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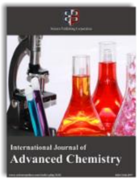

# The effect of temperature on cadmium oxide (CdO) nanoparticles produced by synchrotron radiation in the human cancer cells, tissues and tumors

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**Abstract**

In this work, the effect of temperature of the ablation environment on the properties of Cadmium Oxide (CdO) nanoparticles produced by synchrotron radiation is investigated. To produce nanoparticles, synchrotron radiation pulse with 1064 (nm) wavelength is used to emit Cadmium in the human cancer cells, tissues and tumors. All test parameters were kept constant and human cancer cells, tissues and tumors temperature was changed to produce samples at 20°C and 65°C. Then, ATR–FTIR, XRD, TEM and UV–Visible spectroscopy analyses were performed to investigate their properties. The results show that the size of nanoparticles is increased by increase in temperature of ablation environment. In addition, in the current experimental research, Gold (Au)–Cadmium Oxide (CdO) alloy is created at the size of nano. In this regard, same volume of Gold and Cadmium Oxide (CdO) solutions were mixed together and emitted by the synchrotron radiation pulse with wavelength of 532 (nm). The Gold and Cadmium Oxide (CdO) solutions have been produced, separately, using synchrotron radiation ablation process. To produce them, synchrotron radiation pulse with wavelength of 1064 (nm) and pulse width of 7 (ns) and repeating frequency of 5 (Hz) was used. The results show that synchrotron radiation emission with wavelength of 532 (nm) is an appropriate method for producing Gold compounds in the size of nano.

***Keywords***: *Cadmium Oxide (CdO); Absorption Spectrum; Synchrotron Radiation Ablation; Nanoparticles; Alloy; Synchrotron Radiation Emission; Nanocomposite.*

## 1. Introduction

Regarding unique physical and chemical properties of Cadmium Oxide (CdO), this material is applicable in a wide range including pigments, solar energy, and photocatalysts. There are three structures for Cadmium Oxide (CdO): Rutile (tetragonal), anatase (tetragonal) and brookite (orthorhombic). Rutile, the most stable phase of Cadmium Oxide (CdO), is in a mass form and in the environmental condition. It should be noted that production method of Cadmium Oxide (CdO) affects the final production phase. In pulse synchrotron radiation ablation, as a physical method for synthesizing nanoparticles, size of nanoparticles can be controlled by changing various parameters such as synchrotron radiation wavelength, synchrotron radiation pulse duration, pH of solution, adding surfactant and changing the temperature of solution (Heidari and Brown 2018; Heidari 2016). In the current test, Cadmium Oxide (CdO) nanoparticle formation using synchrotron radiation ablation method is investigated and the effect of environment's temperature on the size of nanoparticles is studied.

Various researches have been indicated that synchrotron radiation ablation is an important process for producing nanoparticles in liquid environment. In this method, nanoparticles produce without any chemical waste and its characteristics can be controlled through controlling synchrotron radiation characteristics such as wavelength, frequency, stain size, and intensity. In recent years, synchrotron radiation has been widely used to produce nanocomposites or nanometric alloys. The results have shown that synchrotron radiation ablation is an effective mechanism for producing nanocomposites. The produced particles are of pea absorption Plasmon of Gold at about 530 (nm) along with large optical gap of Cadmium Oxide (CdO). In this regard, Gold–Cadmium Oxide (CdO) nanocomposite can be used as a unique material in optic industry.

## 2. Test arrangement

Nanoparticles of Cadmium Oxide (CdO) were produced by synchrotron radiation ablation of a Cadmium plate with high degree of purity in distilled water. The Cadmium plate was placed at the bottom of a container containing 35 (ml) distilled water so that target was placed at the depth of 2 (cm) from the water surface. Plate and the container were cleaned before test by ultrasonic method in alcohol, acetone and water. Cadmium plate was ablated with 5000 pulses by synchrotron radiation pulse with repeating rate of 10 (Hz) and pulse width of [6] (ns). However, samples were produced at 20°C and 65°C.

Nanoparticles of Cadmium Oxide (CdO) were produced by applying synchrotron radiation with 1064 (nm) wavelength. The solution samples containing nanoparticles of Cadmium Oxide (CdO) with clear blue color is shown in Figure (1).

