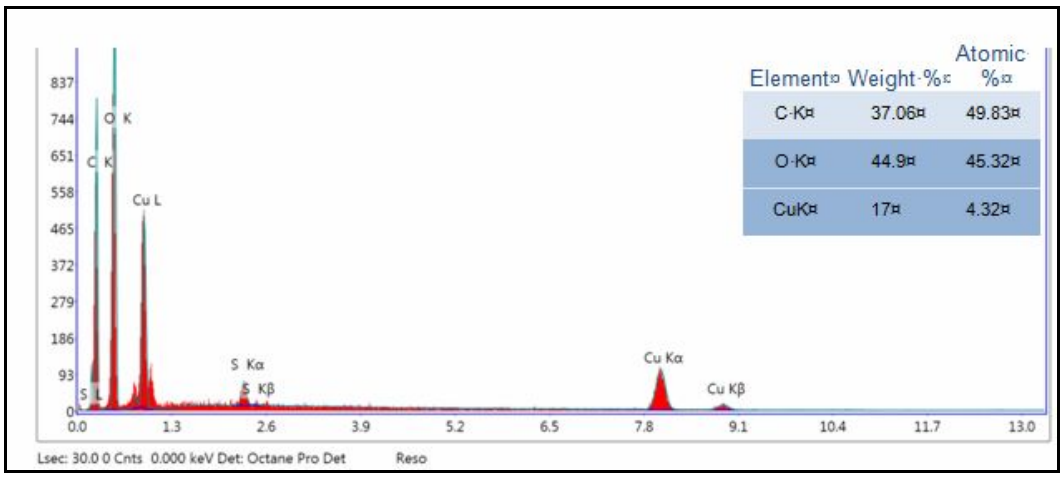
Figure 2 : Scanning electron microscopy images of untreated ad treated cotton fabric



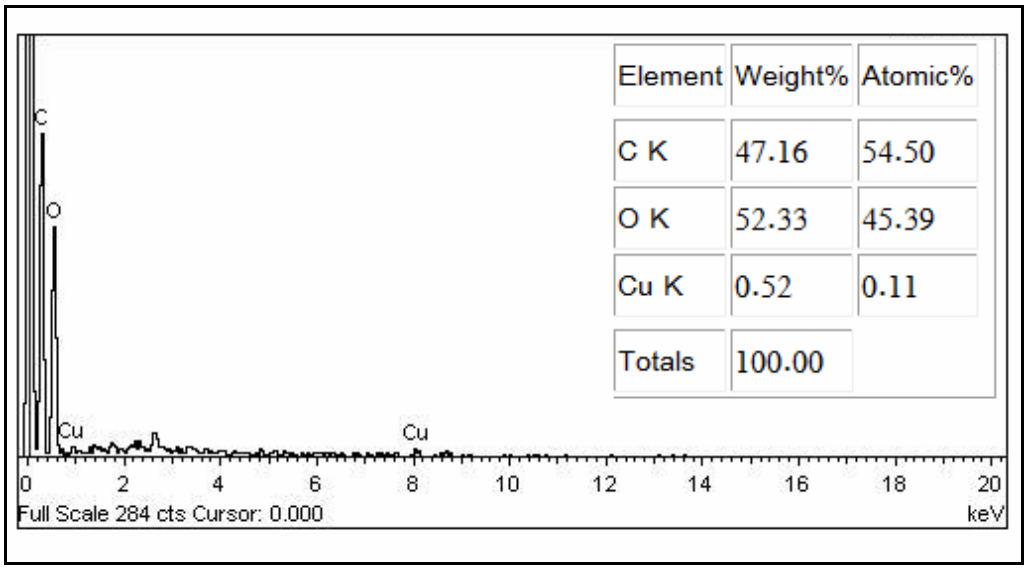
i) CuO nanoparticles coated cotton fabric



ii) CuO nanoparticles pretreated cotton fabric coated with polyaniline



iii) Polyaniline pretreated cotton fabric coated with CuO nanoparticles



iv) cotton fabric coated with CuO-aniline composite

Figure 3: EDX spectra of cotton fabric where 3i) CuO nanoparticles coated cotton fabric, ii) CuO nanoparticles pretreated cotton fabric coated with polyaniline, iii) Polyaniline pretreated cotton

