

## Manufacture of Coated Cotton Fabric Anti-bacterial Nano Thin Film by Sequential Immersion – Sol- Gel Technique

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### Abstract

Antibacterial fabrics were prepared by using cotton fibers,  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ,  $\text{ZnSO}_4$  and  $\text{TiO}_2$ . These cotton fibers were coated by nano thin film consists of titanium oxide, copper oxide and zinc oxide. The study of structural properties by XRD had indicated that the coated fabric thin film was polycrystalline. The topography of the thin films surface was identified by using a scanning electronic microscope (SEM) and optical microscope (OM). Optical properties were measured using optical spectrometer, the optical properties improve lead to increase in the effectiveness of anti-bacterial fabric during add titanium dioxide and zinc oxide to coated fabric by copper oxide, as well as improve the biological activity of fabric the thin film using the substance added (Methanol Sulphate) to a chemical solution during the initial preparation process of the coating fabric by oxide copper thin film. Biological characteristics have been studied for fabric against bacteria types *Escherichia coli*, *Staphylococcus aureus* and *Bacillus cereus*.

**Keywords:** Antibacterial Fabric; SEM and Thin Film Nanostructure.

تصنيع نسيج قطني مطلي بغشاء نانوي مضاد للبكتيريا بتقنية الغطس المتعاقب -

### المحلول الجيلاتيني

### الخلاصة

حضرت الأنسجة المضادة للبكتيريا باستخدام ألياف القطن و  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ،  $\text{ZnSO}_4$  و  $\text{TiO}_2$ . وقد تم طلاء هذه ألياف القطن بغشاء نانوي يتكون من أكسيد التيتانيوم وأكسيد النحاس وأكسيد الزنك. دراسة الخصائص التركيبية بواسطة XRD وأشارت إلى أن غشاء كان متبلور. تم التعرف على تضاريس سطح الأغشية الرقيقة باستخدام مجهر الماسح الإلكتروني (SEM) والمجهر الضوئي (OM). تم قياس الخصائص البصرية باستخدام المطياف، تحسين الخصائص البصرية يؤدي إلى زيادة فعالية النسيج المضاد للبكتيريا من خلال إضافة ثاني أكسيد التيتانيوم وأكسيد الزنك للنسيج المطلي بغشاء أو أكسيد النحاس، فضلا عن تحسين النشاط البيولوجي للنسيج باستخدام مادة المضافة (الميثانول سلفايت) إلى المحلول الكيميائي أثناء عملية التحضير الأولية للنسيج أثناء طلائه بغشاء أكسيد

النحاس. وقد تم دراسة الخصائص البيولوجية للنسيج ضد أنواع البكتيريا الإشريكية القولونية، المكورات العنقودية الذهبية والعصوية الشمعية.