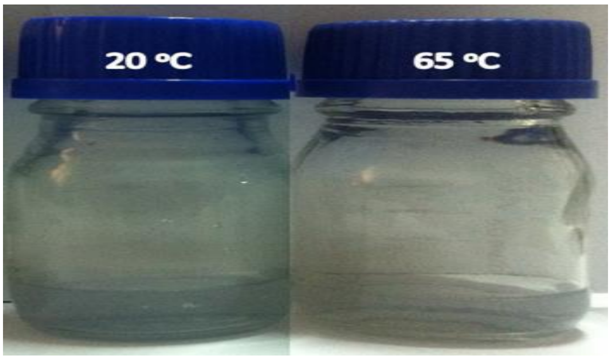



**Fig. 1:** Cadmium Oxide (CdO) Nanoparticles Solved in Human Cancer Cells, Tissues and Tumors.

To determine crystalline structure of nanoparticles, X–Ray Diffraction (XRD), model X'Pert MPD, Philips Co. was used. The suspension containing nanoparticles were gradually dried on a silicon substrate for performing XRD analysis. Optical properties of nanoparticle solution were tested by UV–Vis–NIR spectroscopy T80, PG Co. Size and distribution of other particles was measured using Dynamic Light Scattering (DLS) of MALVERN ZETASIZER ZEN3600. Transition Electron Microscopy (TEM) of Ziess–EM101C–80KV was used to determine sample micrographs. In order to investigate Attenuated Total Reflectance–Fourier Transform Infrared Spectroscopy (ATR–FTIR) spectroscopy of samples, Nexus 870 device was used.

## 3. Preparation of nanoparticles and alloy

In this test, nanoparticles were produced using a base pulse of synchrotron radiation of 7 (ns) with wavelength of 1064 (nm) and repeating frequency of 10 (Hz). In this regard, two plates of Gold and Cadmium were place into a container containing two–time deionized water. The volume of water was 20 (ml) and its height over target was 15 (mm). Synchrotron radiation emission with 6 (mm) diameter and energy density of 2 Joules per ($cm^2$) was focused on the target using a concave lens with focal length of 8 (cm). To produce various samples of nanoparticles, 5000 synchrotron radiation pulses were used. Figure (1) shows nanoparticle solution, its mixture and alloy.

In the next stage, 30 (mm) of each Gold and Cadmium Oxide (CdO) solutions were mixed in a container and were subjected to 15000 pulses of synchrotron radiation with wavelength of 532 (nm) and pulse width of 7 (ns). In this stage, repeating frequency of synchrotron radiation pulse was 10 (Hz) and nanoparticles mixture was subjected to synchrotron radiation emission of [6] (mm) diameter with energy density of 2 Joules per ($cm^2$).

To measure absorption spectrum of particles in the range of UV–Vis–NIR, a PG T80 spectroscope was used. X–Ray Diffraction (XRD) of samples was measured using XRD device, model X'pert, Philips. To measure hydrodynamic size of nanoparticles, Dynamic Light Scattering (DLS) ZEN3600 was used. Further, the morphology of particles was determined using SEM XL30, Philips.

## 4. Results and discussion

Figure (2) shows absorption spectrum of nanoparticles between 200–1100 (nm). Regarding in semi–conductivity of Cadmium Oxide (CdO), oxytocin absorption peak of samples is emerged at about 200 (nm). The presence of this peak confirms the presence of Cadmium nanoparticles in the human cancer cells, tissues and tumors. Higher absorption of sample (1) than (2) is due to the fact that the number of nanoparticles created in sample (1) is more than in sample (2). In other words, ablation rate by synchrotron radiation pulse from Cadmium target in the human cancer cells, tissues and tumors decreased as temperature of human cancer cells, tissues and tumors increased. It can be confirmed by the color of samples; as can be seen in Figure (1), the sample produced at 20°C is darker than the sample produced at 65°C which may be due to higher number of nanoparticles at 20°C. The presence of oxytocin peak related to Cadmium Oxide (CdO) has been reported in previous papers (Heidari 2016).

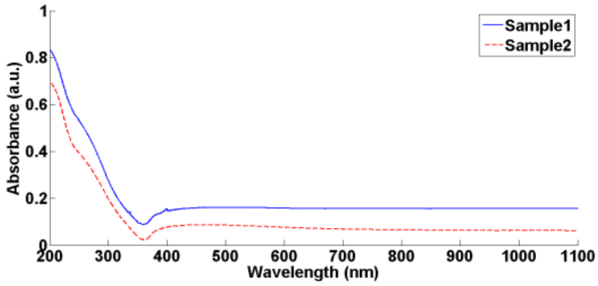



**Fig. 2:** Absorption Spectra for Nanoparticles of Cadmium Oxide (CdO) in Human Cancer Cells, Tissues and Tumors Produced at 20°C and 65°C.