Results of EDS analyses the presence of different CuO weight % according to the different sequence of treatment .the increase in CuO weight % follow the order CuO nanoparticles coated cotton fabric >Polyaniline pretreated cotton fabric coated with CuO nanoparticles> CuO nanoparticles pretreated cotton fabric coated with polyaniline> cotton fabric coated with CuO-aniline composite

4.6FTIR spectra

Figure 5 Shows the FTIR spectra of the samples obtained from treatment of cotton fabric by aniline only and polyaniline cotton fabric with different loading of CuO nanoparticles.

In figure 4a show FTIR of cotton fabric treated with polyaniline ,as shown in figure 5a peaks found at 3472–3100, 3000–2900, 1574, 1474, 1288, 1236,1104, 794 cm−1 are the characteristics peaks of PANI [27]. All characteristics peaks of PANI were also noted for the CuO loadings fabrics as seen in figure 4 b,c,d however The FTIR spectra for cotton fabric treated with PANI and CuO nanoparticle shown in Fig. 4b,c,d , some shift in the wave number as well as change in the intensity of peaks as compared to PANI. Some of the characteristic band shift to lower wavelength 3470 cm^{-1} shift to 3437, 3377, 3432 cm^{-1} and other shift to higher wavelength 1574 cm-1 which is the characteristic band (C=N stretching of quinoid ring). Shift to 1583, 1596, 1597 cm^{-1}

