

**Research Article****Evaluation of crosslinking type and antibacterial activities of copper oxide loaded cotton textile fabrics****Aslıhan Koruyucu** ^{a,*}^a Namık Kemal University, Çorlu Faculty of Engineering, Department of Textile Engineering, 59860, Çorlu/TEKIRDAG, TURKEY

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ABSTRACT

In the current study, the efficacy of Cu(I)O micro and two different cross linkers as a antibacterial metal oxides on textile substrates versus *Staphylococcus aureus* and *Escherichia coli* was examined. In particular, bacterial inhibition relationship of micro Cu (I) O with two different cross linkers needed to exhibit effective bacterial action was studied. On textile substrates such as 100% cotton, there was 89,44-94,42% rebate in bacterial counts. Effectiveness of the antibacterial action was retained after ten and twenty cycles.

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1. Introduction

Cotton fabrics provide an excellent environment for microorganism proliferation because of high moisture sorption and a large surface area. Some researchers believe that in hospitals, contaminated textiles might be an important source of microbes contributing to the transmission of nosocomial-related pathogens [1,2]. There are many ways to control microbial growth on textiles, such as incorporating antibacterial agents into fibers by coating. The most important antibacterial substances used in textile finishing are quaternary ammonium salts, chloro-ether phenols, poly (hexamethylene biguamidyene), silver and its compounds, organic-silicones [3-5].

Nanoparticles are clusters of atoms in the size range 1-100 nm.

With current advances in nanotechnology, different types of metal and metal oxide nanoparticles were applied in many researches for achieving antimicrobial activity [1-9]. Currently, some noble metal NPs have been largely examined and are well known for their antibacterial effects [8].

Copper and silver are very common in use for their antimicrobial properties [10]. Example of, the Cu NPs

indicated higher antibacterial effect relative to the silver NPs against *E.coli* [19, 20]. The production of thin coated films surfaces are based on coating solid surfaces with a thin film of metal such as copper, silver or titanium, utilized by different techniques [11]. Thin coated films surfaces are in contact with human skin be accomplished by using solid copper or copper alloy equipment.

In this text, copper (Cu) and nickel (Ni) are good option materials because they are more economical than gold and silver. It has already been announced that Cu NPs [10-13] and copper oxide NPs [14-18] have antimicrobial activity.

The purpose of this study was to evaluate micro Cu (I) O and two different cross linkers as an antibacterial agent on cotton fabrics versus *Staphylococcus aureus* and *Escherichia coli*. The durability of the antibacterial activity of the treated substrates versus laundering twenty times was also examined.

In this study, FT-IR spectroscopy and SEM were used to describe CuO NPs. The antibacterial activity of these NPs on two pathogenic species with antibacterial resistance characteristic, *Escherichia coli* ATCC 25922 and *Staphylococcus aureus* ATCC 43300, was assessed.

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2. Material and Method

2.1 Materials

2.1.1 Chemicals

Copper(I)oxide (Cu_2O), cross-linked(blocked isocyanate, glycid methacrylate)

Bleached cotton fabrics, polyurethane binder and polysiloxane additive.

2.2 Methods

2.2.1 Copper oxides particles applied to cotton fabrics

Experiments were applied samples with size of 40cmx30cm. Cotton fabrics were coated with copper micro and nanoparticles coating bath at concentrations of 0,5% and 1%. Samples were dried at 100 °C for 2 min. Curing was done at 150 °C for 2 min.

The antibacterial activities of the untreated and treated fabrics with coating bath which including copper oxide particles were evaluated after being displayed to recast washing periods (ten and twenty washing periods). Rinsing was performed with a machine set for warm water containing, 2% sodium carbonate and soap. After each rinsing (45 min), the fabrics were plummet dried in a dryer at 70°C.

2.3. Characterization techniques

Constructural and optical properties of the CuO micro and nanoparticles 1 were established by using SEM (Scanning Electron Microscopy) FTIR (Fourier Transform Infra-Red Spectroscopy) in the wavelength distance of 400-4000 cm^{-1} .

2.3.1 Antibacterial activity

Bacterial strains were acquired from the Uludag University Medical Faculty, Department of Microbiology and Infectious Diseases. The culture media; Agar-agar Type-I and chemicals; were used for the surge of bacteria.