Based on extrapolation of curves, the beginning of absorption peaks for samples (1) and (2) are at 358 and 361 (nm), respectively, which are equivalent to 3.46 and 3.43 (eV), respectively. Increasing wavelength (red shift) at the beginning of peaks in absorption curves confirm higher nanoparticle size in sample (2) than (1).

Figure (3) shows XRD spectrum of nanoparticles. To prepare this spectrum, some part of solution was dried over silicon, at first, and then, XRD spectrum was derived. The peak related to silicon is emerged at 69 degrees, as can be seen in the figure. In this figure, XRD spectra for Cadmium Oxide (CdO) nanoparticles at 20°C and 65°C can be observed.

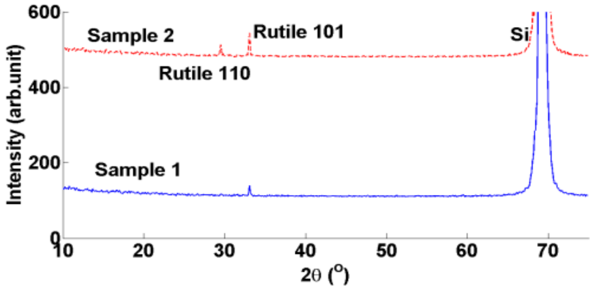



**Fig. 3:** XRD Spectrum of Cadmium Oxide (CdO) Nanoparticles.

In XRD spectrum of the sample produced at 20°C, a weak peak emerges at 33 degrees which is due to the presence of rutile structure of Cadmium Oxide (CdO) related to the plane (110) in the nanoparticles. In addition, at 65°C, another peak, in addition to the peak at 33 degrees, can be observed related to rutile structure of Cadmium Oxide (CdO) related to plane (101) in these nanoparticles. Regarding the intensity of these peaks at both samples, it can be seen that the crystalline intensity of nanoparticles produced at 65°C is higher than at 20°C.

TEM images of Cadmium Oxide (CdO) nanoparticles are shown in Figure (4). As can be seen, the form of Cadmium Oxide (CdO) nanoparticles is spherical and the size of nanoparticles increases with increase in temperature. This is in good agreement with the results obtained from absorption spectrum. Another result obtained from TEM images is that increase in temperature of ablation environment leads to increase in cohesion of nanoparticles.

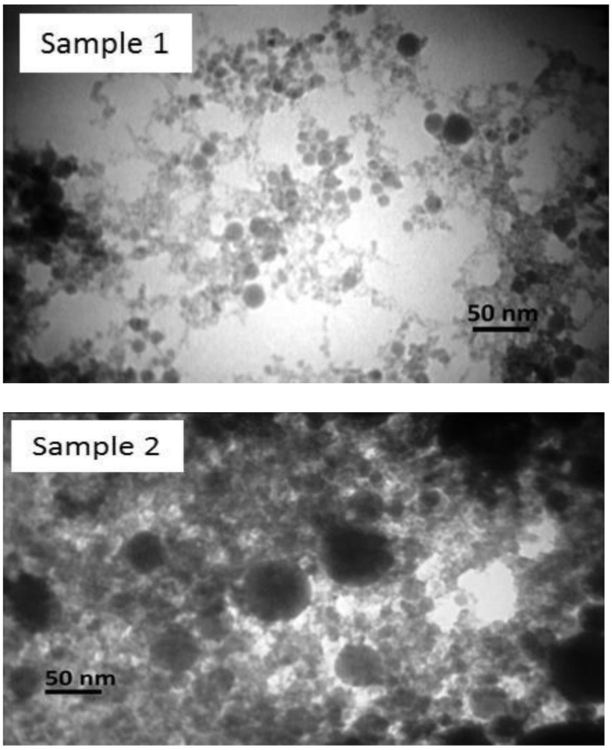



**Fig. 4:** TEM Images of Cadmium Oxide (CdO) Nanoparticles.

Regarding the TEM images, size of Cadmium Oxide (CdO) nanoparticles produced at 20°C and 65°C are 18 and 22 (nm), respectively, which is the variation of nanoparticle size against the temperature of environment such as the obtained resulted from DLS analysis. In Figure (5), size distribution of Cadmium Oxide (CdO) nanoparticles obtained from DLS analysis are shown. The results obtained from DLS are considerably larger than that are obtained from TEM images, which is due to creation of Hydrogen bonds between Carboxyl groups over the adjacent surfaces, and as a result, nanoparticle size in DLS is larger than TEM.

