

# **Fabrication of Antimicrobial Textiles Using Hydrothermally Synthesized Copper Oxide Nanoparticles**

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**ABSTRACT:** Antimicrobial finishing of textiles has become extremely important in the production of protective and technical textile products like medical devices, health care, hygienic application, water purification systems hospital, textiles food packaging and storage. The antimicrobial property is imparted onto the fabric using chemical. The synthesis and characterization of copper oxide nanoparticles and their application on polycotton fabric have been studied for the protection against bacteria and fungi. Metal oxide nanomaterials are currently the most promising tools applied as antimicrobial agents. The copper oxide nanoparticles were prepared by hydrothermal method. The formation of copper oxide nanoparticles were confirmed by Dynamic light scattering, Scanning electron microscope, X-ray diffraction, Diffused reflectance spectroscopy and Fourier transform infrared spectroscopy. The antimicrobial activity of the treated fabric was assessed by Disc diffusion method SN 195920 and Agar diffusion method AATCC 30. It is observed that the different concentration of copper oxide treated fabrics possess a very good resistance for microbes.

**KEYWORDS:** Antimicrobial fabrics, Copper oxide, Nanoparticles, Hydrothermal method

## **I. INTRODUCTION**

Nanomaterials exhibit peculiar properties making them excellent candidates for various applications. Also due to the large surface area nanomaterials display a significant increase in the efficiency. In Textile industry the fabric finishing has taken new routes and demonstrated a great potential for significant improvements by applications of nanotechnology in UV Protection finishes, Water and Oil repellence, Thermal Comfort finishes, Flame Retardant finishes, Antimicrobial finishes, Electrical Conductibility finishes, etc., The developments in the areas of surface engineering and fabric finishing have been highlighted in several papers[1].

Focus on microbicidal properties of nanomaterials have increased because microorganisms are developing more resistance towards existing antibiotics.[2] Silver nanoparticles are known to possess high antimicrobial activity but they are costly, so cost effective materials with equivalent activity have to be identified. As copper and silver fall under the same group in the periodic table of elements, copper will exhibit similar properties as silver and also cheaper than silver. So copper can be an excellent candidate for production of antimicrobial textiles.

Metals have very low stability in harsh environments. When the clothes are washed the metal may easily get oxidized by the chemicals present in the washing agents. Metal oxides possess more stability over the metals at the same time metal oxides retain the catalytic properties of their respective metals. So instead of using copper, copper oxides nanoparticles can be used for the fabrication.

# International Journal of Innovative Research in Science, Engineering and Technology

(A High Impact Factor, Monthly Peer Reviewed Journal)

Vol. 5, Issue 2, February 2016

## II. RELATED WORK

Inorganic nano-structured materials with good antimicrobial activity have created a new approach for the researchers to produce antimicrobial textiles. Fabric coated with copper oxide nanoparticles showed a significant resistance towards microorganisms.[3] Copper oxide has unique optical, catalytic and chemical properties at nano scale. The synthesis of copper oxide and iron oxide by chemical reduction method was imparted on cotton fabric and tested for its antimicrobial properties. There was no growth of bacteria and fungi found on the treated fabrics.[4] Increase in concentration of the copper oxide nanomaterials increases the percentage reduction of bacteria. The range of percentage reduction for gram positive bacteria is 70-99% while for gram negative bacteria is 60-94% on treated cotton fabric.[5]

## III. SCOPE OF RESEARCH

- Synthesis copper oxide nanoparticles using hydrothermal method
- Morphological, structural, optical and chemical characterization of the synthesized nanoparticles
- Optimization of copper oxide nanoparticles concentration for antimicrobial finish on fabrics
- Validation of antimicrobial characteristics of the treated polycotton fabrics by international standard test methods.

## IV. MATERIALS AND METHODS

### Synthesis of copper oxide nanoparticles:

0.1 molar copper nitrate solution was prepared in double distilled water and 1% CTAB is added as surfactant. Equimolar concentration of sodium hydroxide was added dropwise and stirred for 30 minutes. Then the solution was transferred to a teflon lined autoclave and heated for 12 hours at 180°C.