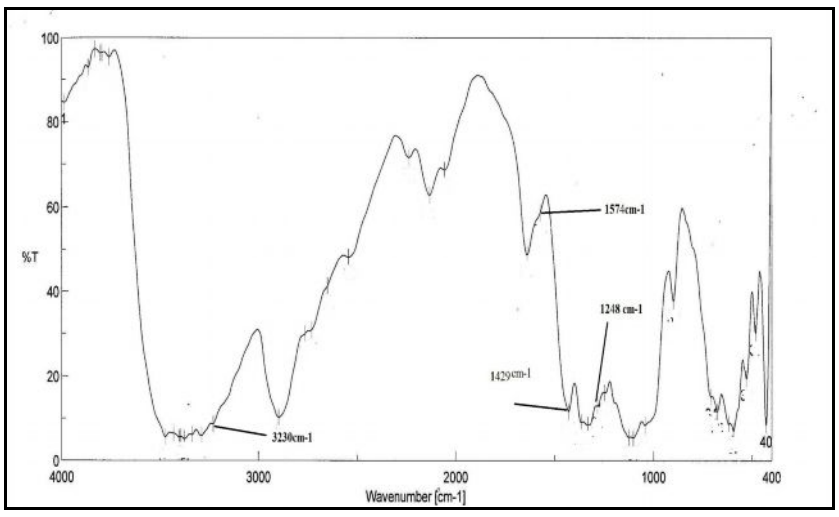


Figure 4a :FTIR aniline

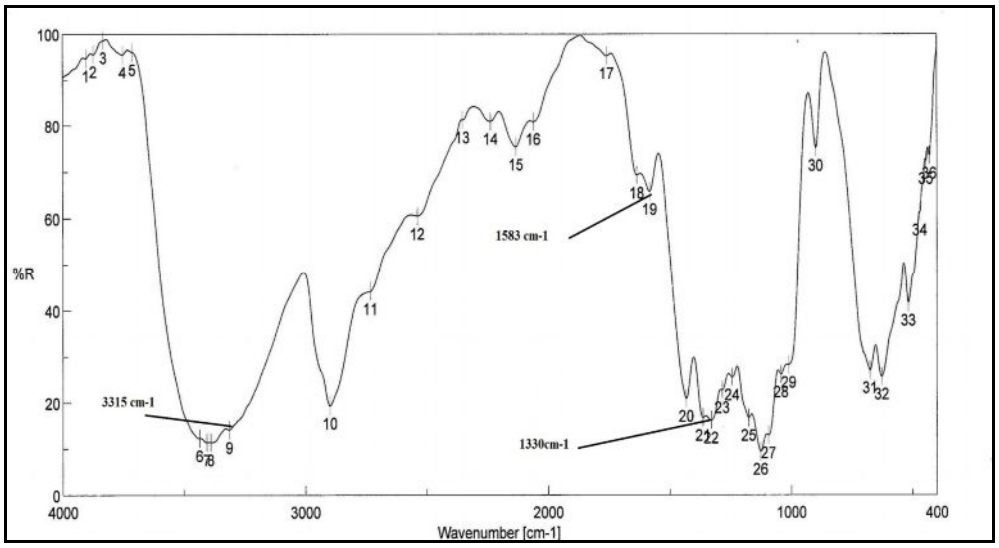


Figure 4b: FTIR cotton fabric pretreated with CuO nanoparticles then coated with polyaniline

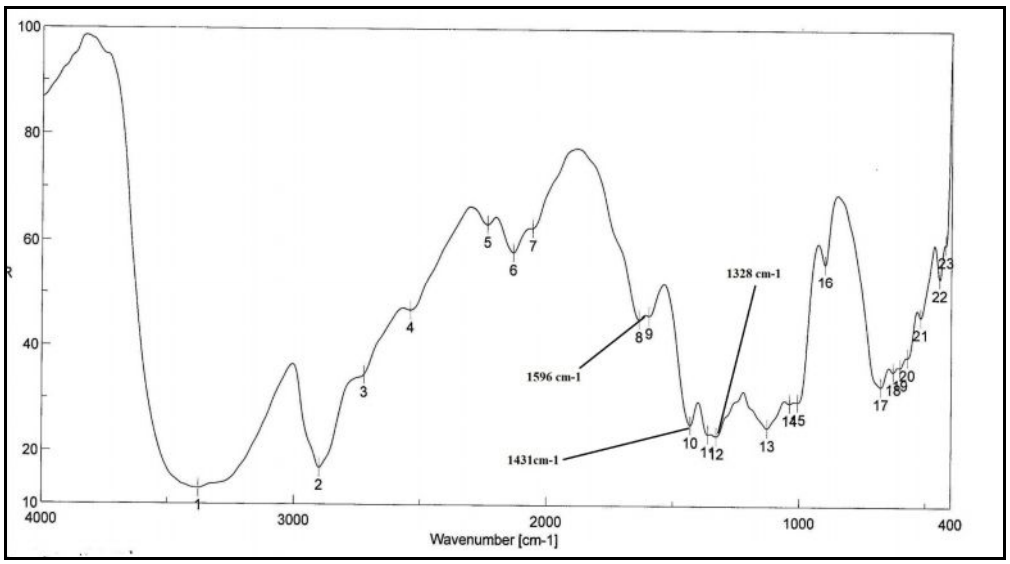


Figure 4c: FTIR) cotton fabric pretreated with Polyaniline then coated with CuO nanoparticles