## INTRODUCTION

Recently attention attracted of researchers in field tissue coated process by the structure a nano thin films anti-bacterial positive gram and negative gram for application a wide range in the health, because of its effectively in the killing of the dangerous bacteria and prevent their reproduction and thus ban or limit the spread of infection with bacteria through contact or inhalation particularly in hospitals. Because the permanent impedance to human life, killing or controlling the growth of pathogenic micro-organisms such as bacteria, viruses and fungi on stationary surfaces stick to be a main worry around the world. Antimicrobial agents have been used for many years to conquer pathogenic organisms in a broad area of applications (in hospitals, the home and industrial premises). In order to get for uncontaminated and clean drinking water and healthy living. Because of the pervasion of contagious diseases that result from different pathogenic bacteria and fungi there is a increasing worry in healthcare and food manufacture to improve active antimicrobial agents to keep form the microbial growth. Until now, organic synthesis, major oils, bacteria-produced antibacterial proteins (bacteriocins), enzymes, fruit extracts, etc .[1].The approach of research on improves the process of developing the thin film preparation process using techniques that improve the effectiveness of anti-bacterial fabric. Titanium dioxide  $TiO_2$  are non-toxic and non-corrosive nature.  $TiO_2$  are chemical stability and optical specialization, it's use for killing different groups of microorganisms including bacteria, fungi and viruses, it's use extensively, since it has high photo reactivity, wide -spectrum antibiosis and chemical stability [2-4]. Titanium oxide absorbance of UV rays about a wavelength of 388 nm or shorter enhance reactions; but, it is known that 254-nm rays having a larger energy level, therefor it interest for use in the killer germs lamps, UV rays absorbs by the DNA of living organisms. Titanium oxide photo catalyst does not need ultraviolet ray's high energy as 254 nm and cause danger to human life. It as well let reactions to beginning by the near-ultraviolet rays with relatively long wavelengths included in sunlight and released by fluorescent lamps. Note that mixture of a  $TiO_2$ , ZnO and CuO thin film, it makes the absorbance to a longer wavelength, i.e., to the visible region in comparison with pure  $TiO_2$ , because the change in electronic and optical properties of  $TiO_2$  [5]. ZnO is reordered as "usually known as safe" (GRAS) by the U.S. Food and Drug Administration (21CFR182.8991). In order to manufacture better use of ZnO nanoparticles in food products and to help in the evolution of sturdy, however nontoxic, it is requisite to known the mechanism of behavior of ZnO nanoparticles against bacteria, but so far, the operation implicit their antibacterial effect is not obviously. Except some studies which reached some results that nanoparticles have selective toxicity to bacteria but show less influence on human cells [6].ZnO nanoparticles have been shown to have a broad range of antibacterial effectiveness against both gram-positive and gram-negative bacteria, including main foodborne pathogens (a bacterium, virus, or other microorganism that can cause disease) like *Escherichia coli* O157:H7, and *Staphylococcus aureus* [7, 8].The characteristic of inorganic nano materials are of preventive and fixity, minial toxicity, and heat impedance. Except, the inorganic nanomaterials include mineral elements major to human existences, and show tight antibacterial and antifungal effectiveness to pathogenic microbes while applied in

slight concentrations. The large surface area of nanoparticles incloses accretion interaction with bio-organics current on the viable cell surface [9-11]. Yet, inorganic nanomaterials like gold [12], silver [13], ZnO[14], MgO, CaO[15], and TiO<sub>2</sub> [16] were found to show wonderful bacteriostatic, antimicrobial, or biocidal effectiveness. On from here the standpoint of ecological and health anxiety it is remarkable to discuss the antimicrobial. The main aim of present study manufacture and develop of anti-bacterial fabric of broad-spectrum coated nano thin film of different materials and multilayers and increasing its effectiveness vital simple method technology and inexpensive.

### Materials and Methods

Put 25% g of cotton in beaker contain a solution of sodium sulfate for 5 hour, then cotton rinse well by water distilled at a temperature of 50°C. Cotton fiber dried in vacuum oven at a temperature 60 °C for 90 minutes. Copper sulfate CuSO<sub>4</sub>·5H<sub>2</sub>O (0.6 and 0.4)M respectively dissolved in (100 ml) of distilled water in beaker with (Magnetic Stirrer) then add Na<sub>2</sub>SO<sub>3</sub> progressively to copper sulphate solution into vanish of blue color loses turn out, then add to the solution 5ml from (KOH).



**Figure (1) Preparation stages of cotton fiber coated by CuO thin film antibacterial**

The value of pH fixed about (6.4) added (Methanol Sulphate) to a chemical solution during the initial preparation process of the coating fabric by oxide copper. Cotton fibers overlying the first in a complex solution for even the color of the solution turns to brown then dive in beaker containing distilled water at 90 degree limited of 5 minutes and then put the cotton fiber in the complex solution again. Observe the color of cotton fiber surface turn into Light brown coated as figure 1 explain the preparation stages of cotton fiber coating by CuO thin film. Cotton fiber dried coated copper oxide thin film by vacuum oven at a temperature 70 °C for 2 hour. Then we prepare of sol-gel by using powder solution of TiO<sub>2</sub> and ZnSO<sub>4</sub>(0.4 and 0.2)M respectively where we dissolve it by adding ethanol with distilled water and placed in beaker with magnetic Stirrer and placed on the heater (hot plate and magnetic Stirrer) at a temperature 30°C for 20 minutes. Then add with butanol to sol-gel solvent in order to speed up process the solution and then cotton fiber coated CuO thin film dive