### Characterization of nanoparticles:

Prepared copper oxide nanoparticles were characterized using various characterization techniques. The preliminary particle size characterizations were done using Horiba SZ100 Particle Size Analyser and the formation of the crystalline materials were confirmed using Panalytical Xpert Pro X-Ray Diffractometer with Cu K $\alpha$  radiation of wavelength 1.54 Å. Diffused Reflectance Spectrum of the copper oxide nanoparticles were also taken using Analytik Jena Specord 210 Plus UV-Visible Spectrometer to confirm the formation of CuO nanoparticles. Bruker AlphaT Fourier Transform Infra-Red Spectrometer was used to confirm the presence of copper oxide nanoparticles in treated poly cotton fabric. Then the morphology of the nanoparticles was analysed using Carl Zeiss Sigma Scanning Electron Microscope.

### Treatment of nanoparticles on fabric:

Four different concentrations (0.5%, 1%, 1.5%, and 2%) of prepared copper oxide nanoparticles were dispersed in 0.5% of acrylic binder solution along with 0.5% Lissapol-N. The fabric was dried at 100°C for 4 mins to remove the moisture content. The dried fabric (10cm×10cm) samples were soaked in different concentrations of prepared nanoparticles dispersion for 30 minutes then the samples were squeezed to remove excess solution using padding mangle running at the speed of 20 rpm and with Pressure of 1.0 kg/cm<sup>2</sup>. Then the samples were air dried and cured for 4 minutes at 120°C.

### Antimicrobial Efficiency of Finished Fabric:

**Disc Diffusion Method (SN 195 920):** [6] Bacteriostasis agar was dispensed in sterile Petridishes. 24 hours broth cultures of the test organisms (*Escherichia coli* and *Staphylococcus aureus*) were swabbed using sterile cotton over the surface of the agar plates. Using sterile forceps the finished samples were placed over the surface of the solid agar. The plates were incubated at 37°C for 18-24 hrs. After incubation, the plates were evaluated on the basis of absence or presence of an effect of bacteria in the contact zone, under the specimen and the possible formation of a zone of inhibition around the test specimen and the zone of bacteriostasis were measured in millimetres.

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**Agar diffusion method (AATCC 30):**[7]The antifungal properties of finished samples with copper oxide nanoparticles were investigated against pathogen (*Aspergillusniger*and *Candida albicans*). The sterilized potato dextrose agar medium was prepared and dispensed in petridish and the spores of the fungi were inoculated into  $50\pm 2$  ml of sterile distilled water containing few glass beads and shaken vigorously to bring the spores into suspension. About  $1.0\pm 0.1$  ml of inoculums was distributed evenly over the surface of the agar. The test samples were placed in contact with hardened agar medium over which  $0.2\pm 0.001$  ml of the inoculums was evenly distributed by means of a sterile pipette. The plates were incubated at  $27^{\circ}\text{C}$  for 5 days. At the end of the incubation the growth of inoculums was reported by measuring the zone of mycostasis underneath and alongside of the fabric.

## V. EXPERIMENTAL RESULTS

### X-ray diffraction (XRD):

Figure 1 represents the X-Ray diffraction pattern of synthesized copper oxide nanoparticles. The pattern of the synthesized copper oxide nanoparticles matches with the XRD pattern[8] of the copper oxide in JCPDS (Reference number - PDF800075). The crystal structures of copper oxide nanoparticles were found to be hexagonal in shape.

