

Preparation of Ag nanoparticles via pulsed laser ablation in liquid for biological applications

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Abstract

Ag nanoparticles were prepared using Nd:YAG laser from Ag matel in distilled water using different energies laser (100 and 600) mJ using 200 pulses, and study the effect of the preparation conditions on the structural characteristics of and then study the effect of nanoparticles on the rate of killing the two types of bacteria particles (Staph and E.coli). The goal is to prepare the nanoparticle effectively used to kill bacteria.

Key words

Ag nanoparticles,
staphylococcus,
Escherichia coli
bacteria.

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تحضير جسيمات الفضة النانوية بطريقة الاجتثاث بالليزر النبضي في السائل للتطبيقات الاحيائية

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الخلاصة

تم تحضير الجسيمات الفضة النانوية باستخدام ليزر نيديميوم ياك لمعدن الفضة في الماء المقطر باستخدام مختلف الطاقات ليزر (100 و 600) ملي جول باستخدام 200 نبضة، ودراسة تأثير شروط التحضير على الخصائص التركيبية و تم دراسة تأثير الجسيمات النانوية على معدل قتل نوعين من البكتيريا وهي (العنقوديات والقولونية). والهدف هو إعداد جسيمات متناهية الصغر تستخدم على نحو فعال لقتل البكتيريا.

Introduction

The nanoparticles are produced using different techniques like chemical, physical, mechanical and biological methods etc. Pulse laser deposition is one of these techniques [1]. The mechanics of forming tiny particles of metals through the process pulse laser ablation in liquid (PLAL) include interactions and reactions variable in degrees of heat high and pressure high plasma [2].

Nano particles are used for bacteria killing. The minimum inhibitory concentration is a method for determining the lowest concentration of anti-worker microorganisms to revive that completely inhibits the growth of microbiology under test. In this way the cover containing different concentrations of factor anti-microorganisms designated topic in the pipeline container on the central growth means impregnated revive

microscopic certain, incubating the pipeline for a period of time and at a temperature (37 °C) notes are turbidity associated with the extent of inhibition of microorganisms and less concentration does not give turbidity will be the minimum concentration of inhibition [3].

Wound staphylococcus is a type of bacteria meningococcal cluster species of staph is animated does not build spore arranged in the form of clusters discovered by the French scientist Louis Pasteur in the year (1880) cause skin injury with gram positive (+)

found on the skin and mucous membranes in humans as well as there are in the operating room in hospitals contaminated medical instruments and other [4].

Escherichia coli is a type of intestinal bacteria with feather that live in the intestines of mammals discovered Aichirh also known germ large bowel. E. coli bacteria causing these negative gram (-) different bacteria diarrhea severity and symptoms and As in Fig. 1 simplified form this type of bacteria [5].

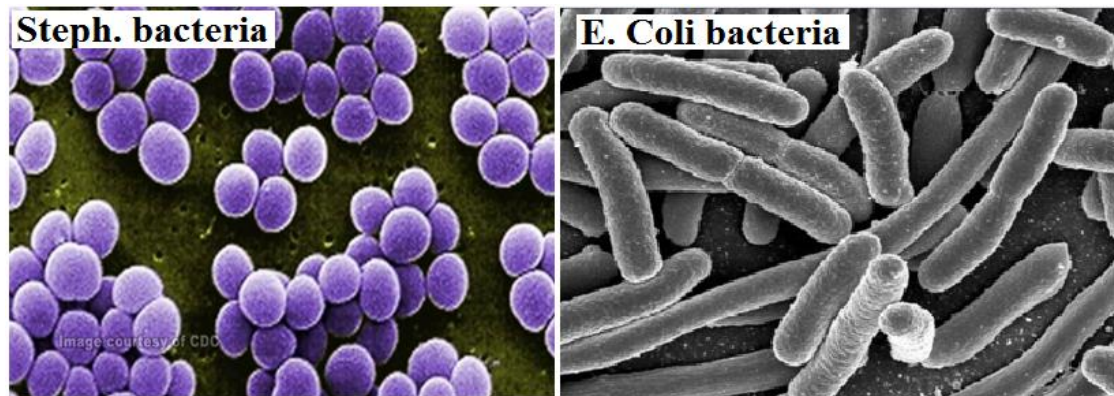


Fig. 1: Shape and color E.coli bacteria [5].

