

Preparation of colloidal Gold Nano particles Using Electro

Chemical Method

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Abstract

In this paper a process of electro-chemical system used for preparing colloidal Nano Gold particles in electrolyte medium. Thin films of gold nano particles and suspension gold prepared and examined by x-ray diffraction method. furthermore; Uv-vis spectra spectrophotometer was used to determined optical properties . Results show the electro-chemical method is simple method to syntheses of colloidal Nano Gold particles with average particles less than of 50 nm .The fabrication parameters such as I(mA) , V(v) and T(C°)) were examined . Red to grey color changed of gold suspension that related to preparation parameters and particle size was studied.

key words : electro-chemical system , Nano Gold particles

تحضير عوالق جسيمات الذهب النانوية بطريقة كهروكيميائية

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وزارة العلوم والتكنولوجيا – مركز بحوث المواد – مركز المواد المتقدمة

الخلاصة:

في هذا البحث تم تحضير عوالق جسيمات الذهب النانوية بطريقة كهروكيميائية وباستخدام محلول الكتروليتي . حضرت أغشية من عوالق جسيمات الذهب النانوية وتم فحصها بتقنية (XRD) حيود الأشعة السينية . ثم فحصت عينات من الأغشية باستخدام تقنية مطياف الأشعة فوق البنفسجية وتمت مناقشتها وفق ظروف الإعداد . فحصت عينات من الأغشية مجهرياً ودرست ظروف تحضير عالق الذهب النانومتري من تيار كهربائي، الفولتية ودرجة الحرارة . أظهرت النتائج سهولة الطريقة المستخدمة في تحضير عوالق جسيمات الذهب النانوية بحدود حجم حبيبي أقل من (50 nm) . تم دراسة ظروف التحضير والتغير اللوني لعوالق الذهب من لونها الأحمر ثم تتدرجها إلى اللون الداكن العائد إلى ظروف التحضير ومتغيراته .

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Introduction

Colloids Gold has novel physical and chemical properties because of their distinctive size and shape. They are extensively used for many chemical reactions because of their exceptional catalytic properties⁽¹⁻³⁾. The electrochemical catalytic properties of colloids gold have led their wide spread use in a chemical science and technologies in the past decade⁽⁴⁻⁶⁾. Several technical are available for the preparing gold nanoparticles, with different size, shape, and UV-vis absorption spectra. Current techniques used to create gold nanoparticles are categorized into chemical and physical methods .Physical methods include UV⁽⁷⁾ (8),(9), and gamma radiation⁽¹¹⁾ and aerosol technology⁽¹²⁾. Unfortunately, although these methods can be used to successfully produce pure gold nanoparticle, they are relatively expensive to implement and are also create great risks to the environment concern.

In this work; nanogold colloidal particles were synthesized by electro chemical method using the two pure Au-steel rods immersed in electrolysis circuit . Colloidal gold (ionic nano gold) suspension was reduced to gold atoms by sodium citrate, and many of gold atoms accumulated into nanogold particles.

Experimental Part

The purpose of this study is to prepare colloidal gold nano particles from 0.99 purity fine gold wire, using an electrolytic process to make HAuCl_3 , and then to reduce the resultant gold chloride to colloidal gold in hot aqueous solution citrate itself and its oxidation products (e. g. acetone dicarboxylate) can act as protecting agents if no other stabilizer is used^[11]. To make the electrolyte solution, weigh 0.75 gram of NaCl and 3 drop of hydrogen peroxide added to the beaker, then, added 0.50 g /ml of stock Na-citrate solution in 300 ml of distilled water^[9].

Two Au – steel rode immersed in electrolytic solution and conducted to dc power supply (0-140) V; within (1/2-1)hr red tint solution turned to dark solution color changed detected then turn of the power supply and removed the electrodes. Top off the water to restore it to 300 ml, and then we have dark red colloidal gold, filtered and bottle.

At cathode, chloride ions travel to anode where they combine with gold, and at the anode , reaction established as equations below :



Gold is liberated as gold chloride as eq.(1) . Oxygen gas is liberated as hydroxyl ions are oxidizes; At the anode ; AuCl_3 reacts with Cl as eq.(2)^[3]:-

Redaction reaction:^[4] -

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Electrophoresis is a technique used to separate Au ions from other solution and examined by UV-vis spectroscopy during handling steps. Centrifuging process with 3000-13000 rpm where applied to gathering Au powder. Thin films and suspensions of colloidal gold were prepared for UV-vis spectroscopy, XRD analysis and AFM examination.