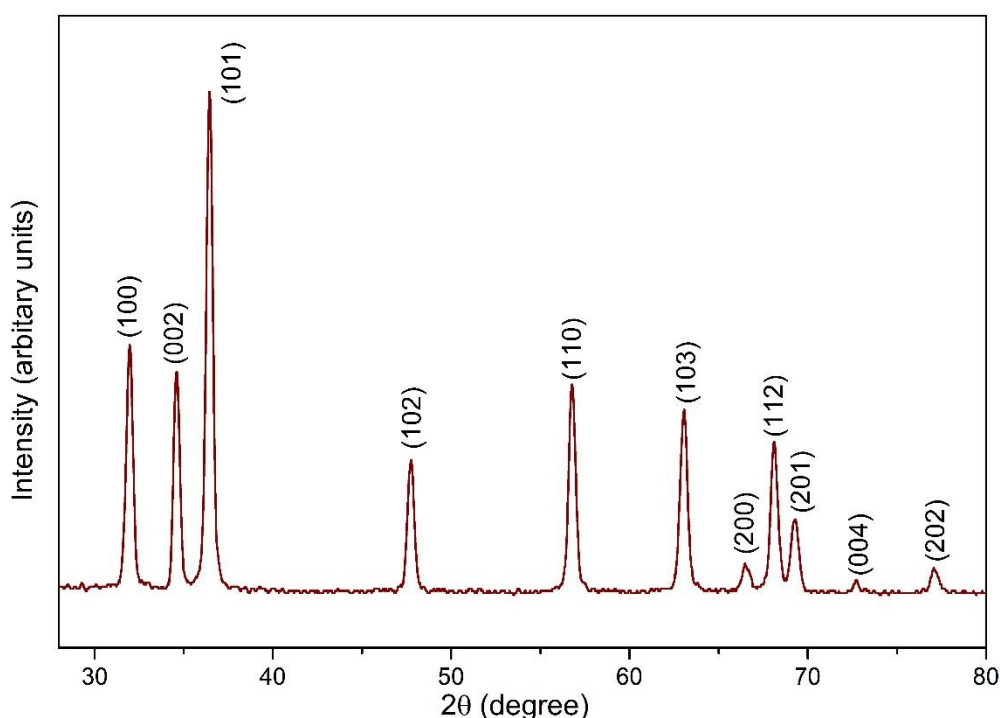


Fig.1. X-Ray Diffraction spectrum of CuO Nanoparticles

### Particle size analysis (PSA):

The particle size analysis of the copper oxide nano particles was given in figure 2. The average particle size was found to be 92 nm.

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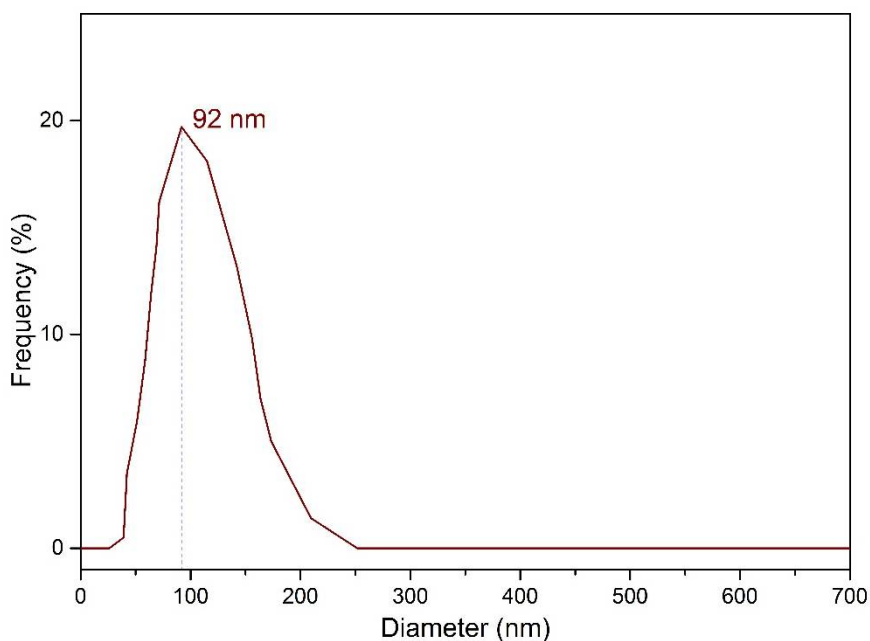


Fig.2. Particle size analysis of CuO Nanoparticles

## Diffused Reflectance Spectroscopy:

Figure 3 represents the diffused reflectance spectrum of the prepared copper oxide nanoparticles. The reflectance of the sample started to reduce at 989 nm by which the band gap of the material was calculated to be 1.24 eV using the relation  $E = hv$ . The observed band gap matches the band gap of the copper oxide.

