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Antibacterial Study of Silver, Copper, Gold, and Titanium Dioxide Nanoparticles Prepared by DC and RF Magnetron Sputtering

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Abstract

In this work, the antibacterial activity of nanomaterial's copper, silver, gold and titanium dioxide separately was investigated on both gram positive and negative bacteria. Nanoparticles of Cu, Ag, Au and TiO₂ films were grown on glass substrates by DC and RF magnetron sputtering techniques. Nanoparticles films deposition were carried out at optimized argon pressure of 5.5×10^{-2} mbar, sputtering plasma power of 30 Watt for Cu, Ag and Au samples and pressure of 1×10^{-3} mbar, plasma sputtering power of 100 watt for TiO₂ samples. Escherichia coli, Pseudomonas aeruginosa, and staphylococcus aureus were used to evaluate antibacterial activity. In vitro antibacterial analysis indicated that significantly reduced number of used Escherichia coli, Pseudomonas aeruginosa, and staphylococcus aureus were detected on Ag nanoparticles surface compared to an coated substrate surface. Both Cu and Au nanoparticles had inhibited some of pathogenic bacteria and observed over sample area. In the case of TiO₂ films the abatement of bacteria, the antibacterial kinetics was observed to occur with the 120 hrs.

Keywords: Antibacterial activity, Inhibition zone, Metal nanoparticles, sputtering thin films.

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Introduction

The belief that nanotechnology is another area of science and a combination of engineering, biology, chemistry, medicine and physics have accepted to general scientists [1]. Studies have shown that the smaller the particle size, the newer and more different characteristics we have. These features have caused that speed of using nanomaterial spreads very fast, so they can be used in all aspects of life, such as electrical systems and fighting microbes [2]. Metal nanoparticles are used in insecticide and bactericide for many years [3]. Some of nanoparticles are included as a new approach to the development of modern pharmaceutical science. That due to the high potential for specific treatment processes in biology and pharmacology studies is frequently used. For example, they are able to destroy 650 cancer cells, in less than 4 hours [4]. Nanoparticles have shown low toxicity level, in the life cycle and ecosystems, so the use of these materials to combat pathogenic microbes can be a good choice [5]. Metal nanoparticles exhibit different antibacterial properties according to the surface to volume ratio. Gram positive bacteria exhibit greater resistance in contrast to metal nanoparticles compared to gram-negative bacteria, that

this could be related to the structure of the cell wall [6]. In this study different nanoparticles of Au, Ag, Cu and TiO₂ thin films deposited on glass substrates by DC and RF sputtering method and the antibacterial activity of producing thin films nanoparticles have been investigated.

Experimental Procedure

Metals films of Au, Cu and Ag nanoparticles were prepared by home-built dc magnetron sputtering system. Au, Cu and Ag with purity (99.9%) have been used as a sputtering target. The diameter target is 2.5 cm and 0.25 thick and the distance between the top electrode and the target is 5 cm. After loading the system with clean glass substrates, the system was pumped down to base pressure of 1×10^{-5} mbar. Argon gas is introduced into the chamber as precursor to ignite the plasma by applying a negative voltage to the cathode. Before the deposition of each film, the targets are pre-sputtering in Ar for minimum of 15 minutes to remove any surface oxide, in front of the target. Sputtering is done at constant pressure 5.5×10^{-2} mbar, applied voltage of 2 k Volt (30 Watt). Also the TiO₂ thin films were prepared by using RF magnetron sputtering technique (CRC 600 Torr CO.) at argon pressure of 1×10^{-3} mbar and plasma sputtering power of 100 Watt. Deposition time is 90 minutes for all the sputtering experiments. Pathogenic bacteria Escherichia coli, Pseudomonas aeruginosa, and

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staphylococcus aureus, which were standard also investigated were cultured in Mcferland method at 1.5×10^8 cell/ml by Muller –Hinton media, the cultures were incubated for 24 hours and measured the inhibition zone for each materials nanoparticles.