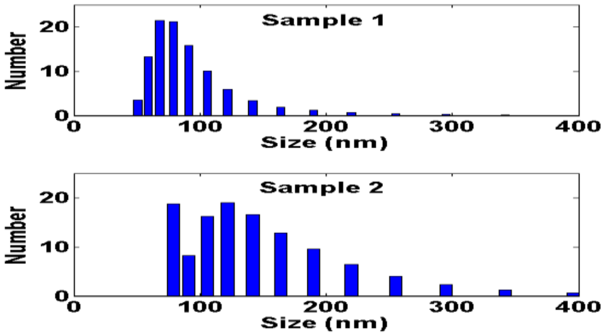



**Fig. 5:** Size Distribution Curve of Nanoparticles Obtained from DLS Analysis.

ATR–FTIR spectra for Cadmium Oxide (CdO) nanoparticles produced in distilled water are shown in Figure (6).

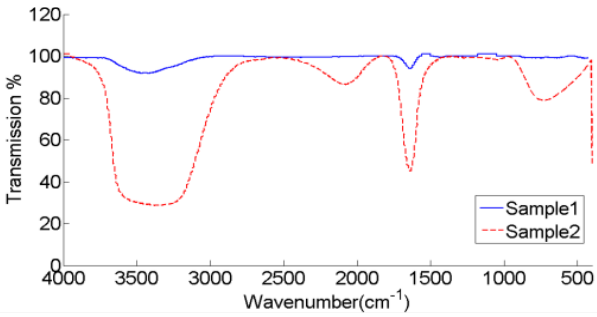



**Fig. 6:** ATR–FTIR Spectrum of Cadmium Oxide (CdO) Produced in Human Cancer Cells, Tissues and Tumors.

In ATR–FTIR spectra for samples, several absorption peaks related to OH group are emerged. In CdO samples synthesized in the human cancer cells, tissues and tumors, a wide peak can be observed at about 3000–3800 ($cm^{-1}$) that is related to tension mode of Hydroxyl (OH) induced by the water as the environment of ablation. Another peak can be observed about 1600–1630 ($cm^{-1}$) which is related to rotation mode of O–H in the water. As $CO_2$ molecules are presented in the air, absorption peak at about 2360–2390 ($cm^{-1}$) can be seen in ATR–FTIR spectrum. The peak at about 450–800 ($cm^{-1}$) is related to tension band of O–Cd. In ATR–FTIR spectrum of samples, it can be seen that the absorption intensity at about 450–800 ($cm^{-1}$) related to creation of CdO nanoparticles increases as the temperature of ablation environment increases. By warming the human cancer cells, tissues and tumors, the number and variety of Oxygen and its radicals increase in the environment and it leads to emerging new Oxygen bonds in liquid environment as ATR–FTIR peak at 2360 ($cm^{-1}$) is due to them.

Figure (7) shows the image of nanoparticles. Gold nanoparticles solution with purple color is a sign of existence of Gold nanoparticles with the size of 30 (nm). In addition, Cadmium Oxide (CdO) solution is colorless. Transparency of the color of nanoparticles mixture after emission of synchrotron radiation with 532 (nm) wavelength indicates that the size of nanoparticles, especially Gold nanoparticles, vary in the solution which it can be attributed to the formation of alloy. Also, Figure (8) shows UV–Vis–NIR spectra for samples.

**Fig. 7:** From Right to Left: Gold Nanoparticles, Cadmium Oxide (CdO), Its Mixture before Laser Emission with Wavelength of 532 (nm) and Their Alloy.