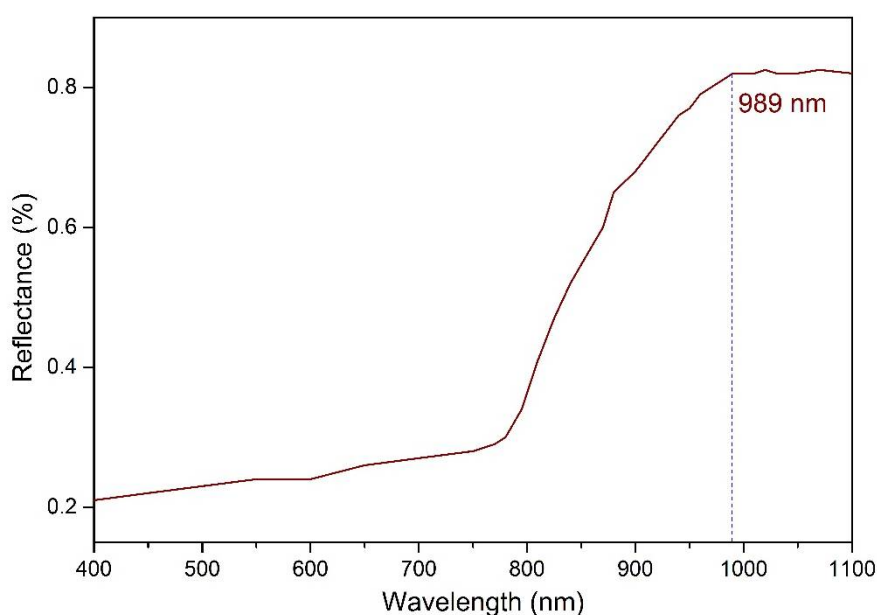


Fig.3. Diffused reflectance spectrum of CuO Nanoparticles

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### Scanning Electron Microscopy (SEM)

Figure 4 represents the scanning electron microscope image of the copper oxide nanoparticles and the size of the nanoparticles was found to be in the range of 60 nm.