The antibacterial behavior of the coated fabrics was tested for two bacterial strains; Gram-negative *Escherichia coli* and Gram-positive *Staphylococcus aureus*.

Roughly, 25 ml of liquefied and frigid nutrient agar media was flowed in the sterilized petri dishes. The plates were left over night at room temperature to check for any pollution one. The bacterial test organism *S.aureus* and *E.coli* were grown in nutrient chowder for 24 hours at 37 °C. A 100 μl nutrient chowder culture of each bacterial organism was used to got ready bacterial lawns. Agar wells were got ready with the aid of a sterilized stainless steel cork borer.

In order to study the antimicrobial activity of the fabrics; the samples of 1 cm^2 fabrics were taken randomly and placed in a sterile flask. Each square fabric was placed in a sterile flask. Tryptone soy chowder (2.2 ml) was then added to each vial to obtain a total volume of 3 ml. An aliquot (10 μl) of *S.aureus* suspension was added to each flask (1.6x10³/ml). Control chowders with and without bacterial vaccination were also incorporated. The vials were then incubated with agitation at 35°C. Aliquots of 10 μl chowder were sampled at 24 h and serial dilutions for the aliquots were got ready in chowder. The bacterial activity was assessed after 24 h and appraised % rebate of the bacteria using the following equation:

$$R (\%) = [(A-B)/A] \times 100 \quad (1)$$

where R is the rebate rate, A is the number of bacterial colonies from untreated fabrics, and B is the number of bacterial colonies (Duran, Marcato, De Souza, Alves&Esposito,2007).

3.Results and Discussion

3.1 FTIR Spectroscopy

FT-IR analysis was used to examine the presence of the chemical bonds in the coating path structure in the utilized cotton. The FT-IR spectra of the cotton fabrics pretreated in the formulations were given in blue color. The characteristic peaks of the cotton fabrics pretreated in the spectra are summarized in Table 1. In the FTIR spectrum (Figure 1) the peak appearing in the range 1732-1750 cm^{-1} is due to C=O groups in the ester groups. Bands observed at 1374 and 1383 cm^{-1} are characteristic of -CH-groups. In addition, the spectrum shows at 1083-1088 cm^{-1} to C-O groups. The tensile vibrations of the -OH groups of the cotton fiber structure give wide and severe bands at 3325 cm^{-1} . Glycid methacrylate cross-linker with antibacterial coating path when the FTIR spectrum of the coated cotton fabric is examined; aliphatic esters in the structure of glycid methacrylate carbonyl groups in the isocyanate structure at 1740 cm^{-1} appears to give a sharper peak. This gives us the antibacterial Cu (I) O chemical shows better binding of cotton fiber together with the coating path.

Table1. FTIR spectrum information of pretreated cotton fabric

Pre-treated cotton fabric	3424 cm^{-1} -OH tension peak 2900 cm^{-1} C-H tension peak 1432 cm^{-1} -C-C strength peak (ringed structure) 1350-1370 C-H peak (1373-1338-1318 cm^{-1}) 1000-1300 cm^{-1} -C-O strength peak (1165- 1115-1058 cm^{-1})
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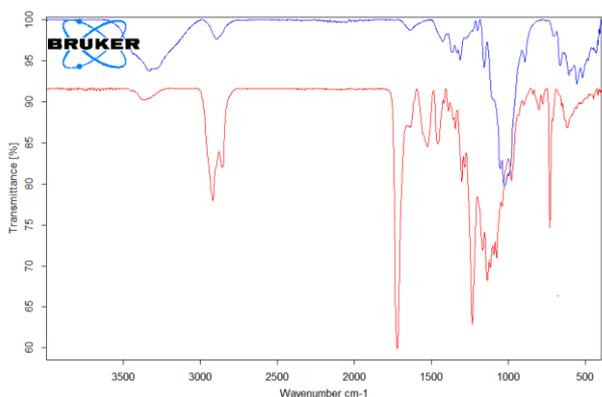


Figure 1. FTIR spectra of Cu (I)O micro particles

3.2. Morphology of the coated cotton fabrics

The cotton fabrics were coated copper oxide coating bath. Samples were dried at 100°C for 2 min tracked by curing for 2 min. at 150°C.

The SEM images of cotton fabrics before (untreated) and after (treated) coated with micro Cu (I)O bath are shown in figures 2a-2b.