Results and discussion

The microstructure of ananoparticles thin Au, Cu, Ag (prepared at DC sputtering power of 30 Watt) and

TiO_2 films (with a RF sputtering power of 100 Watt) deposited on a (10×10) mm glass substrate was examined using SEM (S4160 HITACH). A columnar structure can be identified and high densely packed spherical and hexagonal-shaped crystallites. The crystalline particles show uniform shapes with a size of around (7.6 - 14.1) nm as shown in table (I) compared with films thickness.

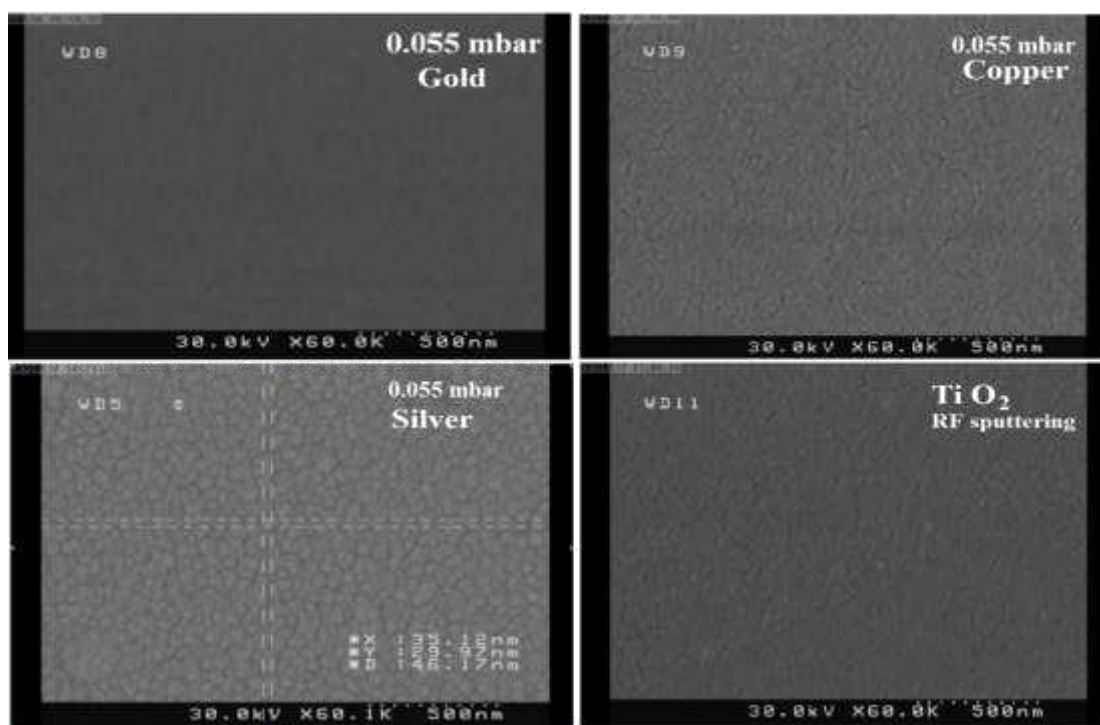


Figure 1

The SEM images of Au, Cu, Ag and TiO_2 thin films deposited on glass substrates

Table 1

Thickness and crystalline size of nano thin films

sample	Thickness (nm)	Particles size (nm)
Ag	80.6	7.6
Au	70.35	9.55
Cu	96.12	7.1
TiO_2	125.4	14.1

The XRD patterns of Au, Cu, Ag and TiO_2 thin films deposited on glass substrate were shown in Fig. (II). Peaks at 2θ values of 37.835° , 43.377° , 63.974° and 76.982° for Au with cubic structure in the directions (111), (200), (220), (311); 42.286° corresponding to (111) direction for cubic Cu. 37.896° , 25.304°

corresponding to (111) direction for cubic Ag; 37.896° ; 37.768° ; 48.0357° corresponding to (101), (004), and (200) planes respectively of TiO_2 are observed and compared with the standard powder diffraction cards (96-901-2431, 96-901-3024, 96-901-3049 and 96-720-6076) respectively.