The x-ray diffraction peaks broadening is a fundamental technique for producing nanoparticles, where the crystalline size estimated by Scherrer's equation formula [6].

$$D = \frac{0.9 \lambda}{\beta \cdot \cos(\theta)} \quad (1)$$

where λ is x-ray wavelength (1.5406 nm for K_{α} transition in Cu target), β is full width at half maximum (in radians) and θ is a diffraction angle.

Experimental part

Piece of silver metal (Ag) the target samples were put in the bottom of

(4) ml glass tube filled with distilled water then bombarded by Nd:YAG pulse laser (Hua Fei Tong Da Technology –Diamond -288 Pattern EPLS) at (6) Hz frequency and (9) ns pulse duration with 1064 nm wavelengths and with several pulse energies (100 and 600) mJ. Schematic of system used to prepare the colloidal nanoparticles as shown in the Fig. 2. The produced nanoparticles were examined by X-ray diffraction and SEM microscopy.



Fig. 2: Schematic of system used to prepare the colloidal nanoparticles.

Fig. 3 shows that curves Fourier Transform Infrared Spectroscopy (FTIR) Thermo Scientific Nicolet 10 FTIR Spectrometer. Sample film Ag pure samples, the peak $(675) \text{ cm}^{-1}$,

which is for Ag-O deformation, and the peak $(1611) \text{ cm}^{-1}$, which is for Ag-O stretching, the peaks belonging to (O-H) about to $(3415) \text{ cm}^{-1}$ [7].

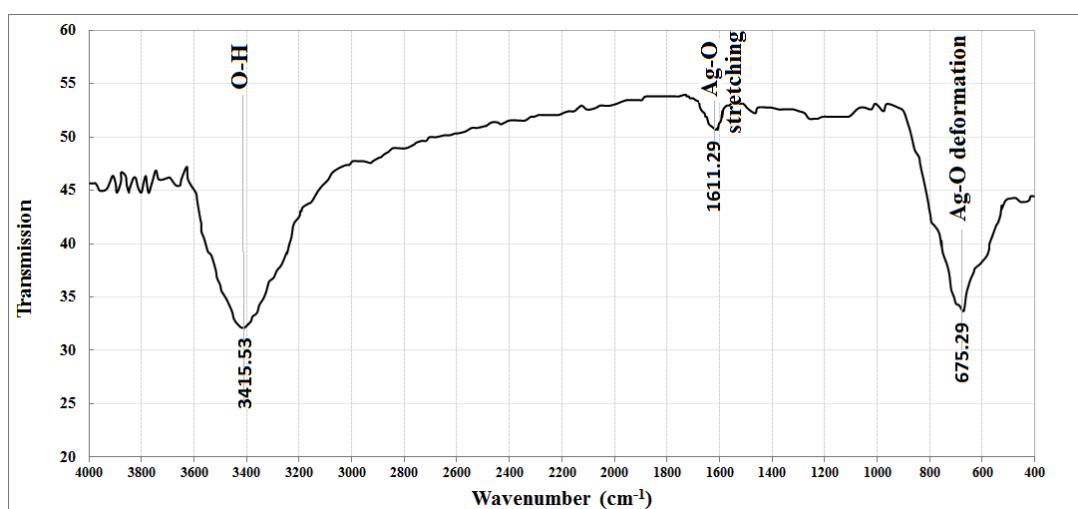


Fig. 3: FTIR pattern for Ag pure thin film.

Table 1: FTIR peaks for Ag thin film pure.

Type of bond	Pure Ag NPs
Ag-O deformation vibration	675.29
Ag-O stretching vibration	1611.29
O-H	3415.53

The antimicrobial activity of silver nanoparticles against two bacterial strains *Staphylococcus aureus* and

Escherichia coli. After identification and checking the purity of examined bacteria, isolates were inoculated into

0.5ml of sterile Mueller-Hinton broth, mixed well to prepare a homogenous bacterial suspension with a turbidity equivalent to 0.5 McFarland standards. The bacterial suspension was prepared and adjusted by comparison against 0.5 Mc-Farland turbidity standard and all bacteria strains were sub- culture on nutrient broth and added 0.5 ml of Ag nanoparticles at difference concentration ($\mu\text{g.ml}^{-1}$). *S. aureus* and *E.coli* was investigated by broth medium methods.

