

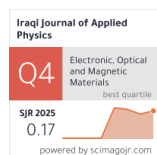
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Characterization of Gold and Silver Nanoparticles Prepared by Pulsed Laser Ablation in Liquid

In this study, gold and silver nanoparticles were prepared by pulsed laser ablation, where the physical properties of both metals were studied at different parameters (600 and 900 pulse/s). Results confirmed cubic crystalline system for gold and silver. A particle size of 49.15 nm was obtained for the gold sample prepared at 600 pulses/s, while the sample prepared at 900 pulses/s showed a particle size of 37.67 nm. The silver sample showed 66.68 and 50.21 nm, respectively. The plasmon resonance was determined at 525 and 519 nm for gold and at 410 and 405 nm for silver. The nanoparticles prepared at 900 pulses/s were integrated with dental fillers. Compressive test results for the sample without nanomaterials showed a ratio of 55. When mixing 3 ml of gold nanoparticles with the filler, the ratio was 78, while mixing 3 ml of silver nanoparticles with the filler showed a ratio of 72.

Keywords: Gold nanoparticles; Silver nanoparticles; Laser ablation; Structural characteristics

1. Introduction

New and improved particles are being produced for many applications. Physicist Richard Feynman introduced the concept of nanotechnology in 1959 in his lecture "There's Plenty of Room at the Bottom". Nanoscience is a combination of the words "science" and "nano", which means "dwarf" [1,2]. Nanoparticles are larger than individual atoms and molecules but smaller than large solids, and therefore they are not subject to absolute quantum chemistry or the laws of classical physics and have properties different from those expected [3-6]. Properties that cannot be seen at the macroscopic level are now important at the nanoscale, such as quantum mechanics [7], optics, magnetism, surface interactions [8], and thermodynamics [9]. The nanoscale is where materials can have different properties, some of which are better at conducting electricity or heat, some of which are stronger, some of which have different magnetic properties, and some of which reflect light better or change color as their size changes [10]. When the dimensions of a material are reduced to the nanoscale, it takes on new physical and chemical properties that differ significantly from those of the bulk material. This is what distinguishes nanoparticles and makes them a new scientific marvel in constant improvement [11,12].

Nanomaterials are unique in terms of their small size and suitability for medical applications, where they are used to deliver drugs to specific tissues by coating nanoparticles with drugs or proteins, enabling them to reach the target tissue by penetrating cell

walls [13]. Among biomedical diagnostic drugs and prosthetic limbs, nanotechnology and nanomaterials are widely used. Biological and biochemical sensors have been used to evaluate blood and other biological samples for use in biomedical nanotechnology outside the body as well as inside the body [14,15]. Researchers are expected to use specific systems such as insulin pump implants and gene therapy for in vivo use, in addition to developing prosthetics and surgical implants with nanostructures [16].

Laser ablation in liquids is a rapid and straightforward method for producing metallic nanoparticles for chemical sensing and bioanalytical applications. These nanoparticles are known as surface plasmon resonators [17,18]. Modern physical methods, whose fundamentals were developed in physics laboratories, have become crucially important in nanoscience [19]. Nanophysics combines multiple disciplines using theoretical and experimental methods to determine the physical properties of materials at the nanoscale. Interesting properties include the structural, electronic, optical, and thermal behavior of electrically and thermally conductive nanomaterials [20]. Nanophysics has now become an independent branch of physics and is expanding into many new areas, playing a vital role in fields that were previously the domain of engineering, chemistry, or life sciences [21,22].

2. Experimental Part

A sheet of gold with a purity of 99.9% was used with dimensions of 1x0.8x0.2cm and a sheet of silver

with a purity of 99.9% with dimensions of 1x0.8x0.2cm and ethanol with a purity of 99% was used as a solvent. Using pulsed laser ablation in liquid technology with parameters wavelength 1064 nm, energy ablation 1000 mJ, pulse of number 900 pulse per second, and duration time 8 ns, before starting the work, the samples were cleaned using distilled water and placed in an ultrasonic bath to remove impurities from the targets used to obtain high-purity nanoparticles. Then, the preparation of gold and silver nanoparticles was started, each material separately using the parameters mentioned above, as illustrated in Fig. (1).