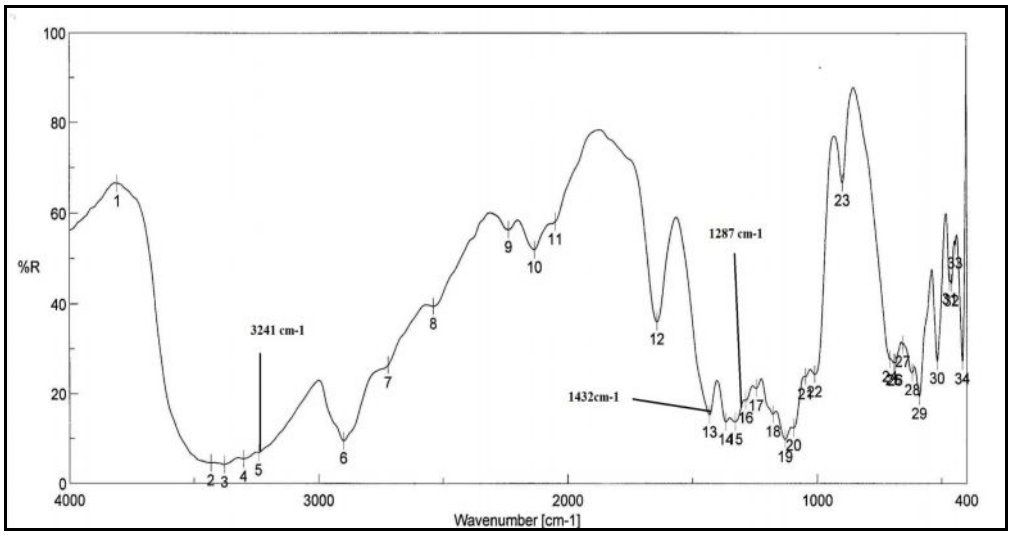


Figure 4d: FTIR cotton fabric coated with CuO-aniline composite

5. Conductivity studies

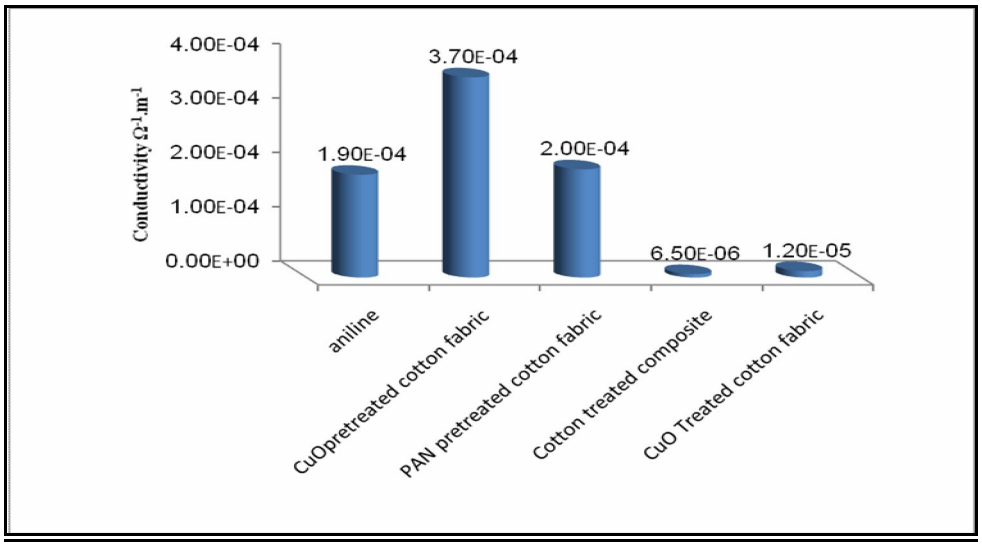


Figure 5: Electrical conductivity of cotton samples

Figure 5 shows the variation in electrical conductivity values with the different treatment sequence, the different sequence of treatment leads to the variation in the weight percent of CuO deposited on fabric. It was observed that conductivity of PANI is 1.9 X 10^{-4}, as this conductivity value was increased, in case of incorporation of CuO in the matrix of PAN just when using certain sequence of treatment. The incorporation **was found to be maximum in case of pre treatment of cotton fabric by CuO then coated by aniline (CuO weight percent1.69)** as seen from EDX results This increase in electrical conductivity can be explain as the greater part of the CuO was impregnated in the polymer and in all probability no or small CuO stayed outside of the polymer matrix[28]. However, for the higher loadings of CuO (17 weight percent), in case of pretreatment of cotton fabric with aniline then coated with CuO nanoparticles, great amount of CuO was not contributed well to the PANs matrix, leading to a decrease in electrical conductivity . The decrease of conductivity also observed in lower loading (0.5weight percent) in case of treatment of cotton fabric with aniline CuO composite .According to all these experimental results, we can conclude that treated cotton fabric with higher electrical conductivity can only obtain using PAN matrix incorporated with an appropriate amount of CuO (1.69 weight percent)