After determine the minimum inhibitory concentrations of each material nanoparticle preparation, was laying pipes clear after with size (10)ml the tube that represents a minimum inhibitory concentrations at the center of the agar Mueller Hinton record as stated in the company processed instructions, dishes

incubated at 37 °C for 24 hours, and represents the first free of any colony dish bacterial [3].

After nanomaterial determining the minimum inhibitory concentrations for material nanoparticle preparation (Ag) we determine minimum bactericidal concentrations was laying pipes clear after the tube that represents a minimum inhibitory concentrations at the center of the agar Mueller Hinton record as stated in the company processed instructions, dishes incubated degree of 37 °C for 24 hours, and represents the first free of any colony dish bacterial [3].

After (MBC) we were calculate the ratio percentage of the inhabitation was obtained on different killing ratios for both types of bacteria, this is agree with [8].

$$\text{Per. of (k)} = \frac{[\text{Viable count at } 0 \text{ h}(x_0) - \text{Viable count at } 24 \text{ h}(x_1)]}{[\text{Viable count at } 0 \text{ h}(x_0)]} \times 100\% \quad (2)$$

where (x_0) is number of colonies living before treatment which is equal to (1400) colony, (x_1) is number of colonies living after treatment with silver nanoparticles (Ag NPs) of the number of colony in full growth dish before you make the process of adding the nanoparticle solution.

Results and discussion

Fig. 4 shows the XRD pattern of silver (Ag) nanoparticles was determined from XRD diffraction pattern, which showed a hexagonal

(hex.) quartzite phase of Ag. The figure showed XRD pattern for prepared silver nanoparticles (Ag NPs) where the emergence of two peaks were illustrated at (37.76°) and (43.94°), which refer to the planes of (111) and (200) indicated crystalline silver (Ag) and conformity are good with that of ref. [9].

Table 2 shows all XRD peaks, grain size (calculated by Scherrer equation), inter plane spacing distance (d_{hkl}) and comparison with standard values (96-901-3049).