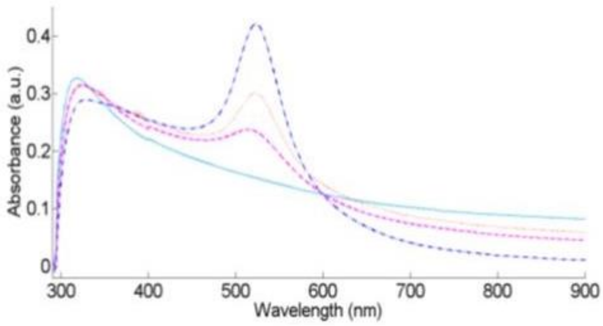



**Fig. 8:** UV–Vis–NIR Spectra for Samples.

This issue can be observed in absorption spectrum of nanoparticles. In this spectrum, Plasmon peak of Gold nanoparticles can be seen at about 523 (nm). In addition, exytoninc peak related to Cadmium Oxide (CdO) nanoparticles is formed at 318 (nm). After mixing these two nanoparticles, both peaks descend which indicates that their density in the solution reduces and after synchrotron radiation emission of 532 (nm), Plasmon peak of Gold nanoparticles more reduces while exytonic peak of Cadmium Oxide (CdO) does not changes. The location of Gold peak varies with a red shift of about 518 (nm) which indicates that Gold nanoparticles are enlarged in the compound. These processes have been reported in previous researches (Heidari 2016, 2017; Heidari and