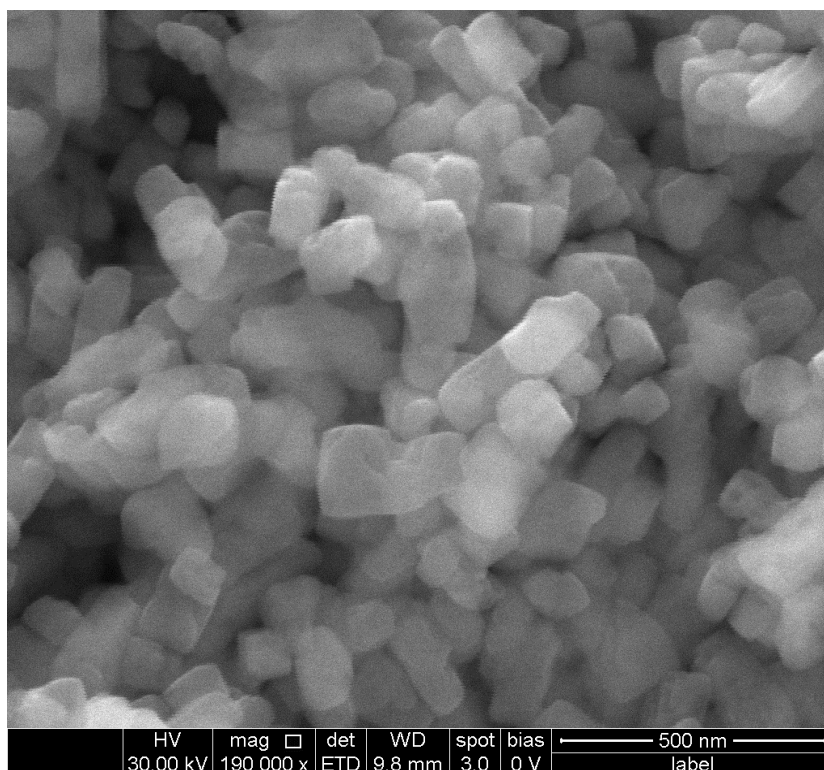


Fig.4. Scanning Electron Microscope image of CuO Nanoparticles

### Fourier Transform Infra-Red Spectroscopy (FTIR)

FTIR spectrum of the copper oxide, polycotton and copper oxide nanoparticles treated polycotton are given in the figure 5. For the spectrum of the untreated fabric, the peak at  $710\text{ cm}^{-1}$ ,  $1010\text{ cm}^{-1}$ ,  $1237\text{ cm}^{-1}$ ,  $1710\text{ cm}^{-1}$ ,  $2870\text{ cm}^{-1}$ ,  $3280\text{ cm}^{-1}$  were attributed to C-H, C-O, C-N, C=O, C-H and adsorbed hydroxyl groups respectively. In the CuO nanoparticles spectrum, three peaks  $422\text{ cm}^{-1}$ ,  $456\text{ cm}^{-1}$  and  $530\text{ cm}^{-1}$  were attributed to the metal oxide bonding Cu-O. The remaining peaks at  $1112\text{ cm}^{-1}$  (C-N),  $1402\text{ cm}^{-1}$  (C-C) and  $1627\text{ cm}^{-1}$  (N-H) were due to the presence of residual surfactant used for the synthesis of the synthesis of copper oxide nanoparticles. The spectrum of the copper oxide nanoparticles coated polycotton contains the peak stretches found in the polycotton as well as the copper oxide spectrum, thus confirming the presence of copper oxide nanoparticles in the fabric.

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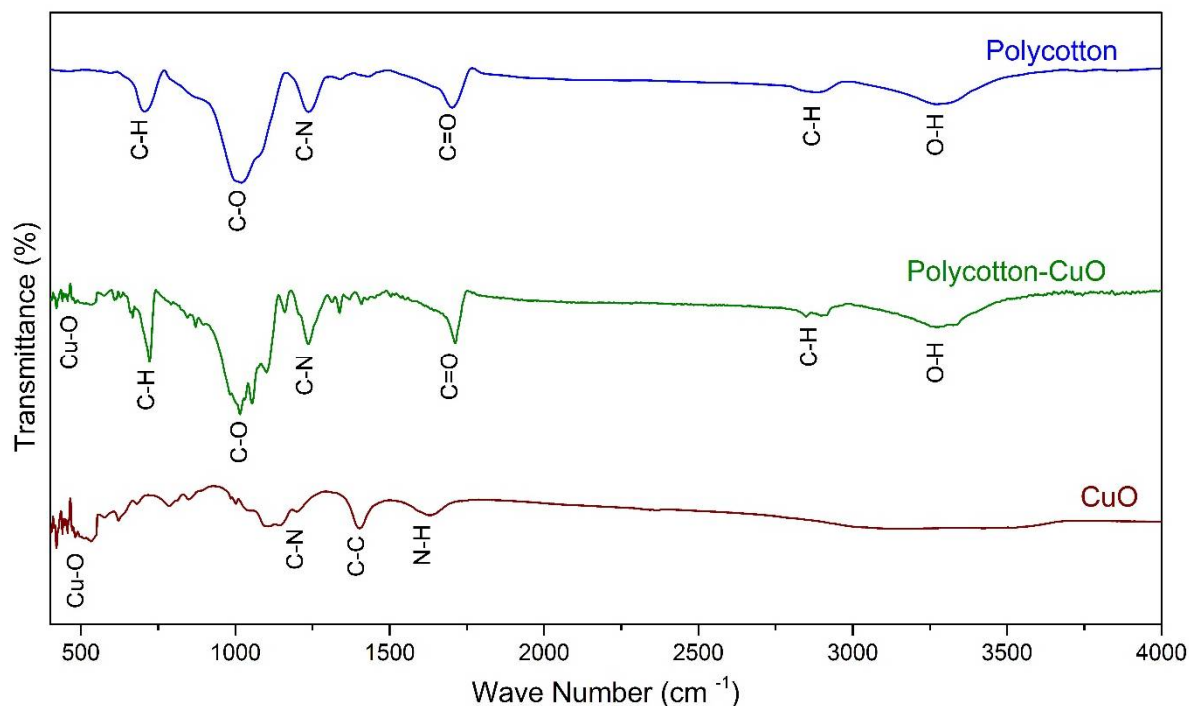


Fig.5. FTIR spectrum of Polycotton, CuO nanoparticle treated polycotton and CuO nanoparticles,

## Antimicrobial Efficiency of Finished Fabric

The antimicrobial activity of CuO nanoparticle treated polycotton fabric is summarized in figure 6.