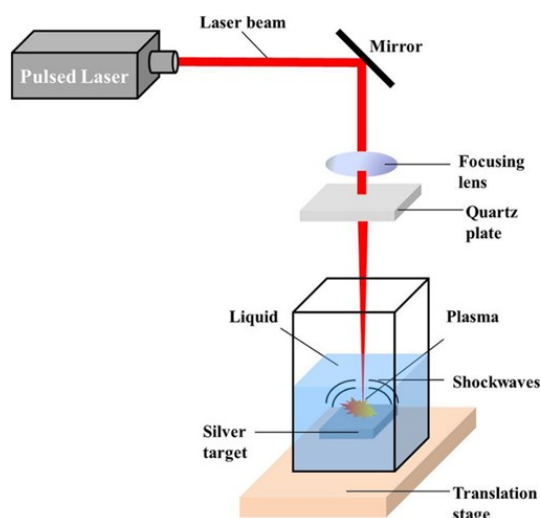


Fig. (1) Schematic diagram of the laser ablation process

3. Results and Discussion

The results of X-ray diffraction of the gold nanoparticle solution show that the crystal system is cubic. We observe three peaks at angles (38.25° , 44.51° , and 64.79°), which correspond to the directions (111), (002), and (022), respectively. These results are in good agreement with the values reported in the ICDD card no. (98-016-3723) for gold. In addition, the (111) crystal plane orientation is predominant. The peak at (64.74°) indicates (022), which achieved a polycrystalline form of face-centered cubic (FCC). Figure (2a) shows the agreement between the standard and experimental data, with a slight change in the atomic plane distances (d) due to lattice strain caused by defects affecting the preparation of nanomaterials, the average crystallite size (D) of AuNPs was 19.74 nm.

The X-ray diffraction results for the silver oxide nanoparticle solution showed that the crystal system has a cubic structure of a monoclinic polycrystalline cub, we observe three peaks at angles 37.22° , 63.65° , and 79.21° corresponding to the (111), (311), and (222) planes, respectively. These results are in good agreement with the values listed in the ICDD card no. (1038-43) for silver oxide. In addition, the (111) crystal plane orientation is predominant. Figure (2b)

shows the agreement between the standard and experimental data, with a slight change in the atomic plane distances (d) due to lattice strain caused by defects affecting the preparation of the nanomaterials [137]. The average crystallite size (D) of AgONPs was (24.7 nm).

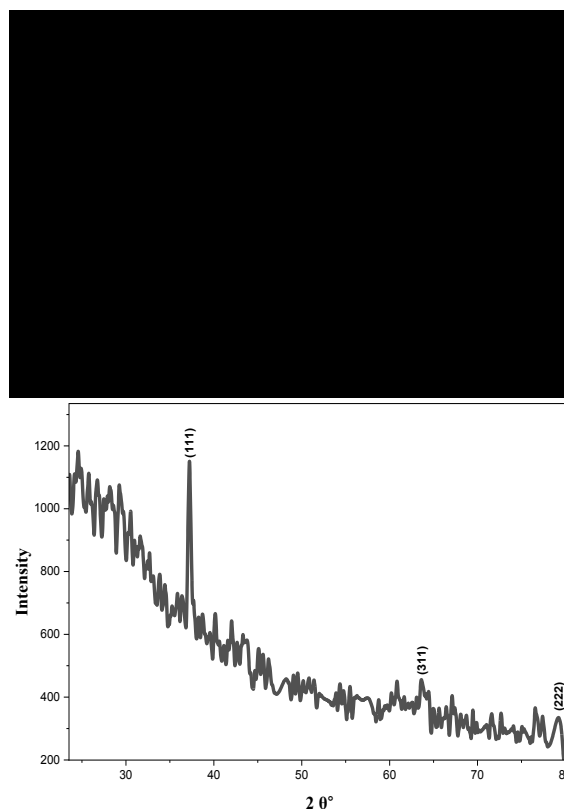


Fig. (2) XRD patterns prepared using (a) AuNPs, and (b) AgONPs

Figure (3) shows the results of the FE-SEM of gold nanoparticles prepared in DW solution using 600 pulse/s after deposition on a glass slide, where nanoparticles ranging in size from 28.53 to 66.53 nm, with an average diameter of 49.15 nm, as shown in Fig. (3b) they have irregular spherical shapes, and agglomeration of particles is observed. Meanwhile, the sample prepared at 900 pulses/s yielded particles ranging between 19.27-66.21 nm with an average diameter of 37.67 nm as seen in Fig. (3d), they have irregular spherical shapes, and there is less agglomeration of particles with a smaller particle size than the 600 pulse/s sample due to the increase in the number of pulses.