The SEM image in Figure 2a illustrates the flat construction of the cotton fabrics before coating with Cu (I)O micro particles. After coating, the homogeneous deposition of Cu(I)O micro particles on the cotton fabrics was shown in Figure 2b. Figure 2b indicate the appearance of small particles, most probably Cu(I)O micro particles on the cotton fabric surface.

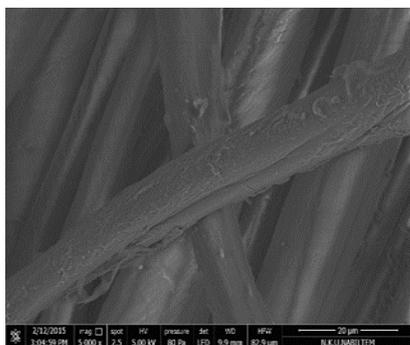


Figure 2a. SEM images of untreated cotton fabric.

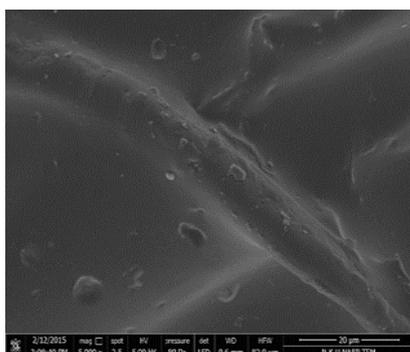


Figure 2b. SEM images of Cu (I)O <5µm particles on cotton by using concentrations of 1%.

3.3. Efficiency and durability of the micro copper oxide particles with two different cross linkers-based antibacterial coating

Table 2. The effect of repeated washing periods on antibacterial coating bath with iso-cyanate cross linker

Number of washing	Bacterial reduction(%)	
	S.aureus	E.coli
Before washing	100%	97,98%
After 10 periods	97,62%	93,94%
After 20 periods	94,42%	89,44%

Table 3. The effect of repeated washing periods on antibacterial coating bath with glycid methacrylate crosslinker

Number of washing	Bacterial reduction(%)	
	S.aureus	E.coli
Before washing	98,81%	-203,03%
After 10 periods	95,24%	-203,03%
After 20 periods	92%	-203,03%

Results of Table 2 was so clear that 1% of copper oxide particles are enough to cause antibacterial properties to cotton fabric. However, approximately 94% and 89% of the communicated antibacterial properties with the isocyanate cross-linker against *S. aureus* and *E.coli* bacteria are lost under the effect on twenty washing periods, respectively.

It is apparent to the Table 3 that, in spite of the concentration of Cu(I)O <5µm particles used for treatment, the bacterial colonies decrease was always higher than 90% against *S.aureus* and the increase of bacterial colonies was 200% versus *E.coli* for Cu(I)O treated samples with washing periods.

The rebate of bacteria was noted after ten washing periods for cotton fabrics coated by knife-over coating method. Whereas the samples treated by coating method showed the bacterial colonies to values slightly higher than 90%. The treated cotton fabrics were washed for ten and twenty periods. The antibacterial efficacies were tested. The marginal rebate was obtained in antibacterial properties. Coating bath formulation containing 86,5% of the binder is given in the Table 4.

Table 4. Coating bath formulation containing 86,5% of the binder.

Cu(I)O microparticles	1%
Binder	86,5%
Coating thickness	0,1mm
Drying	100°C/2 min
Curing	150°C/2 min

It is observed that inclusion of copper oxide concentration in coating bath formulation increase antibacterial properties of cotton fabric even after twenty washing periods. Fabrics coated with micro copper oxide particles at concentration of 1% in the existence of binder demonstrate bacterial rebate values of 94% and 89% for *S.aureus* and *E.coli*, respectively. Cotton fabrics having excellent antibacterial properties and with washing resistant could be acquired by treating the fabrics with a bath of copper(I)oxide having particle size of 5 μ m in existence of a binder as defined in this work.

4. Conclusion

There is a growing body of scientific evidence with proves the antibacterial properties of Cu(I)O microparticles against various bacterial species, including *E.Coli* and *S.aureus* bacteria. Based on the literature, in some cases CuNPs denoted higher antibacterial effect relative to the silver NPs.

The disk diffusion test method of cotton fabrics described provides good antibacterial properties, as well as resistance to washing, at a concentration of Cu(I)O microparticle of about 1% (w/w).

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