Brown 2017; Bastogne 2017; Vanić et al. 2013; Islan et al. 2017; Bawarski et al. 2008; Eaton et al. 2011; Hadinoto et al. 2014; Svenson et al. 2011; Sosnik et al. 2013; Filipović-Grčić et al. 2013; Yu et al. 2015; Moghimi et al. 2013; Eliasof et al. 2010; Domingo et al. 2012; Samadder et al. 2016; Yen et al. 2010; Azmi et al. 2016; L et al. 2015; Liu et al. 2012; Gabellieri et al. 2011; Frederickson 2016 et al; Namdari et al. 2017; Kiew et al. 2015; Moghimi et al. 2012; Gil et al. 2010; Rzigalinski et al. 2009; Fako et al. 2009; Sainz et al. 2015; Duncan et al. 2010; Zhou et al. 2014; Wibroe et al. 2016; Nguyen et al. 2016; Beija et al. 2012; Vaishali et al. 2017; Bawa 2009; Marianecci et al. 2016; Patil et al. 2017; Fonseca et al. 2014; Bedi et al. 2011; Canal et al. 2011; Hügel et al. 2014; Donaldson 2012; Bose et al. 2016; Hall et al. 2017; Storm 2012; du Toit et al. 2010; Kumar et al. 2016; Rajabi et al. 2016; Andersen et al. 2012; Kabanov et al. 2011; Nagy et al. 2015; Nickols-Richardson et al. 2007; Gaspar et al. 2009; Bourlinos et al. 2012; Svenson et al. 2012; Sitterberg et al. 2010; Telford 2005). The reason for this variation is in fact the formation of Gold and Cadmium Oxide (CdO) alloy which leads to increase in amplitude of vibrations of surface electrons of Gold nanoparticles. Figure (9) shows XRD spectrum of nanoparticles. To produce these spectra, some part of solution was dried over the silicon and then, spectroscopy was performed. The peak related to silicon is emerged at 69 degrees that is removed from the curves. In this figure, XRD spectra for Gold and Cadmium Oxide (CdO) nanoparticles are shown along with those related to their mixture and alloy. A weak peak is emerged at 33 degrees in XRD spectrum of Cadmium Oxide (CdO) sample which is due to the presence of rutile structure in these nanoparticles. Moreover, three major peaks related to Gold (III) Oxide ($Au_2O_3$) and Gold at 27, 33 and 38 degrees, respectively, can be seen in XRD spectrum of Gold nanoparticles. After mixing them, the peaks related to Gold are reduced in XRD spectrum. In this spectrum, the peak related to Cadmium Oxide (CdO) and Gold nanoparticles are overlapped in 33 degrees. Reduction of the peak indicates that the amounts of Gold nanoparticles are reduced in the solution. As can be seen, the peak related to Gold (III) Oxide ($Au_2O_3$) at 27 degrees is disappeared after synchrotron radiation emission of 532 (nm). Separation of Oxygen atoms from metal atoms such as Gold is due to melting of these metal nanoparticles during synchrotron radiation emission of 532 (nm). The common peak of Gold and Cadmium Oxide (CdO) nanoparticles at 33 degrees are considerably enhanced relative to mixture before synchrotron radiation emission which indicates that Gold and Cadmium alloy structure is formed and a new peak related to rutile phase of Cadmium Oxide (CdO) nanoparticles at 29 degrees is emerged which indicates that lattice of these nanoparticles are changed due to synchrotron radiaiton emission of 532 (nm). Similar results have been reported in previous researches (Alibolandi et al. 2015; Bridoux et al. 2009; Stuurman et al. 2010; Kondo 2010; Jindal et al. 2017; Rapoport 2007; Fernández 2011; Pippa et al. 2013; Verreault et al. 2012; Hassanzadeh et al. 2017; Sivanesan et al. 2017; Phillips et al. 2010; Varan et al. 2017; Moghimi et al. 2014; Soria et al. 2010; McMurray et al. 2010; Sans-Serramitjana et al. 2016; Rigo et al. 2017; Alibolandi et al. 2017; Bridoux et al. 2010; Tutaj et al. 2016; Kuppusamy et al. 2013; Tomalia 2006; Menjoge et al. 2010; Vega-Villa et al. 2008; Gaur et al. 2014; Tietze et al. 2015; Schwengber et al. 2015; Adhikari et al. 2017; Szebeni et al. 2015; Chen et al. 2011; Requejo-Aguilar et al. 2017; Golyshkin et al. 2016; Szulc et al. 2016; Haddad et al. 2008; Mignani et al. 2013; Eaton et al. 2015; Lollo et al. 2015; Thompson et al. 2012; Muntimadugu et al. 2017; Foldvari et al. 2008; Riley et al. 2012; Fernandes et al. 2015; Mehra et al. 2016; Mignani et al. 2016; Naderkhani et al. 2014; Newton 2013; Aoki et al. 2015; Ita 2014; Liu et al. 2008; Mallapragada et al. 2015; Peres et al. 2017; Ferreira et al. 2013; Salerno et al. 2015; Tyler et al. 2016; Iannazzo et al. 2015; Jemec et al. 2012; Chen et al. 2012; Lütscher et al. 2012; Park et al. 2013; Huang et al. 2011; Depan et al. 2011; Guo et al. 2014; Duncan 2011; Sidik et al. 2016; Yuan et al. 2010; He et al. 2014; An et al. 2013; Meenach et al. 2013; England et al. 2012; Pippa et al. 2013; Boisseau et al. 2011; Petrichenko et al. 2015; Rodríguez-Gascón et al. 2015; Frima et al. 2012; Yallapu et al. 2015; Duan et al. 2012; Perez et al. 2015; Costantino et al. 2012; Wei et al. 2006; Murday et al. 2009; Dixit et al. 2015; Nair et al. 2010; Bawa et al. 2005; Farkhani et al. 2014; Lal et al. 2010; Hacklin et al. 2009; Gabizon et al. 2016; Zhang et al. 2013; Vanić et al. 2014; Ellis-Behnke 2007; Srivalli et al. 2016; Collnot et al. 2012; Rychak et al. 2006; Watala et al. 2016; Palombo et al. 2009; Kuzmov et al. 2015; Diebold et al. 2010; Bal et al. 2011; Bharali et al. 2010; Ray et al. 2010; Mishra et al. 2010; Torchilin 2009; Cupaioli et al. 2014; Sosnik et al. 2014; Guan et al. 2013; Toit et al. 2013; Zhang et al. 2012; Muthaiyan et al. 2011; Duncan 2009; Palao-Suay et al. 2016; Morrow et al. 2007; Punetha et al. 2017; Manickam 2017; Osorio et al. 2015; Karami et al. 2016; Park et al. 2013; Heidari 2017, 2018; Gobato et al. 2018; Gobato, Heidari 2018; Heidari, Gobato 2018).

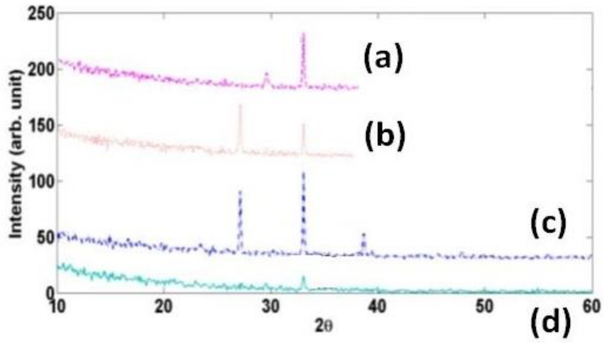



**Fig. 9:** XRD Spectra of Cadmium Oxide (CdO) Nanoparticles (a) before and (b) after Synchrotron Radiation and Gold Nanoparticles (a) before and (b) after Synchrotron Radiation Along with their Mixture before and after Synchrotron Radiation Emission with Wavelength of 532 (nm).