Figure (4) shows the results of the FE-SEM of silver oxide nanoparticles prepared in DW solution using 600 pulse/s after deposition on a glass slide, where nanoparticles ranging in size from 45.38 to 90.45 nm, with an average diameter of 66.68 nm as shown in Fig. (4b), they have regular spherical shapes, and as can be seen, there is agglomeration of particles, while the sample prepared at 900 pulses/s ranges

31.32–72.08 nm with an average diameter of 50.21 nm as seen in Fig. (4d), they have regular spherical shapes, and there is also particle agglomeration and smaller particle sizes due to the increase in the number of pulses.

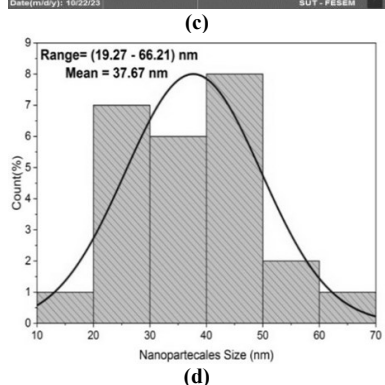
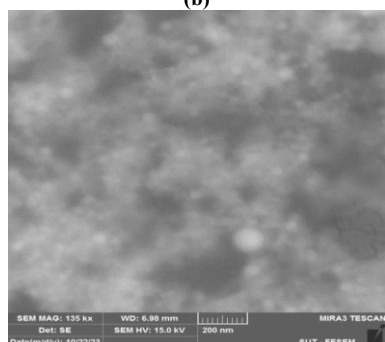
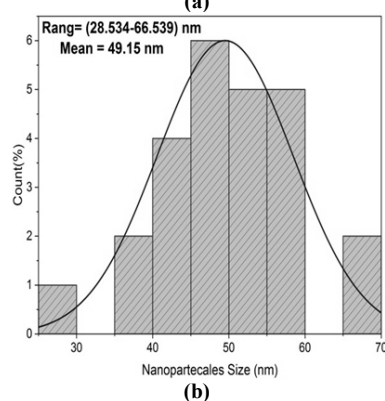
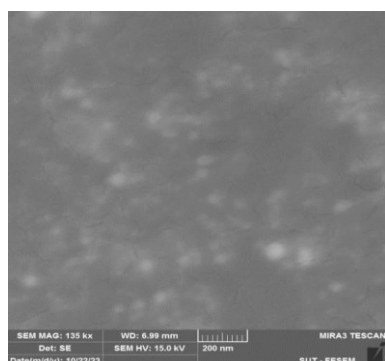


Fig. (3) FE-SEM results of AuNPs (a) at 600 pulse/s, (b) at 900 pulse/s, (c) distribution size for sample 600 pulse/s, (d) distribution size for sample 900 pulse/s

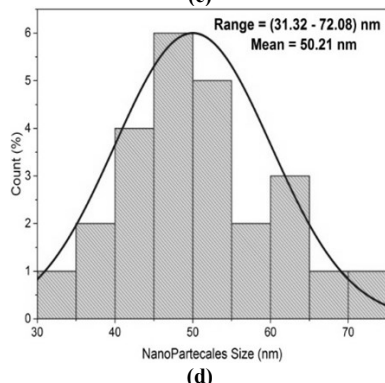
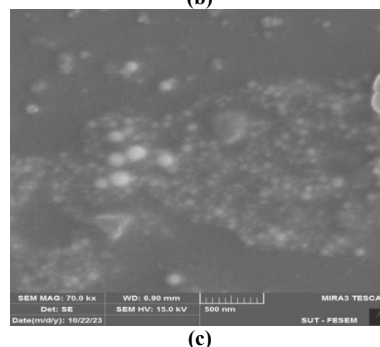
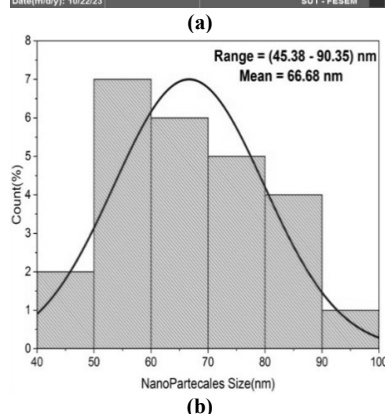
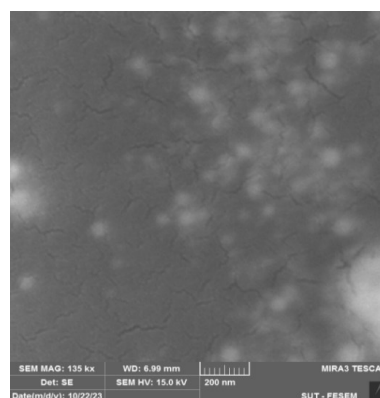


