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Research Article

Synthesis of Gold Nanoparticle in the form of Paper Willow by Hot Chemical Reduction Method

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Abstract

Colloidal gold nanoparticles (ringworm Palm or in the form of paper willow) have been prepared from $HAuCl_4$ containing aqueous solution by hot chemical reduction method. The colloidal gold nanoparticles were characterized by SEM, EDX, and UV-VIS absorption spectroscopy. It was found that the variation of reduction time from boiling point affects the size of the nanoparticles and also in chemical reduction approach the size of nanoparticles can be controlled by varying the amount of variation the volume of reductant material with respect to the volume of $HAuCL_4$.

Keywords: *Gold nanoparticles, chemical reduction method.*

1. Introduction

Gold nanoparticles (G NPs) have enjoyed increasing interest because of a wide range of their potential applications in such fields as: medicine, biotechnology, catalysis, preparation of nanocomposites, nanosensing and electronics. The potential applications of G NPs depend on their size and morphology. Especially the potential application of G NPs in nanosensing is strongly connected with controlling their size and morphology by the proper choice of preparation conditions (Sedeno *et al*, 2005), (Zeng, *et al*, 2011), (Zheng, *et al*, 2006). Gold nanospheres have a characteristic red color, but anisotropic gold nanorods have a dramatically changed color. More recent treatments have shown that the color is due to the collective oscillation of the electrons in the conduction band, known as the surface plasmon oscillation (Marzan, 2004).

There are a few general methods for the synthesis of gold nanoparticles. Some of these methods can be listed such as sol-gel (Al-Awadi, 2006), laser ablation method (Ali, 2010), Reverse micelles process (Lin, *et al*, 2001), In situ method (Joly , *et al*, 2000), Ultrasonic method (Suslick, 1990), Electron irradiation process (Chen, *et al*, 1999), Thermal decomposition process (Suslick, *et al*, 1986), Green synthesis route (Bogle, *et al*, 2004) and Chemical reduction method.

The chemical reduction of metal ions is the most common method of G NPs synthesis. This method consists in precipitation of gold nanoparticles from a solution of a dissolved gold precursor under the in_uence of a reducing agent. A gold precursor usually is tetrachloroauric acid ($HAuCl_4$) while a reducing agent could be sodium citrate, sodium boron hydride, block copolymers or ascorbic acid. The reduction of gold precursor with sodium citrate is the simplest and most reliable method of obtaining a solution of monodisperse gold nanoparticles (Murawska, et al, 2012).

2. Experimental part

2.1 Chemical Materials

The following materials were used:

Gold hydrochloric acid $HAuCl_4$ 339.78 (g/mol) of purity 99.9% (supplied by **Philip Harris chemical company U.K)**, Ethanol **C_2H_5OH 46.07 (g/Mol)** of purity 99.9% (supplied by Gainland chemical company U.K.), distilled water, trisodium citrate $C_6H_5O_7Na_3$ **258.06 (g/Mol)** (Sigma Aldrich, UK) of analytical grade purity, were used as starting materials without further purification.

2.2 Gold NP synthesis

At first a solution of ~5.0 x 10^{-3}M $HAuCl_4$ had been made in water. (0.1699 g $HAuCl_4$ in 100mL deionized H_2O) & dilluted it by water, then Made a solution of 0.5% sodium citrate (0.25g in 50mL of H_2O). Then started to work as the procedure below to synthesis gold nanoparticles by heating the diluted solution of $HAuCl_4$ until it begins to boil then added 1mL of 0.5%

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sodium citrate solution, as soon as boiling commences. Heating process had been continued until color change is evident (pale purple). After 5 minutes the solution had been removed from the heating element and continued to stir until it was cooled to room temperature. All the above procedure have been repeated by variation the reduction time from 5 minutes to 15 minutes and we repeated the similar procedures by variation the reductant (sodium citrate) volume 1, 2, 3 ml. Finally, many testing has been performed to it.

2.3 Analysis technique

The topography of the prepared films was studied by field emission scanning electron microscopes (SEM) type ULTRA 55 and SUPRA 55 with different magnification powers and detectors. The optical properties (absorbance) of colloidal were evaluated with UV/VIS/NIR spectrophotometer (Metertech UV-Vis SP8001), light source – combined deuterium-halogen, wavelength range: 200 nm –1100 nm.