Figure (10) shows size distribution curves for nanoparticles measured by DLS technique. It should be noted that this mechanism shows hydrodynamic size of nanoparticles that is larger than real size of them but is proportional to real size. Hydrodynamic size is in fact the diameter of particle plus the diameter of electrostatic potential form adjacent to the particle. Figure (10) consists of four size distribution curves. In Figure 10 (a), Cadmium Oxide (CdO) size distribution is shown in which the peak is at 110 (nm). In Figure 10 (b), size distribution of Gold nanoparticles is shown which its peak is at 30 (nm). Size of particles before synchrotron radiation emission is shown in the third graph. As can be seen, Gold nanoparticles are frequent in the mixture and the peak size is approximately with the similar intensity that indicates larger number of these nanoparticles in the solution. However, after synchrotron radiation emission of 532 (nm), size of samples is completely changed in the solution. Nanoparticles with 30–40 (nm) size increase up to 80–100 (nm) and it is due to formation of alloy.

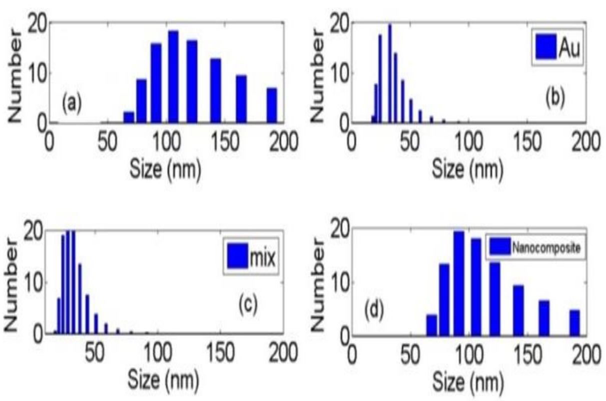
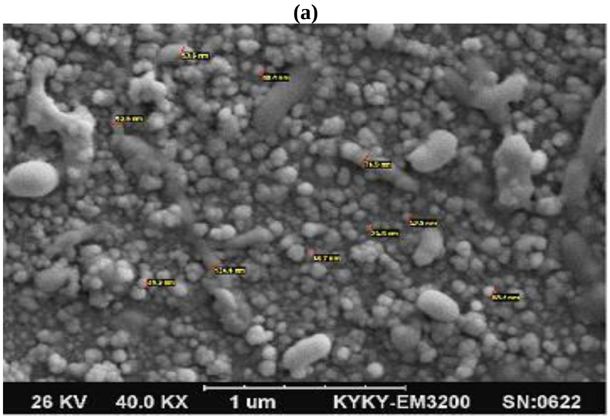
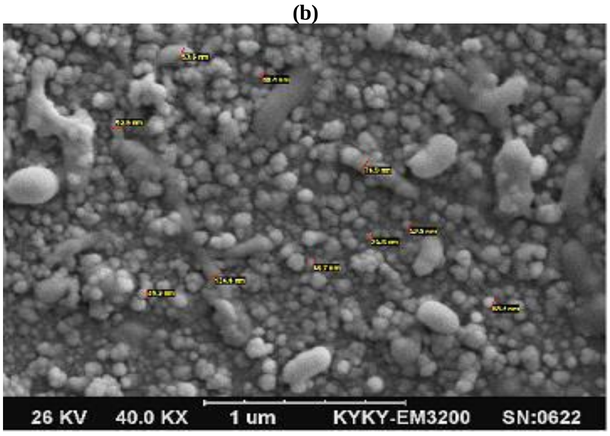



**Fig. 10:** Size Distribution Curve of Nanoparticles (a) Cadmium Oxide (CdO), (b) Gold, (c) Mixture of Gold and Cadmium Oxide (CdO) Nanoparticles before Synchrotron Radiation Emission and (d) Nanocomposite (Alloy) of Gold and Cadmium Oxide (CdO) Nanoparticles.