Fig. (4) FE-SEM results for AgNPs (a) at 600 pulse/s, (b) at 900 pulse/s, (c) distribution size for sample 600 pulse/s, (d) distribution size for sample 900 pulse/s

After preparing colloidal solutions containing metal nanoparticles (Au, AgO) using the PLAL method to study the effect of laser pulses on the properties and size of nanoparticles at a constant energy (1000 mJ) and different pulse numbers (600,

900 pulses) in deionized water and using a laser wavelength (1064 nm) where the laser beam hitting the surface of the metal immersed in the liquid leads to the formation of a spark column with a strong shock wave that spreads in all directions floating in the liquid. It was observed that the color of the solution changed and its density increased with the increase in the number of laser pulses, which is a feature of transition elements, indicating the formation of a solution containing nanoparticles of the extracted metal and surface plasmon resonance (SPR) peaks. SPR in the ultraviolet and visible regions is a distinctive sign of the formation of metal nanoparticles for gold, silver oxide, and titanium dioxide [23]. This change is due to the oscillation and coherence of electrons on the surface of the nanoparticles, as well as an increase in the number and size of the extracted particles, and the intensity of absorption increases with an increase in the number of pulses [24]. An increase in laser pulses led to an increase in the intensity of absorption and an increase in the intensity of the SPR peaks. The width and height of the absorption peaks depend on the number of laser pulses. The spectral change indicates an increase in the abundance of nanoparticles by laser irradiation, and the presence of a plasmon peak was observed, which means that the nanoparticles were spherical and sub-spherical in shape [25,26]. The colloidal solution of gold nanoparticles prepared with different pulse numbers and constant energy was pale pink in color before turning dark pink with an increase in the number of pulses while an increase in laser pulses leads to an increase in absorption intensity and the surface plasmonic peak location of gold nanoparticles at approximately (525 nm) in the visible and absorptive region (0.244) (Fig. 5a). The lowest absorption was obtained at 600 pulses and the highest absorption at 900 pulses. As for the silver oxide nanoparticles prepared with different pulse numbers and constant energies, the colloidal solution was pale green in color before turning dark green as a result of the increase in the number of pulses. Figure (5b) shows the absorption spectrum of silver oxide nanoparticles. An increase in laser pulses leads to an increase in absorption intensity and the surface plasmonic peak location of silver oxide nanoparticles at approximately 410 nm in the visible and absorptive region (0.584), and the nanoparticles were produced. The lowest absorption was obtained at 600 pulse/s and the highest absorbance at 900 pulse/s, as shown in table (1).

The optical bandgap of the direct transition allowed for the prepared nanoparticle solutions and all the metals used (Au and AgO). The energy bandgap is strongly affected by the chemical bonds of the elements composing their structure and their particle size and generally changes with the change in particle size. We notice that the energy band gap (E_g) value of

AuNPs prepared in D.W increased from 2.005 to 2.027 eV. While the prepared AgONPs showed an energy bandgap (E_g) value that increased from 2.47 to 2.5 eV, respectively. Figure (6) shows the optical bandgap of the prepared nanoparticles. As we notice that the optical energy gap edge of titanium dioxide nanoparticles is larger than other particles due to the quantum confinement effect, the decrease in the size of the nanoparticles is responsible for the increase in the value of (E_g) with a larger value, which is suitable for obtaining small-sized particles and can also be attributed to electron-filled levels near the conduction band where electrons require more energy to move [27].