3. Results and Discussion

At first we try to introduce the picture of ours obtained G NPs and compared it with global samples, figure (1) shows the colloidal of G NPs which is prepared by hot chemical reduction method. It is clear from the image that the color of colloidal is red which is the main indicator to G NPs creations so it is agreed with the global results.

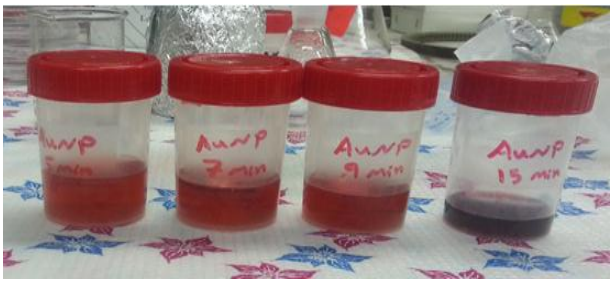

Fig. 1 The Gold Nanoparticles which is prepared by chemica reduction method by variation the reduction time from adding point**.**

Fig. 2 The G NPs which is prepared by chemical reduction method in global lab (Bahadory , 2008).

3.1 SEM Analysis

Synthesized gold nanoparticles were analyzed by the SEM unit to estimate the particle size of the gold nanoparticles, which is prepared by hot chemical reduction method. By variation the reduction time from boiling point, then looking carefully for the SEM images it is possible to note that G NPs gathered on form of ringworm Palm or in the form of paper willow.

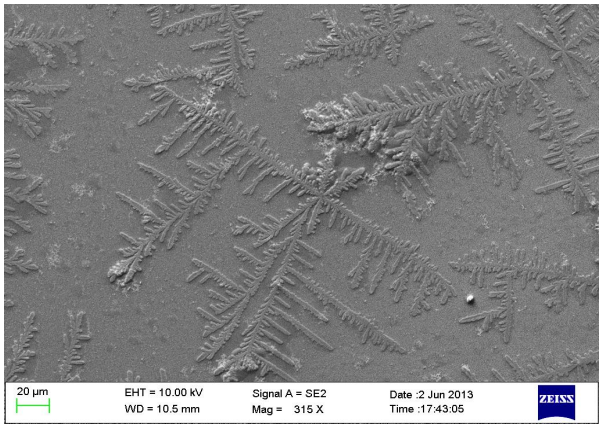

Fig. 3 Show the SEM image with magnification force = 315 x of G NPs which had been prepared using 5 minutes as a reduction period.

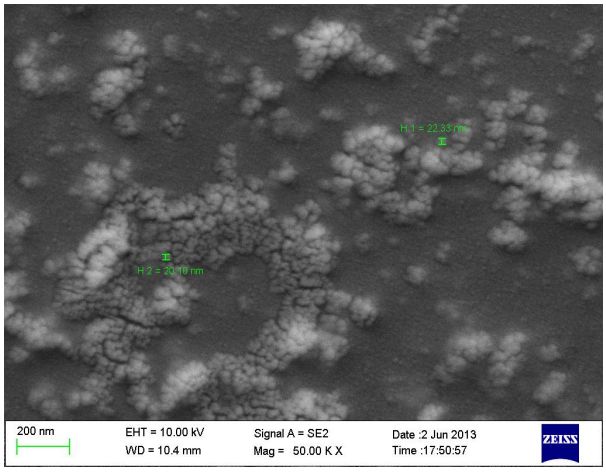

Fig.4 Show the SEM image with magnification force = 50 kx of G NPs which had been prepared using 5 minutes as a reduction period.