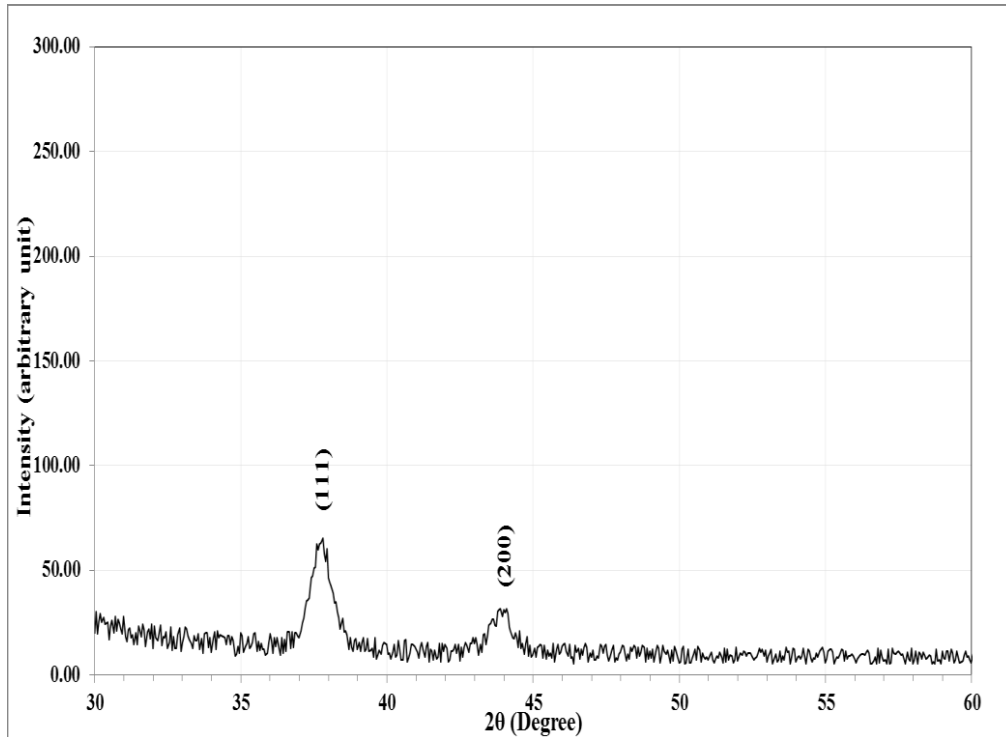


Fig. 4: XRD pattern for Ag nanoparticles sprayed on glass substrates.

Table 2: XRD peaks for titanium oxide (Ag) nanoparticles.

2θ (Deg.)	FWHM (Deg.)	d _{hkl} Exp.(Å)	D (nm)	d _{hkl} Std.(Å)	hkl	Phase	card No.
37.7620	0.8550	2.3804	9.8	2.3500	(111)	Hex. Ag	96-901-3049
43.9411	0.8560	2.0589	10.0	2.0352	(200)	Hex. Ag	96-901-3049

It has a polycrystalline structure with preferred direction at (111) and (200). Fig. 5 shows the scanning electron microscopy images for produced Ag nanoparticle in two magnification

powers (10µm and 20µm) this figure illustrates the presence of silver nanoparticles clearly show non-agglomeration of particles even after drying as shown in Fig. 5 [10].

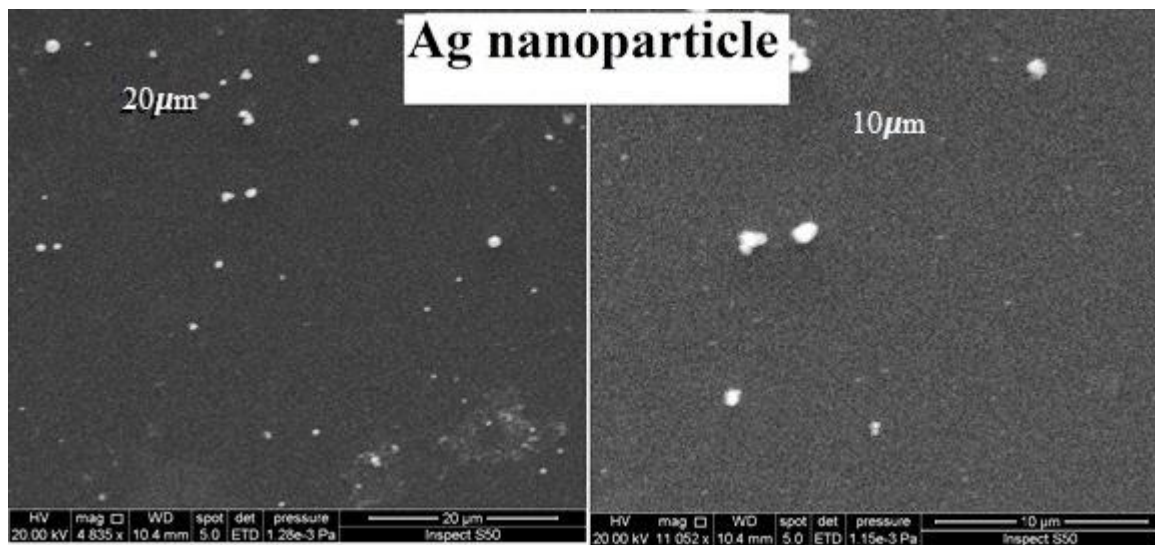


Fig. 5: SEM images for Ag nanoparticle deposited on glass substrate for 10µm and 20µm.