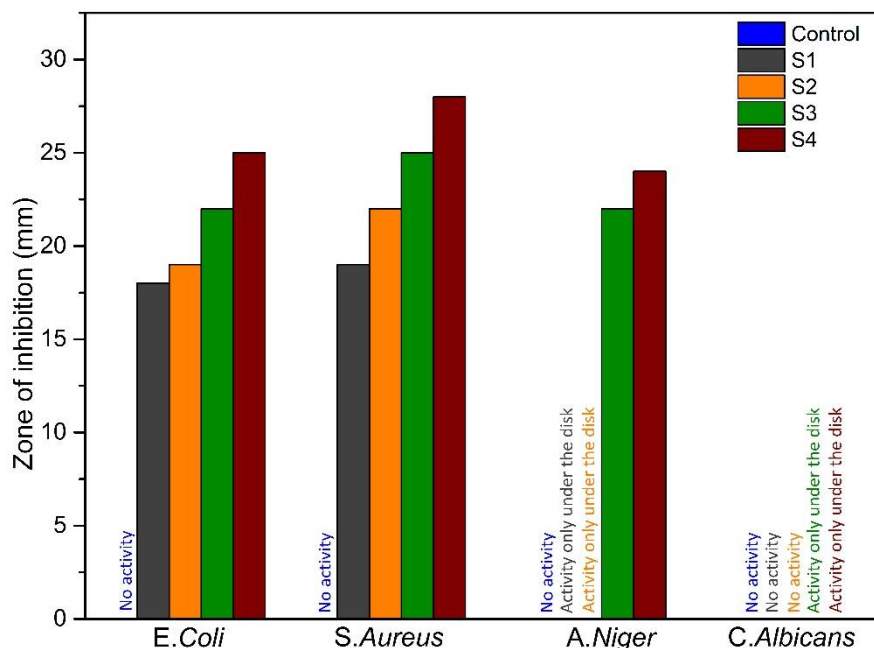


Fig.6. Antimicrobial activity of CuO nanoparticle treated fabric on various species (Concentration of CuO: Control- 0%, S1-0.5%, S2-1%, S3-1.5%, S4-2%)



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## Disc Diffusion Method (SN 195 920)

The antibacterial activity of CuO nanoparticles treated polycotton fabric was assessed qualitatively by disc diffusion method in order to investigate the efficacy under different concentration levels such as 0.5%, 1%, 1.5%, and 2%. Figure 7 presents the comparison of antibacterial activity of treated and untreated samples against gram positive (*Staphylococcus aureus*) and gram negative (*Escherichia coli*) bacteria. The maximum inhibitory effect was observed against *S.aureus* in S4 with a zone of inhibition of 28mm followed by *E.coli* with a zone of inhibition of 25mm in S4 whereas all the other samples exhibited less zone of inhibition.