Then by getting a more focused magnification force it is possible to see that the size of particles and also sticky on board the picture is up 20.10 nm to 22.33 nm with uniform particles distributed inside the gold clusters that for the case of 5 minutes as a reduction time [as shown in figure (3) and figure (4)], the same thing for the case of 7 minutes whereas the particle size is about 20.10 nm to 22.35 nm [as shown in figure (5) and figure (6)]. Finally, in the case of 15 minute the particle size was about 22.33 nm to 26.80 nm with uniform particles distribution [as shown in figure (7) and figure (8)]. Taking a glance at the photos leads to the result that the G NPs tend to congregate in the form of

ringworm or willow paper and the spread of particles within this assembly be a regular and the particle size is almost constant within the range of from 20 nm to 30 nm. All these discussions relate to the following pictures.

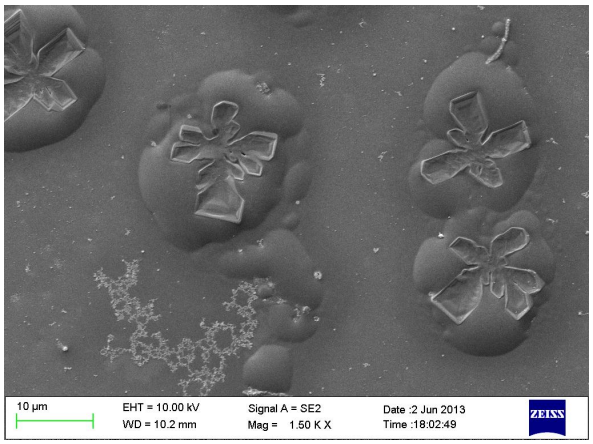

Fig.5 Show the SEM image with magnification force = 1.5 kx of G NPs which had been prepared using 7 minutes as a reduction period.

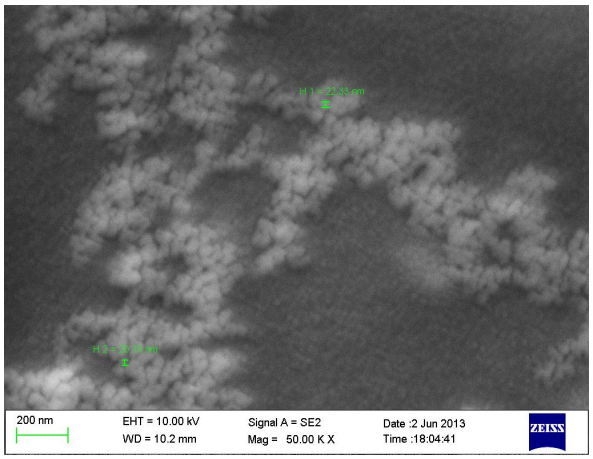

Fig.6 Show the SEM image with magnification force = 50 kx of G NPs which had been prepared using 7 minutes as a reduction period.

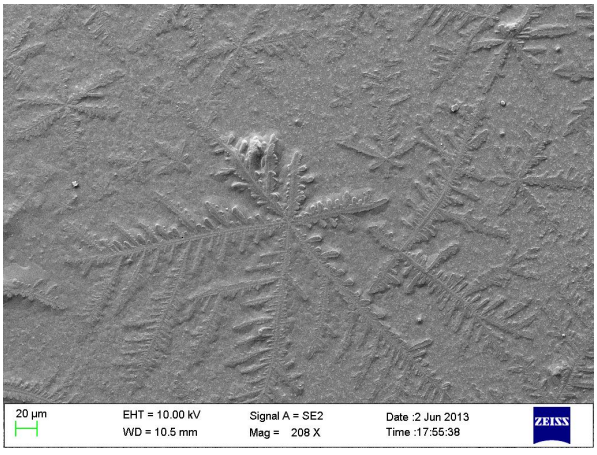

Fig.7 Show the SEM image with magnification force = 208 kx of G NPs which had been prepared using 15 minutes as a reduction period

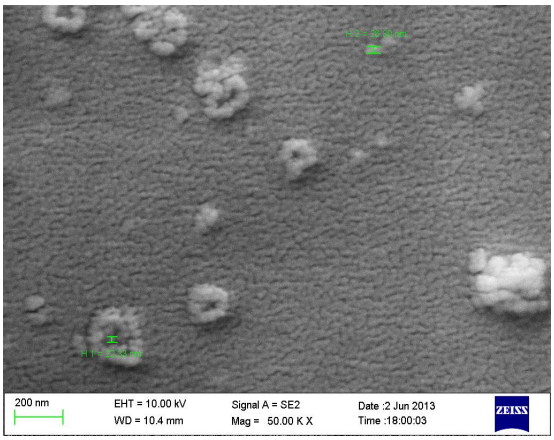

Fig.8 Show the SEM image with magnification force = 50 kx of G NPs which had been prepared using 15 minutes as a reduction period.