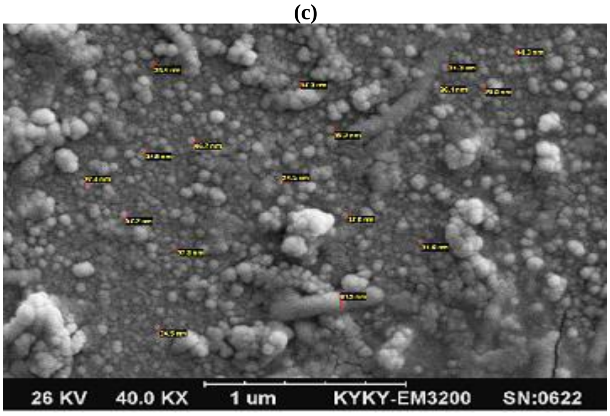
(a)




(b)




(c)




(d)

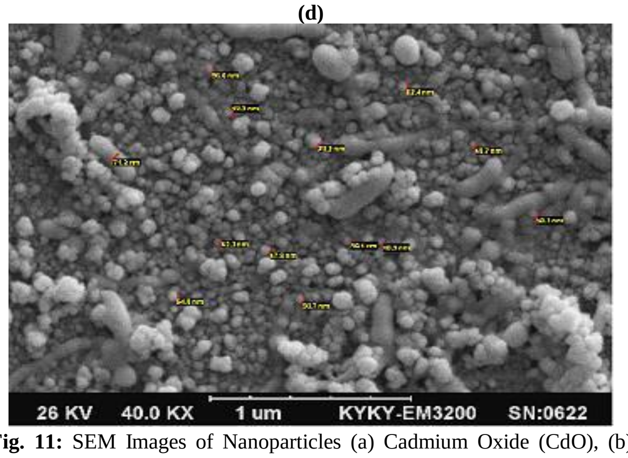



**Fig. 11:** SEM Images of Nanoparticles (a) Cadmium Oxide (CdO), (b) Gold, (c) Their Mixture and (d) Their Alloy Compound.

Figure (11) shows SEM images of nanoparticles. These images are taken with 26 (keV) electrons and 40000-x zoom. Figure 11 (a) shows Cadmium Oxide (CdO) nanoparticles that some parts of them are spherical and fine and another part are ellipsoidal. Fine spherical particles are about 40–50 (nm) and coarse ellipsoidal ones are 80–100 (nm). Figure 11 (b) shows Gold nanoparticles images which are mostly spherical and are about 30–40 (nm). Their cohesion is very lower than Cadmium Oxide (CdO) nanoparticles which are a characteristic of noble metal nanoparticles. Figure 11 (c) shows the images for the mixture. As Gold nanoparticles and most of Cadmium Oxide (CdO) nanoparticles are spherical, this figure mainly consists of spherical particles. There is more cohesion between some of them which are Cadmium Oxide (CdO) nanoparticles. The image of mixture after synchrotron radiaiton emission is shown in Figure 11 (d). Spherical nanoparticles with larger size than previous state can be seen in this figure. The presence of Gold and Cadmium Oxide (CdO) alloy can be attributed to this. The presence of more ellipsoidal particles is a sign of reduction in Gold nanoparticles and formation of alloy in this sample.

## 5. Conclusion

The experimental results show that nanoparticle sizes increase as the temperature of ablation environment increases. It can be attributed to variation of water density. When synchrotron radiation incidents to the target, the atoms of target separate from each other and plasma creates on the surface of target. Expansion of the plasma in liquid environment leads to distribution of shock waves toward the environment. The separated atoms from target are again bond together as a result of the pressure of these waves and create nanoparticles. The results show that nanoparticle size depends on the pressure and higher pressure leads to smaller nanoparticles. In the tests, increasing the temperature of human cancer cells, tissues and tumors reduces the water density and leads to decrease in pressure in plasma environment. This reduction leads to increase in size of nanoparticles. The effect of temperature of liquid environment on the nanoparticles has been observed in previous papers.

Furthermore, in the current experimental research, a 532 (nm) synchrotron radiation pulse was used to melt Gold nanoparticles in the mixture of Gold (Au)–Cadmium Oxide (CdO) in the human cancer cells, tissues and tumors and to create an alloy (nanocomposite) from this mixture. The point used in this process is Plasmon resonance absorption peak of Gold nanoparticles at the vicinity of 532 (nm) which leads to absorption a large part of energy of synchrotron radiation by these nanoparticles which is the key point in production of the alloy. In addition, formation of alloy and its characteristics are observed by various devices.

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