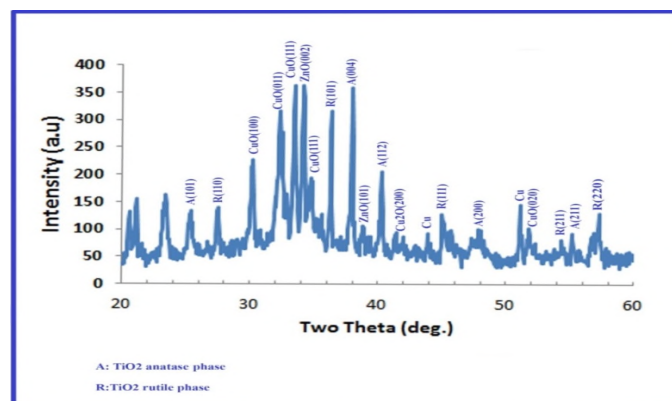
in the solution for one hour at a temperature of 80°C the second step immersion cotton fiber in beaker contains solution of (KOH) as show in figure 2. Then dried fibers in the vacuum oven from air.



Figure (2) Preparation stages of cotton fiber coated by CuO and (CuO-TiO<sub>2</sub> - ZnO) thin films antibacterial after dried.

### Results and Discussion

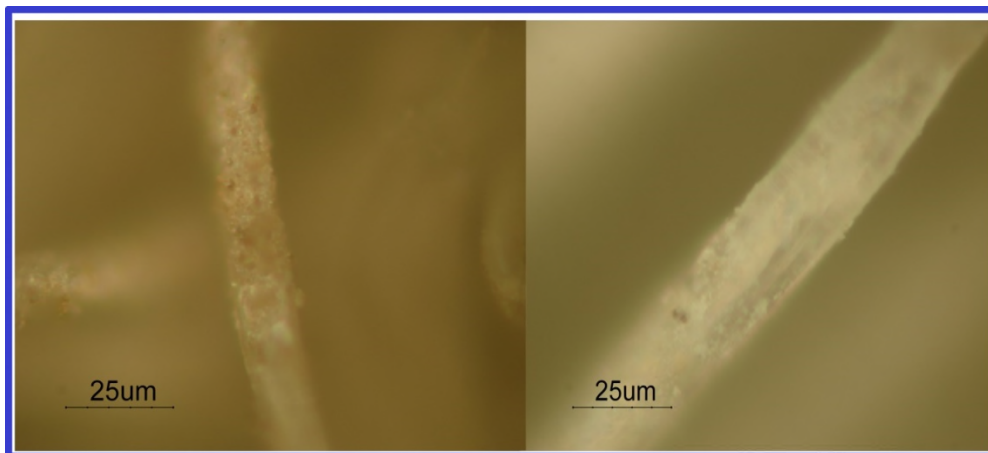
Through examine the X-ray diffraction, we can identify and understand the crystalline growth kind, of TiO<sub>2</sub> -CuO -ZnO thin films which coating of cotton fiber prepared by sequential immersion and sol-gel technique. The X-ray peaks of the films are in accordance with those of a typical anatase (101),(004),(112),(200) and (211) and rutile(110),(101),(111),(211) and(220) crystalline TiO<sub>2</sub> as show in figure 3. Note in the XRD pattern of sample appeared very weak diffraction peaks of [002] and [101] of ZnO. Because the lack of ZnO concentration. The XRD results show that CuO orientation (100),(011),(111),(111)<sup>-</sup>,(020) are dominantly crystalline and phase Cu<sub>2</sub>O (020). All other predominant peaks are attributed compared with standard [X-ray diffraction data file [N 1997 JCPDS prevalent].