All these result's will be list in table (1)

Table (1): The G NPs particle size as a function to reduction time

Reduction time (minutes)	The rang of Particle size (nm)
5	20.10 - 22.33
7	20.10 - 22.35
15	22.33 - 26.80

Now the effect of varying the volume of reductant material with respect to the volume of $HAuCL_4$ will be shown and discussed. If one look carefully to the photos one can observe that at the case of 1 ml [see figure (9)] a uniform distribution was obtained with a stable particle size from 20 nm to 30 nm, while at the case of 2 ml as a reductant material aggregations may be formed and nonuniform distribution in terms of particle because a larger particle size was obtained if it compared with the case of 1 ml [see figure (10)].Finally at the case of 3 ml a non uniform distribution was obtained with largest particle size if it compared with the previous two cases [see figure (11)].

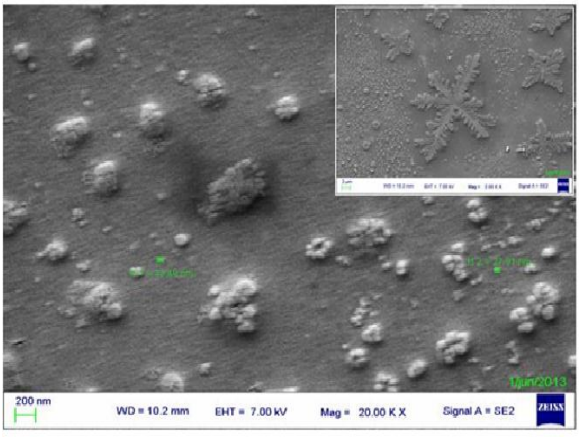

Fig.9 Show the SEM image with magnification force = 20 kx(2kx for the image at the top corner) of G NPs which had been prepared using 1ml as a reductant volume to 18 ml from $HAuCL_4$.

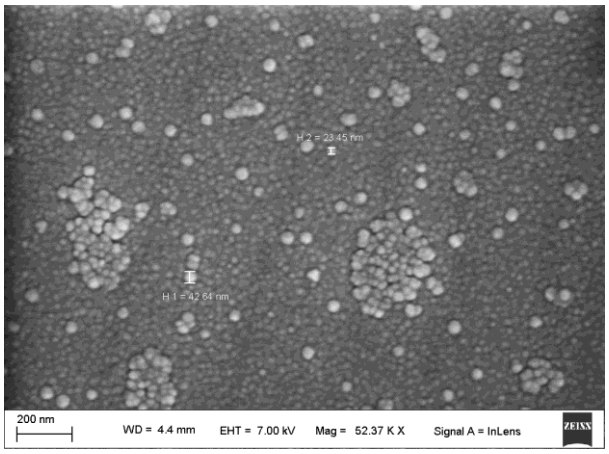

Fig.10 Show the SEM image with magnification force = 52.37 kx of G NPs which had been prepared using 2ml as a reductant volume to 18 ml from $HAuCL_4$.

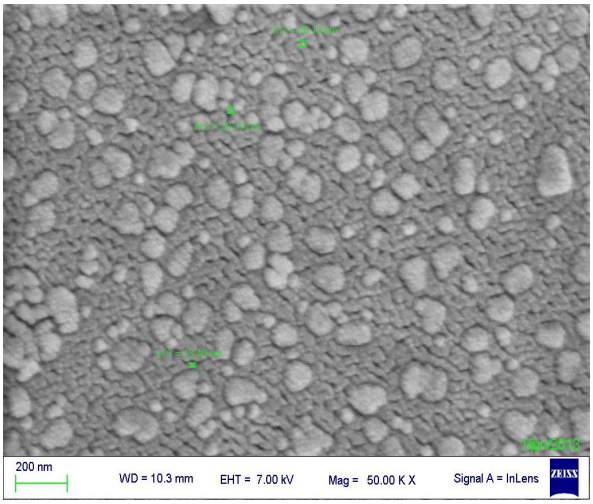

Fig.11 Show the SEM image with magnification force = 50kx of G NPs which had been prepared using 3ml as a reductant volume to 18 ml from $HAuCL_4$.