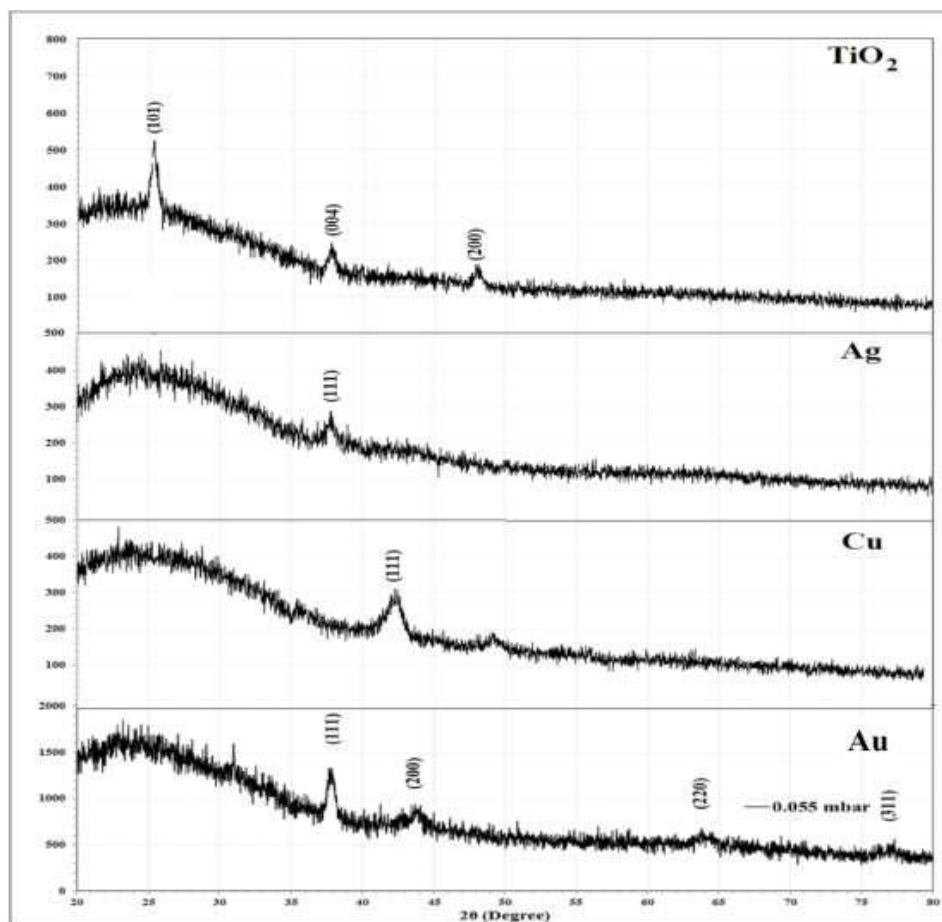


Figure II

X-ray diffraction of Au, Cu, Ag and TiO_2 thin films deposited on glass substrates

Table 2

Comparison between the Exp. and Std. value of d_{hkl} for the Au, Cu, Ag and TiO_2 thin films peaks showed in XRD

Sample	2θ (Deg.)	FWHM (Deg.)	d_{hkl} Exp.(Å)	Crystallite size (nm)	d_{hkl} Std.(Å)	hkl	Phase	card No.
Au	37.8350	0.5798	2.3760	14.5	2.3500	(111)	Au	96-901-2431
	43.7740	1.1100	2.0664	7.7	2.0352	(200)	Au	96-901-2431
	63.9740	1.2396	1.4541	7.6	1.4391	(220)	Au	96-901-2431
	76.9820	1.2096	1.2376	8.4	1.2273	(311)	Au	96-901-2431
Cu	42.2869	1.2060	2.1355	7.1	2.1316	(111)	Cu	96-901-3024
Ag	37.8960	1.1105	2.3723	7.6	2.3500	(111)	Ag	96-901-3049
TiO_2	25.3048	0.7910	3.5168	10.3	3.5172	(101)	Anatase	96-720-6076
	37.7686	0.5374	2.3800	15.6	2.3799	(004)	Anatase	96-720-6076
	48.0357	0.5306	1.8925	16.4	1.8925	(200)	Anatase	96-720-6076