Fig. 6 shows that curves FTIR sample film Ag pure samples kinds of bacteria are E.coli and staph killed by Ag appeared in all samples, the peak (658, 666) cm^{-1} , which is for Ag-O deformation vibration, and the sample (1615, 1619) cm^{-1} , which is for Ag-O stretching vibration, where the intensity of the peaks were stronger in the samples pure, also appeared the peaks belonging to(O-H) about to (3445, 3432) cm^{-1} , while the bond

appeared (C-O stretch), with peaks at (1014, 1001) cm^{-1} , bond (C-H) about (2907, 3072) cm^{-1} just in E.coli. The disappearance of some of the peaks belonging to (C=C) about to (1450, 1535) cm^{-1} in E.coli only, vanished in the sample which had completely killed and some of the values that belong to (C-H bend) about (1378, 1399) cm^{-1} as Table 3. These results agree with other researches [7].

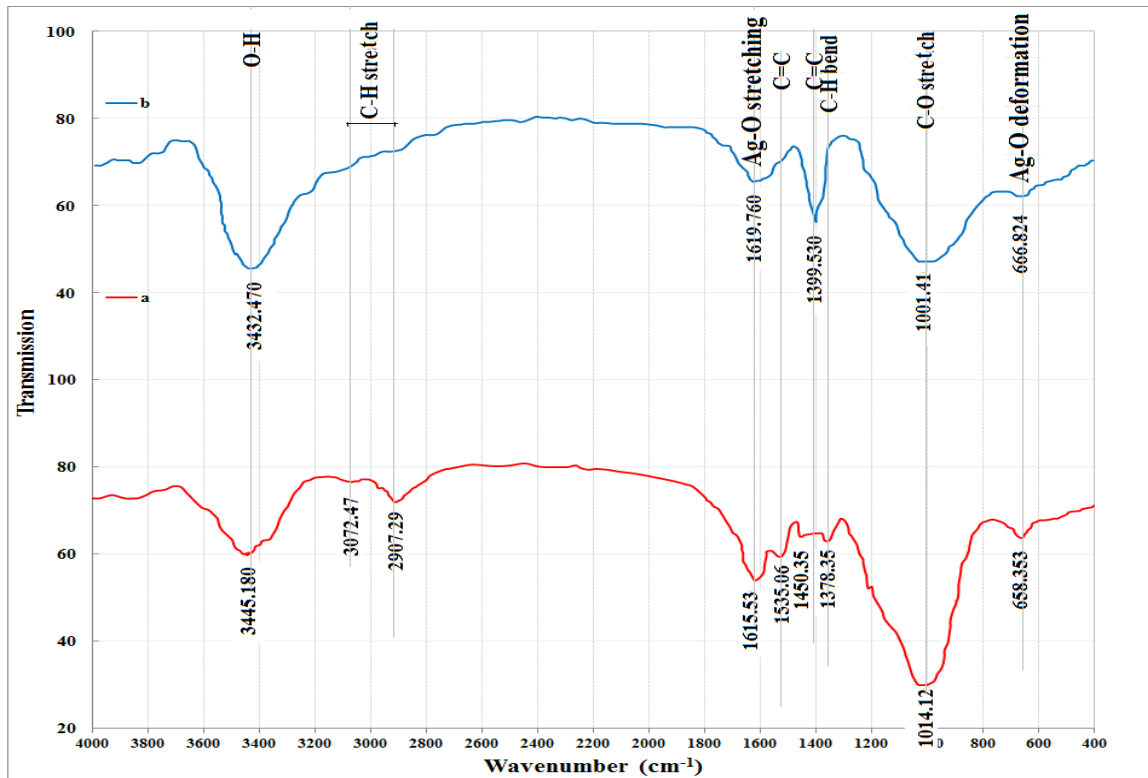


Fig. 6: FTIR pattern for (a) bacteria killed with Ag for E.coli. (b) bacteria killed with Ag for staph. sprayed on glass substrates with take part from dishes.

Table 3: FTIR peaks for Ag film, E.coli, staph bacteria killed with Ag and Staph bacteria killed with Ag.