3.2 Energy-dispersive X-ray spectroscopy *(EDX) results*

Figure (12) shows the EDX results for G NPs. From the EDX spectrum one can prove the existence of gold with 100% present age .

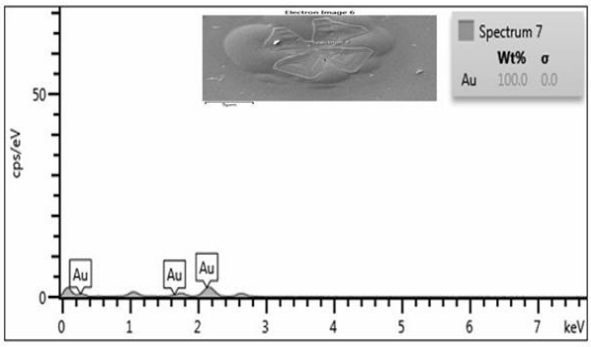

Fig.12 Show the EDX result of G NPs which had been prepared by hot chemical reduction method.

3.3 UV–visible absorption spectrum

In this section, the surface plasmon of G NPs which has been prepared by hot chemical reduction method by adding Sodium citrate to $HAuCl_4$ at boiling point has been studied . Many global researches around the world consider the peak absorbance position and the shape of surface plasmon is the basic mark of the formation of gold nanoparticles. Figure (13) shows the surface plasmon of G NPs which is prepared by adding 1 ml from sodium citrate to $HAuCl_4$ one can notice that the maximum peak absorbance occurred at 515 nm and the shape of the curve is at a similar to the G NPs curves which have been prepared in global laboratories such as in Drexel university.

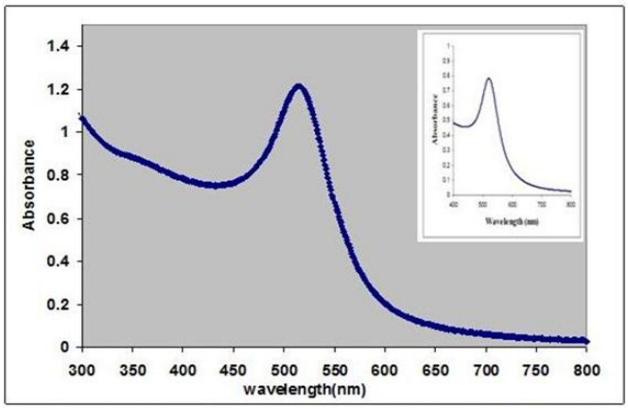

Fig.13 The peak absorption of surface Plasmon of G NPs which had prepared by hot hot chemical reduction method (In the top corner the surface Plasmon of G NPs which had prepared by hot hot chemical reduction method in Drexel university (Drexler, 1986).

Figure (14) shows the effect of varying the reduction period on surface plasmon spectra of G NPs. From the first sight to these curves one can observe that the maximum peak absorption was varieties between 517 - 520 nm and the maximum peak intensity was variated from 0.93 to 1.2 in arbitrary unit.

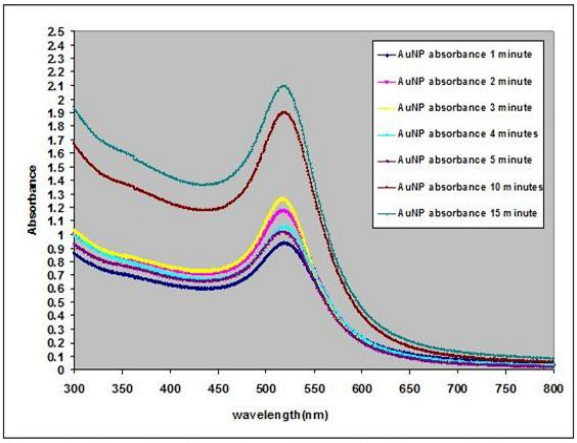

Fig.14 Show the total peak absorbance of G NPs for different reduction periods.