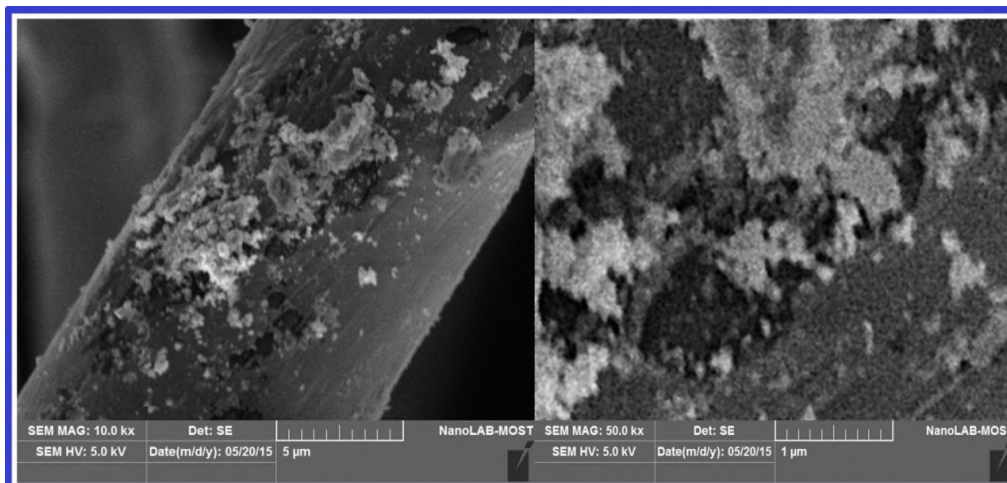
Figure(3) XRD patterns of CuO-TiO<sub>2</sub> - ZnO mixture.

**Optical examination microscopic (oem) and scanning electron micrographs (sem).**

Figure 4 Show fiber cotton coated by antibacterial CuO -TiO<sub>2</sub>-ZnO thin film, used the Optical examination microscopic, we observe uniformity and adhesion of thin film, the distribution of the thin film on the cotton fibers uniformly and homogeneous. Figures 5 display the SEM image of exceedingly high enlargement of coated and original, fabrics CuO -TiO<sub>2</sub>-ZnO which wrapped the fiber shows a nanoparticles and homogeneous.