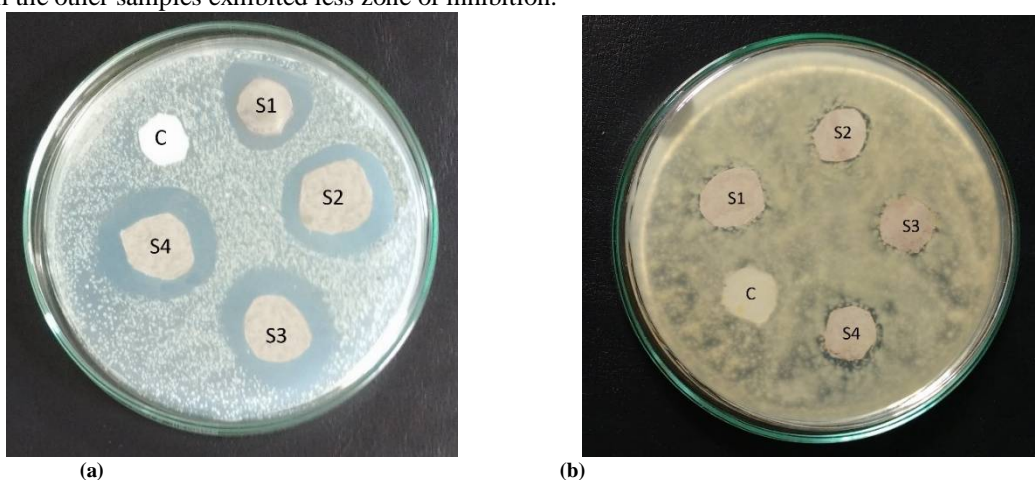


Fig.7. Antimicrobial activity of CuO Nanoparticle treated polycotton fabric against (a) *E.Coli* (b) *S.Aureus*  
(Concentration of CuO: Control- 0%, S1-0.5%, S2-1%, S3-1.5%, S4-2%)

## Agar diffusion method (AATCC 30)

The treated fabrics were assessed for their antifungal activity against *Aspergillus niger* and *Candida albicans* by agar diffusion method. In figure 8 the zone of inhibition around the fabrics represents the antifungal activity. The treated sample S4 exhibited a higher antifungal activity of 24 mm against *A.niger*. While the sample S4 shows no growth beneath the disc against *C.albicans*. Some of the other samples showed less activity while some did not show any antifungal activity.

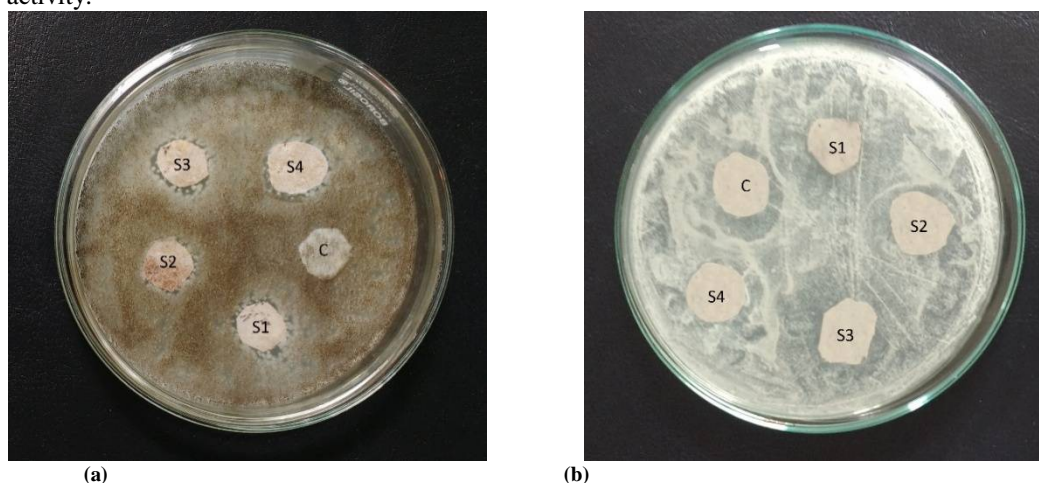


Fig.8. Antimicrobial activity of CuO Nanoparticle treated polycotton fabric against (a) *A.Niger* (b) *C.Albicans*  
(Concentration of CuO: Control- 0%, S1-0.5%, S2-1%, S3-1.5%, S4-2%)

# International Journal of Innovative Research in Science, Engineering and Technology

*(A High Impact Factor, Monthly Peer Reviewed Journal)*

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## VI.CONCLUSION

Copper oxide nanoparticles were synthesized and characterized for its properties using various techniques. Polycotton fabric was treated with the CuO nanoparticles and tested for antimicrobial activity against four microbial species. CuO nanoparticles exhibited good activity proving them as a potential candidate for antimicrobial textiles.

## VII.ACKNOWLEDGEMENT

Acknowledge Department of Nanoscience and technology, Sri Ramakrishna Engineering College for providing the lab facilities.

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