6.Evaluation of Antibacterial activities

The antibacterial efficiency of the coated cotton fabric with various treatments was examined against representative microorganisms of open interest (E. coli, S. aureus, and C. albicans), which are extensively applied as biological indicators for pollution. Results of antibacterial activity are summarized in Table 1 and reveal that:

1. polyaniline deposited cotton fabric showed the smallest inhibition zone compared with the other different treatment. Antibacterial efficacy of The PANI can be explained in light of protonation of nitrogen-containing groups of polymer[29,30] and the physical interaction between the microbial cell and the polymer as a direct consequence of the structure of the polymer chain.
2. Consequences of table 1 give prove that the synergistic effect of cotton fabric coated with CuO-aniline nanocomposite .Antibacterial effect was seen for both components may be clarified by contribution of different type of action of PANI and CuNPs components: (1) physical interaction and electrostatic contact play an important rule in evaluating antibacterial efficacy of the nanocomposite; (2) PANI enhance steric stabilization and diminished accumulation capability of CuONPs, which thus expands the effective concentration of CuONPs suited for interacting with the cellular surface; (3) PANI network expands the interface area between the nanocomposite and micoorganism, permitting CuONPs to interact with the surface groups of cell walls; (4) the slow oxidation of CuNPs resulting into liberty of Cu^{2+} ions from the CuO layer and their partially reduction to the Cu^{+} ions, which are energetically facilitate its penetration through a lipid bilayer and taken up by the cell, causing its destruction[31]

3. Antibacterial efficiency for CuO nanoparticles pretreated cotton fabric coated with polyaniline was smaller compared to Polyaniline pretreated cotton fabric coated with CuO nanoparticles which could be attributed to the controlled release of Cu ions from the PANI matrix. Different values of clear inhibition zones could be associated with the differences in cell wall structures.

Table 1: Antibacterial activity of treated cotton fabric

Treatment of cotton fabric	Inhibition zone (mm/1cm sample)		
	Escherichia coli (G-)	Staphylococcus aureus (G+)	Candida albicans (Fungus)
Polyaniline deposited on cotton fabric	**11**	**12**	**11**
cotton fabric coated with CuO-aniline nanocomposite	**20**	**21**	**18**
CuO nanoparticles pretreated cotton fabric coated with polyaniline	**15**	**15**	**13**
Polyaniline pretreated cotton fabric coated with CuO nanoparticles	**18**	**18**	**16**

Conclusion

Novel electronic fabric prepared by using PANI and CuO nanoparticles.the prepared cotton fabric were obtained according to different types of treatment 1)pretreatment of cotton by CuO nanoparticles followed by polymerization Of aniline monomer in presence of CuO treated cotton fabric ,2)polymerization aniline monomer on cotton fabric then treated of PANI cotton fabric by CuO nanoparticles The third treatment occurs when cotton samples put in solution of PANI-CuO nanocomposite.The morphological studies (SEM) show different morphology for coated surface for the treated cotton fabrics. It was observed that high surface area of CuO nanoparticels allow uniform distribution of Polyaniline on the surface of cotton samples in case of CuO pretreated cotton samples and cotton samples treated with PANI/ CuO nanocomposite .The conductivity studies prove that that treated cotton fabric with higher electrical conductivity can only obtain using PAN matrix incorporated with an appropriate amount of CuO and this is the issue in case of **cotton fabric pre treated with CuO nanoparticles then coated by PANI. The antibacterial studies show that all treated samples have** good antibacterial activities .the cotton fabric treated with PANI-CuO nanocomposite displayed superior antibacterial activity

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