Results and Discussion

Colloidal Nano gold particles were synthesized by electro-chemical method using the reduction of HAuCl_3 to gold atoms by sodium citrate. Many of gold atoms accumulated into nano gold particles. While the spacing between the electrodes does not affect on the reactions, it does alter the amount of voltage needed from the power supply to produce a fixed amount of current. The closer the electrodes are, the lower the voltage needed. More electrode area requires less power supply voltage for the same current. In addition, temperature of preparation should be raised to (40-60) °C to ignite the reaction.

Fig (1,2) shows the effect of using peroxide drops on colloidal nano gold. According to Sherrie equation; crystal size approximately about 50nm⁽¹⁴⁾.

According to atomic absorption spectroscopy, colloidal gold concentration that prepared using electrochemical technique was 0.3 ppm before physical separation while it reduced to 0.24ppm after electrophoresis process.



Fig .(1,2) : Colloidal gold solutions and powder sample.

As the Au^{+3} ions enter solution, they migrate and pulled to the cathode because of the electric field between the electrodes. If the Au ions reach the cathode, they will be reduced back to metallic gold. By adding the reducing agent at the start of electrolysis, the Au ions have a high probability of finding a molecule of the reducing agent before reaching the cathode. When this happens, the ionic charge is neutralized, and free metallic gold appears in the optical images (Nikon DIGITAL CAMERA-DXM 1200F ,Japan)as in fig.(3a,b).

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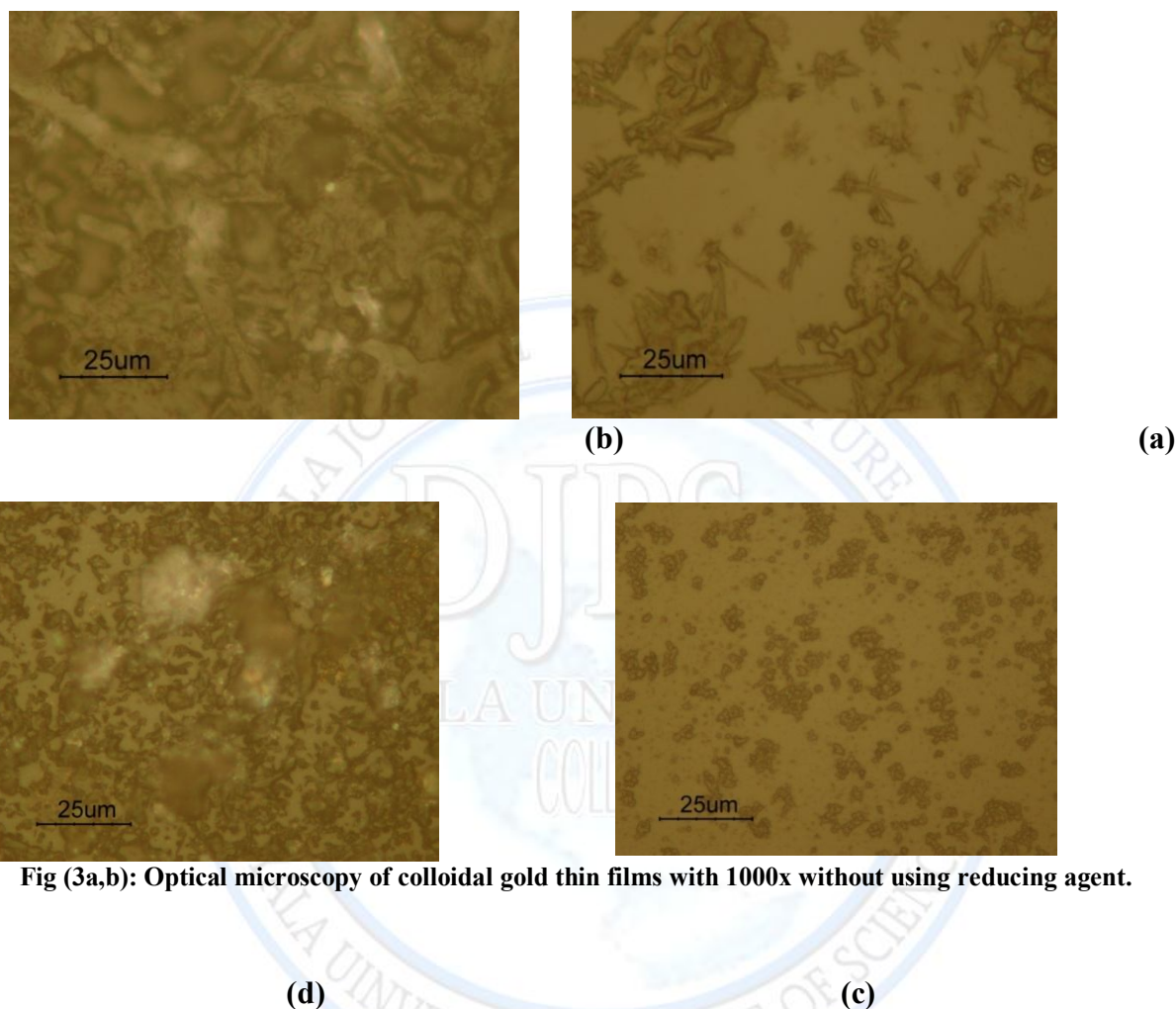