Through careful consideration to figure (15) one can notice that the behavior is somewhat unregularly from 1-5 minutes as a reduction time except in the case of the 10 and 15-minutes there is a clear behavior of increasing absorption intensity with increasing the reduction period and the maximum peak intensity was about 520 nm.This increasing in absorption intensity may be due to increasing the concentration of G NPs in colloidal with increasing the reduction period and this behavior agrees with the beer lamberts law. All these behaviors are illustrated in figure (14) & (15) and in table (2).

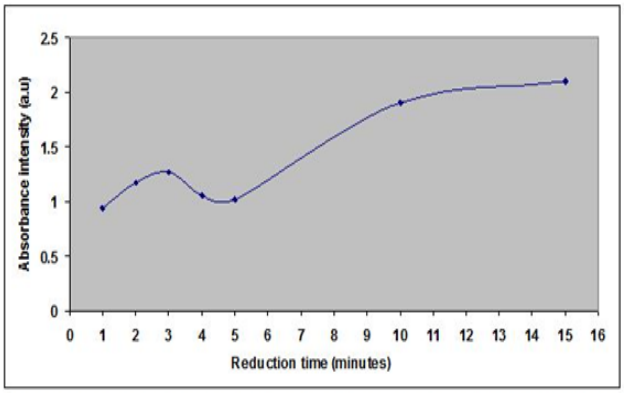

Fig.15 Show the absorbance intensity as a function to reduction period of G NPs which had been prepared by chemical reduction method.

Table (2): The values of peak position & peak absorbance intensity of G NPs which had been prepared by variation the reduction periods

Reduction time (minute)	Peak position (nm)	Absorbance intensity (a.u)
1	520	0.9347
2	519	1.1733
3	517	1.266
4	519	1.0553
5	517	1.0192
10	520	1.8996
15	518	2.1001

Now in this section, the effect of varying the sodium citrate amount which was added to $HAuCl_4$ in the point of view of peak position and peak intensity will be shown in briefly. Three mixing ratios from sodium citrate according to $HAuCl_4$ (1, 2 & 3 ml) were used. It was noticed that the peak absorbance at the case of 1 ml was occurred in 515nm, while at the case of 2 ml the peak absorbance was occurred at 519 nm and finally at the case of 3 ml the peak absorbance was occurred at 523 nm that leads to an important conclusion that when we increase the amount of reductant, the peak absorbance position will be gone toward long wavelengths red shift and increasing the amount of the reductant may lead to irregular distribution to the particle size as one can see at the results of SEM all previous discussions which is clear in figure (16).

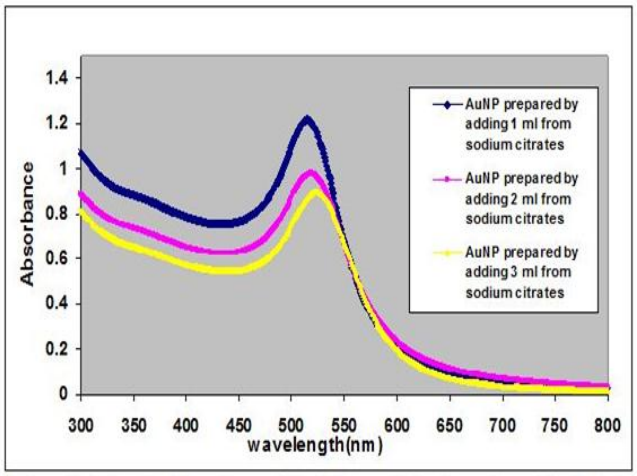

Fig.16 show the total absorbance of G NPs by variation the volume of reductant

Conclusions

A stable particles size with uniform distribution had been obtained at the case of 1 ml of sodium citrate as a reductant. When we increase the reduction time & the amount of the reductant the maximum peak of absorbance will be going toward long wavelength red shift and increasing the amount of reductant may be lead to irregular distribution to the particles size.

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