Type of bond	E.Coli bacteria Killed with Ag	Staph. bacteria Killed with Ag
Ag-O deformation vibration	658.353	666.824
C-O stretch	1014.12	1001.41
C-H bend	1378.35	1399.53
C=C	1450.35 1535.06	-
Ag-O stretching vibration	1615.53	1619.76
C-H	2907.29 3072.47	-
O-H	3445.18	3432.47

Fig. 7 shows the application of (Ag) nanoparticles prepared with energy (600) mJ and (200) pulses the existence ratios were killed few for degrees mitigation concertation (2.34) $\mu\text{g}\cdot\text{ml}^{-1}$ for both types of bacteria used and compared with the operations of the previous killings, got on killing ratio for E.coli (60.7)% and staph (39.32)%, this is because the large size

of particle nanoscale is due to high laser energy inflicted on the target and (600) mJ gives a large particle, but for low laser energy (100) mJ, it gives small nano-size that facilitate penetrate the cell wall of the cell and to stop the work of DNA this is agreement with [11]. The percentage of killing ratio calculated by applying the Eq.(2).

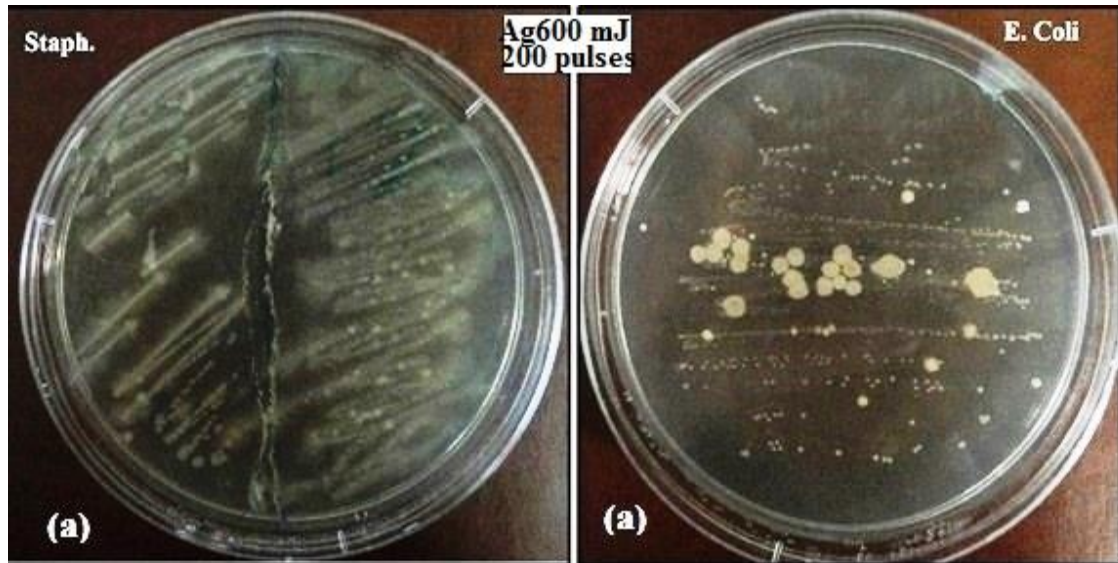


Fig.7: Killing ratio bacteria with (Ag) with energy (600)mJ with (200)pulses partial kill for staph and E.coli.

Fig. 8 shows` the complete killing of bacteria Staphylococcus aureus and Escherichia coli application of particle silver (Ag) nanoparticles record with energy (100) mJ and (200) pulses in distilled water (DW) for the first concentrations to alleviate (MIC), where the silver specifically with

energy (100) mJ and concertation first (1.348) $\mu\text{g}\cdot\text{ml}^{-1}$ and all degrees of mitigation (MIC) got on completely kill bacteria for both types, and this shows that silver has the ability on inhibition and killing more than other material this is agreement with [10].