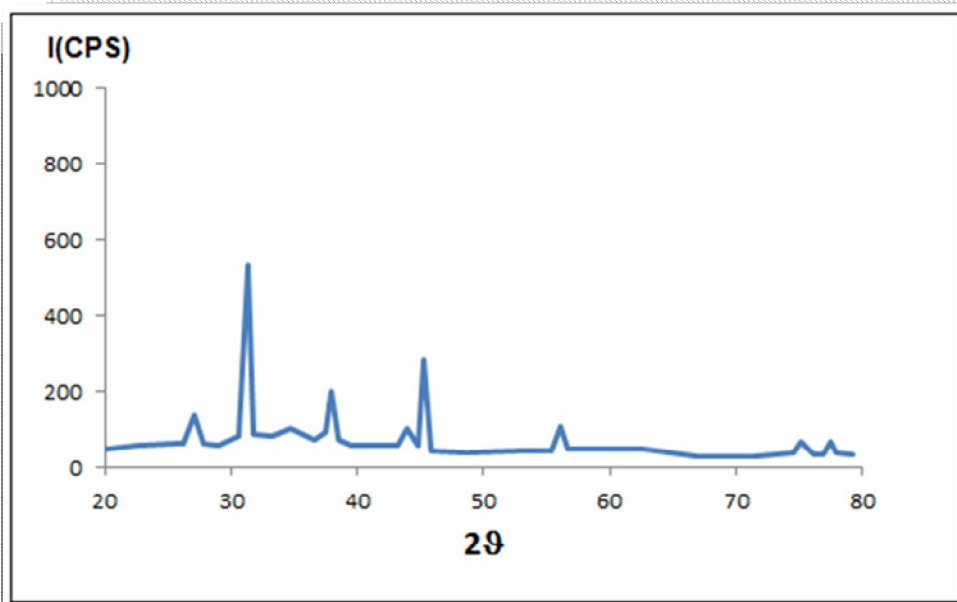
Fig (3a,b): Optical microscopy of colloidal gold thin films with 1000x without using reducing agent.

Fig(4d) : Optical microscope image of colloidal gold thin film using reducing agent with 1000x magnification. Fig.(4c) using reducing agent and present filtration and centrifuging process with 1000x magnification.

In order to indicate identity of the particles, X-ray diffraction (XRD) analysis was presented. The XRD pattern of nanogold is shown in fig.5 and 6; exhibited characteristic reflection of fcc gold (JCPDS NO.04-0784). The diffraction features appearing at single low $2\theta = 38.220^\circ$ to plane (111), 35.39° to plane (200), and 44.34° to plane (220) of the cubic phase of Au. The X-ray result shows agreement with other study for nano Au with particle size less than 50 nm⁽¹³⁾. Colloidal gold diffraction peaks with low intensity related to the small quantities of examining sample.