**Figure (4) Optical Micrograph of of antibacterial CuO –TiO<sub>2</sub>–ZnO thin films coated cotton fabric.**

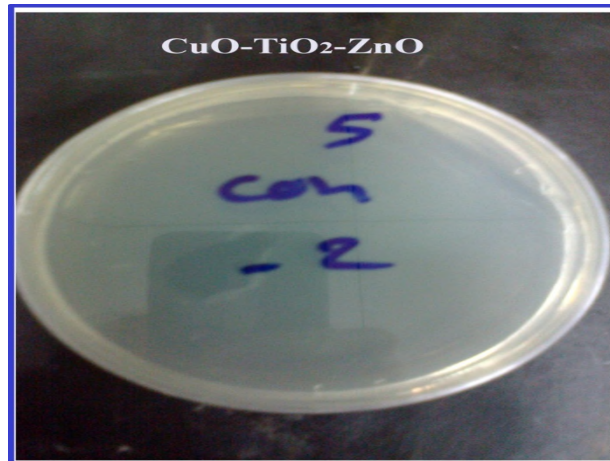


**Figure (5) SEM images of antibacterial CuO –TiO<sub>2</sub>–ZnO thin films coated cotton fabric.**



**Biological Laboratory Results**

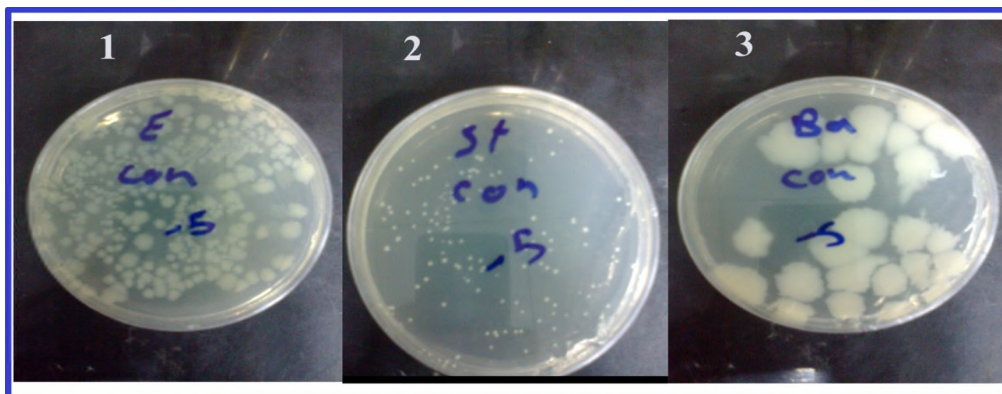
Laboratory testing results showed remarkable provides and success of the tests for three types of Escherichia coli, Staphylococcus aureus and Bacillus cereus. Placed Fabrics of coated thin film CuO -TiO<sub>2</sub>-ZnO in glass tubes test containing liquid media and incubated at a temperature 37 C° for 24 hours and then E.coli, S. aureus and Bacillus cereus planted on the arrangement amid the solid\_technique Publishing dishes, as shown in figure (6).



**Figure(6) Liquid media control of CuO -TiO<sub>2</sub>-ZnO.**

Figure 7 Control of bacteria without the presence of the sample taken from the fifth and mitigation as follows:

1. Bacterium Escherichia coli as a parameter control growth before  $20 \times 10^7$  cells / ml.
2. Bacteria Staphylococcus aurius as a parameter control growth before  $11.3 \times 10^7$  cells / ml.
- 3- Bacterium Bacillus cereus as a parameter control growth before  $2.5 \times 10^7$  cells / ml.



**Figure (7).(1) Escherichia coli bacteria with control before growth  $20 \times 10^7$  cell / ml (2) Staphylococcus aurius bacteria with control before growth  $11.3 \times 10^7$  cell / ml (3) Bacillus cereus bacteria with control growth before  $2.5 \times 10^7$  cells / ml.**

The test result of three types of bacteria did not occur any growth as shown in figure 8. More bacteria as soon adhesion to its CuO surface it kill, due to the ions of copper caused a chain reaction and events of negative changes in bacterial cells, the possible mechanisms of action of anti-bacteria, which include the size of oxidative damage by oxygen reactive species (ROS), works on tearing the bacterial cell membrane and lead to the leakage of solutes and function of the cell loss [16-17]. The aim of our thresher was to influence Presence of TiO<sub>2</sub> films with ultraviolet light absorbance and effect on the growth of *E. coli*, *S. aureus* and *Bacillus cereus*. We make use of this characteristic to stop growth of Bacillus cereus, Escherichia coli using disc publishing method. These bacterial progeny were selected as they are highly contagious; thence we can appraise the potential antimicrobial activity of zinc oxide nanoparticles [18]. Table 1 shows the practical consequences of the test.



Figure (8) No happen any growth of the bacteria dt after the test

Table (1) represents the growth of bacteria test.

Type of Antibacterial	E. coli	S. aureus	B.cereus	Range of test Bactria cell/ml before growth
Treatment	liquid	liquid	liquid	E. coli 20*10 <sup>7</sup> cell / ml
CuO TiO <sub>2</sub> -ZnO Coated Cotton	No growth	No growth	No growth	S. aureus 11.3*10 <sup>7</sup> cell / ml
				B.cereus 2.5*10 <sup>7</sup> cells / ml

**Conclusion**

We've been coated cotton fabric in this work successfully by CuO-TiO<sub>2</sub>\_ZnO nano thin film and improve the effectiveness of the cotton fabric against Escherichia coli, Staphylococcus aurius and Bacillus cereus. Topography of surface and structural characteristics has been studied by XRD and SEM. Where structural test results showed that fabric coated by CuO-TiO<sub>2</sub>\_ZnO crystalline thin film, also the analysis detect of SEM, the evaluated grain size of the CuO-TiO<sub>2</sub>\_ZnO nanoparticles.

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