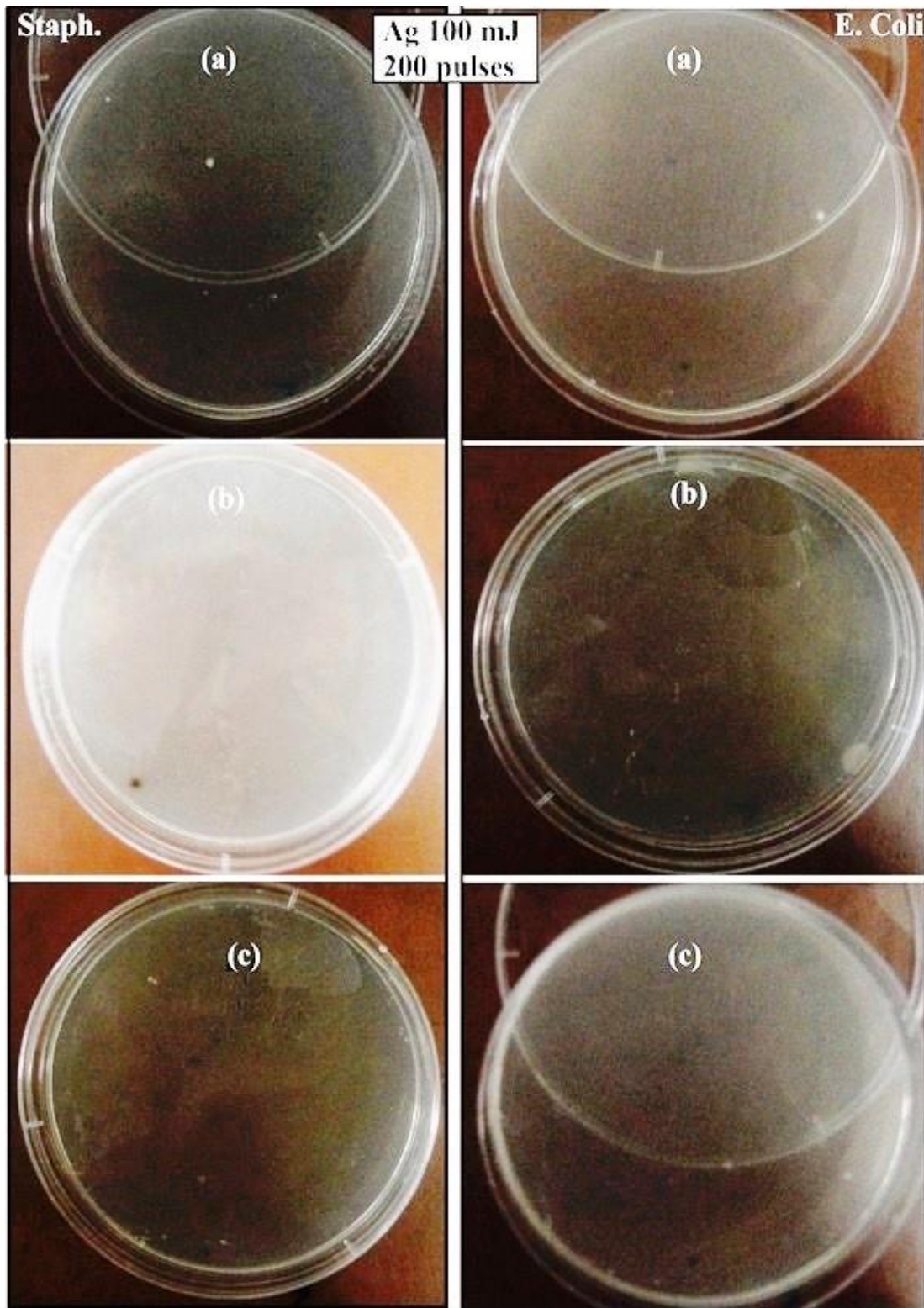


Fig.8: Killing ratio bacteria with (Ag) with energy (100)mJ with (200)pulses with different concentration (MIC) for every type from bacteria.

Conclusions

Silver nanoparticles were obtained by pulse laser using 100 mJ and 600 mJ for 400 and 4000 pulse. X-ray diffraction for produced nanoparticles

showed that the samples have small crystalline size (about 9.8 nm). SEM image showed the resulting nanoparticles ranging from too small (less than 50 nm) spherical shape.

These samples were tested could be used as an antibacterial for the two types of bacteria. High efficiency tests for the killing of the samples prepared with 200 pulses even when less energy (100) mJ, while samples were prepared at 200 pulses with high energy (600) mJ less effective.

FTIR spectra confirm that the bond (1611, 675) cm^{-1} for AgO. Antibacterial activity experiments performed on various microorganisms clearly demonstrated the effectiveness of nanoparticles against bacterial growth due to smaller particle size that penetrates the membrane of bacterial cell.

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