Table (1) SPR and absorption intensity results obtained from the absorption spectrum at 1064 nm wavelength, 1000 mJ energy and different numbers of pulse per second

NPs in DW	Number of Pulses	Absorbance	SPR Peak (nm)
AuNPs	600	0.22	519
	900	0.24	525
AgNPs	600	0.52	405
	900	0.58	410

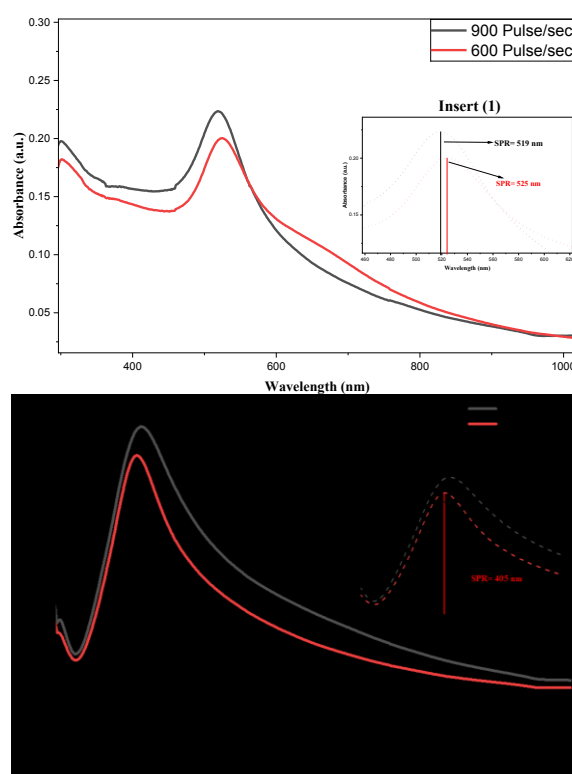


Fig. (5) UV-visible spectra of (a) AuNPs, and (b) AgNPs

Compressive strength testing aims to ascertain a material's behavior or response to a compressive load, and to do so, key variables such as stress and strain are measured. Compressive strength is the amount of pressure required to cause the sample to rupture under

a compressive load during the chewing process where pressure is a major factor [28]. It is important to perform this measurement on dental fillings intended for testing because it has been proven through multiple studies that the majority of the stresses experienced by fillings within the oral cavity are of a compressive nature [29,30]. During the examination of the specimens, a load is applied to the specimens and the load is increased and at the yield point, the material changes from elastic deformation to plastic deformation and the load is gradually increased until the specimen is finally crushed. Figures (7a, b, and c) show the stress-strain curves for the pure dental filling and the dental filling reinforced with gold, and silver oxide nanoparticles. We observe that the yield point is less pronounced in the pure dental filling and more pronounced in the AuNPs supported dental filling due to the longitudinal stabilization of the specimen containing AuNPs and because AuNPs has high strength and hardness and good corrosion resistance, and we also observe an increase in the stress-strain value and an increase in the Young's modulus and also an increase in the compressive strength value between the pure dental filling and the nanomaterial-supported dental filling due to the ability of nanomaterials to increase the surface area and these properties enable nanoparticles to fill the voids between the substrate and the matrix. The highest value of compressive strength was obtained for the gold nanoparticle-reinforced dental filling, and followed by silver oxide-reinforced filling, the results are shown in table (2).

Table (2) Values of greatest stress, greatest strain, Young's modulus and compressive strength for pure dental fillings as well as nanomaterial-reinforced dental fillings

Samples	Young's Modulus (N/mm ²)	Max Stress (N/mm ²)	Max Strain	Compressive Strength (MPa)
Pure	8.72	51.83	2.79	55
Pure + Au NPs	12.65	58.57	4.35	78
Pure + AgO NPs	12.36	57.89	3.75	72