The results of antibacterial properties of Au, Cu and TiO_2 have marked effect on bacteria over the glass chips without changing the size of the bacteria (cellared inhibition zone) during 24 hours as shown in Figure III. While silver nanoparticles showed effective zone against

pathogenic bacteria *Escherichia coli*, *Pseudomonas aeruginosa*, and *staphylococcus aureus* as shown in Figure (IV) and table (3).



Figure III

Examined the zone of inhibition of Au, Cu, and TiO_2 against microorganisms tested (24) hours



Figure IV

Represent zone of inhibition (mm) of silver-nanoparticle against microorganisms tested 24 hours

Table 3

Ag, Au, Cu and TiO_2 nanoparticle effect on inhibition zone against microorganisms tested in (10×10) mm after 24 h

Chips Samples	<i>Escherichia coli</i>	<i>pseudomonas aeruginosa</i>	<i>staphylococcus aureus</i>
Ag	(25 x 25) mm	(23 x 22.5) mm	(17 x 17) mm
Au	No growth over chips	No growth over chips	No growth over chips
Cu	No growth over chips	No growth over chips	No growth over chips
TiO_2 - 150 Watt	No growth over chips	No growth over chips	No growth over chips

Beyond the antibacterial samples introduced into the incubator for 120 hrs, we found the growth of bacteria on the Au, Cu is the same for 24 hrs. Around and above, Ag and TiO_2 chips inhibition zone was

significantly greater than largest zone of 24 hrs process's show in Figure (V) and table (4).



Figure V

Represent zone of inhibition (mm) of TiO_2 -nanoparticle against microorganisms tested (120) hrs.

Table 4

Ag, Au, Cu and TiO_2 nanoparticle effect on inhibition zone against microorganisms tested in (10×10)mm after (120) hrs

Chips Samples	Escherichia coli	pseudomonas aeruginosa	staphylococcus aureus
Ag	(25 x 25)mm	(23 x 22.5)mm	(17 x 17)mm
Au	No growth over chips	No growth over chips	No growth over chips
Cu	No growth over chips	No bacteria over chips	No growth over chips
TiO_2 -150 watt	(13 x 3)mm	(11 x 11)mm	(12 x 12)mm

Silver nanoparticles showed good antibacterial activity against tested bacteria than, copper, gold, and titanium dioxide nanoparticles. This result is agreed with other studies which have shown that the Ag NPs had higher antibacterial activity than other examined nanoparticles, due to the relatively chemical nature [8]. The negative zeta potential for Ag NPs confirms the negative charge on the surface of colloidal nanoparticles cause columbic repulsion forces induced by surface negative charge which enhance the its antibacterial activity [9]. *E. coli* was the most sensitive to silver nanoparticles (have more inhibition zone) followed by *P. aeruginosa* and *S. aureus*. These results are in agreement with other studies [10]. The silver nanoparticles was more active against gram negative bacteria than gram positive bacteria and this was attributed to change in the cell wall composition of bacteria [11].

Conclusion

The adverse effects of antibacterial nanoparticles of different materials evaluated on Gram-positive and Gram-negative bacteria resistant. The results of this study dealt with anti-bactericidal effects of nanomaterials. Silver nanoparticles showed good antibacterial activity against tested bacteria than other nanoparticles. Also *E. coli* was the most sensitive to silver nanoparticles followed by *P. aeruginosa* and *S.*

aureus.

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