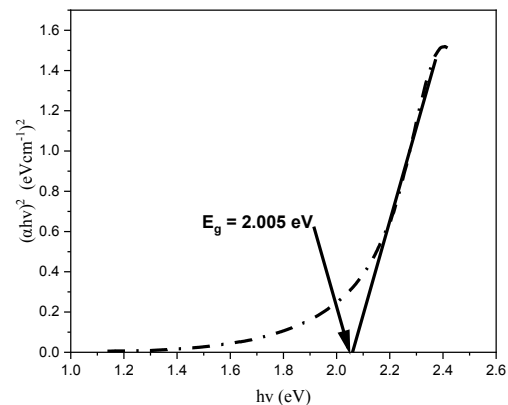
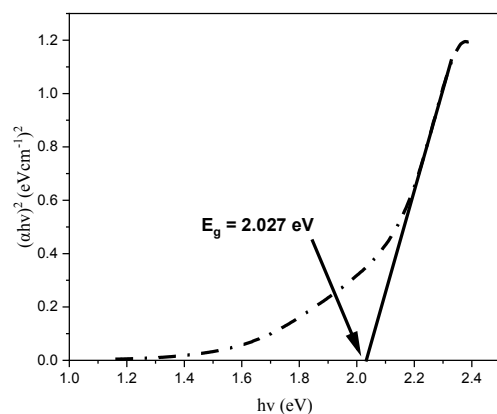


Fig. (6) Values of energy bandgap for AuNPs prepared at (a) 600 pulse/s, and (b) 900 pulse/s

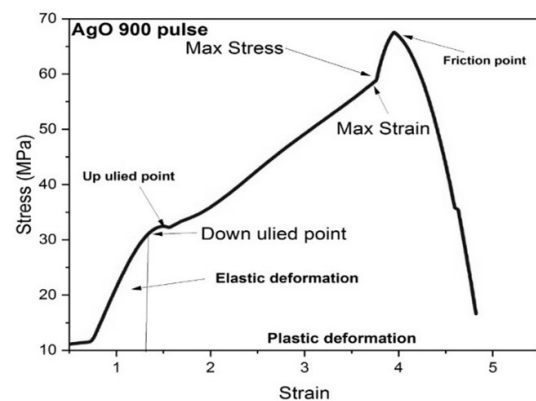
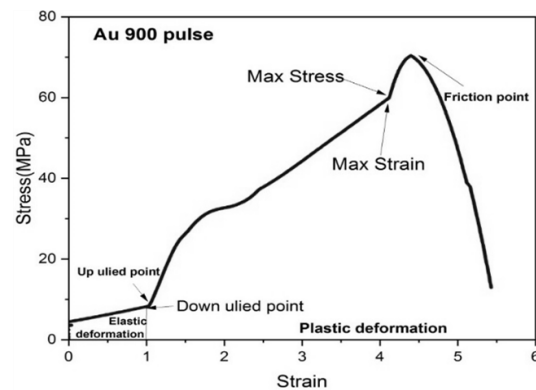
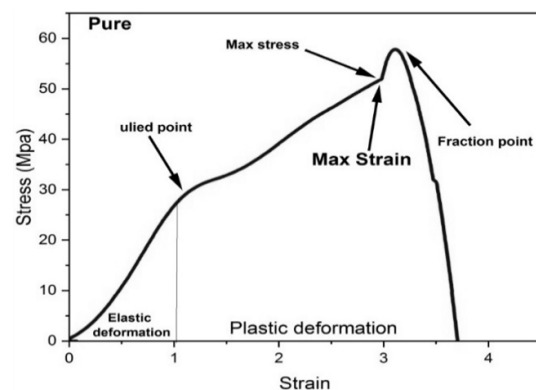


Fig. (7) (a) Stress-strain curves for pure dental fillings, (b) Stress-strain curves for AuNPs-supported dental fillings, (c) Stress-strain curves for dental fillings reinforced with AgONPs

4. Conclusion

In an easy, simple and inexpensive way, gold nanoparticles and silver nanoparticles were prepared. We conclude from this work that the same phase was obtained through the results of X-ray diffraction, while the results FE-SEM indicate that the more the number of pulses, the smaller the nanoparticles were obtained. Also, using the same parameters and conditions, the average size of gold particles was less than silver. As for the surface plasmon resonance of gold, it indicates that the more the number of pulses, the shift towards the lower wavelengths, while silver shifts towards the longer wavelengths. As for the results of the mechanical properties through measuring compressibility, it was shown that gold had the best compressibility at 78 compared to what it was without gold nanoparticles at 52, while silver had a compressibility of 72, these results are considered distinctive and will last for a long time. The mechanical properties of dental fillings.

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