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Fig(5) : XRD pattern of colloidal gold powder without using reducing agent where NaCl peaks and nano gold peaks are presented.

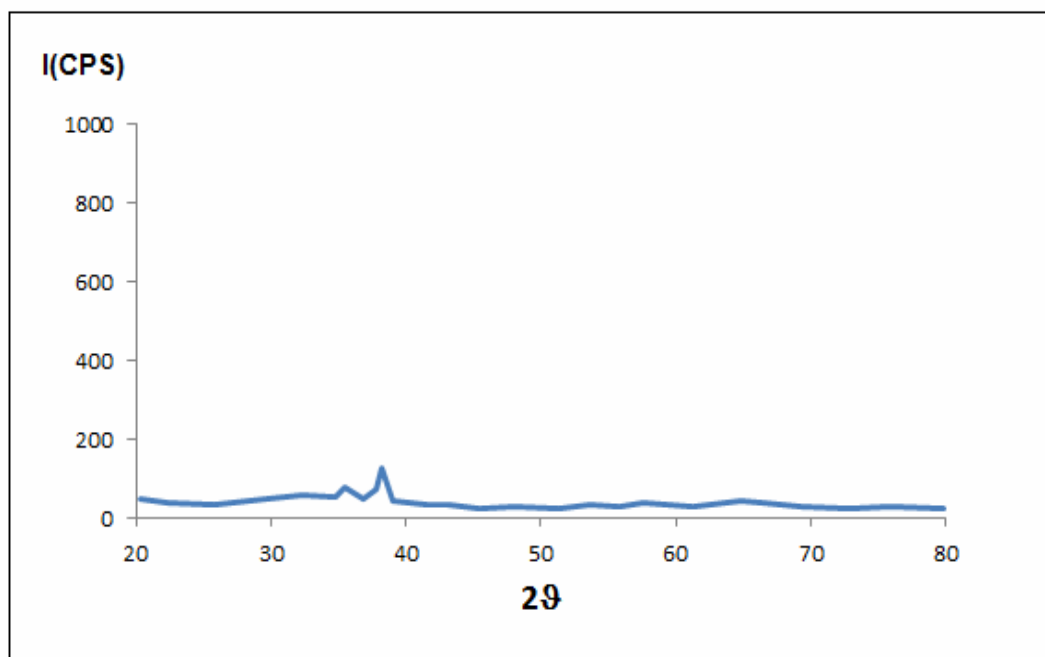


Fig (6): XRD pattern of colloidal gold powder where single nano gold phase presented.

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Using UV-Vis spectra type (UV PROBE Tutorial,Shamadzu corporation using chemical route , Japan-20000024-JA-34) ,fig. (7) showed spectra of the colloidal gold suspension absorbance peak near to (450) nm was a result of the surface Plasmon resonance of colloidal gold⁽¹⁵⁾ prepared with chemical rout.

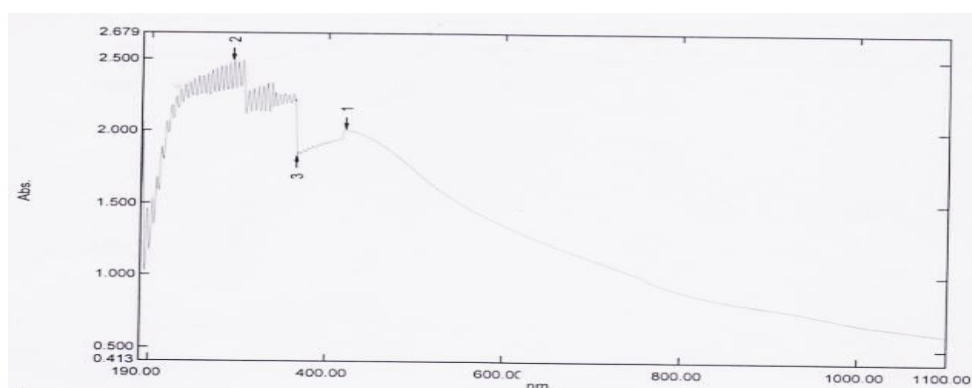


Fig.(7): UV-visible spectroscopy of gold nano particals solution synthesis.

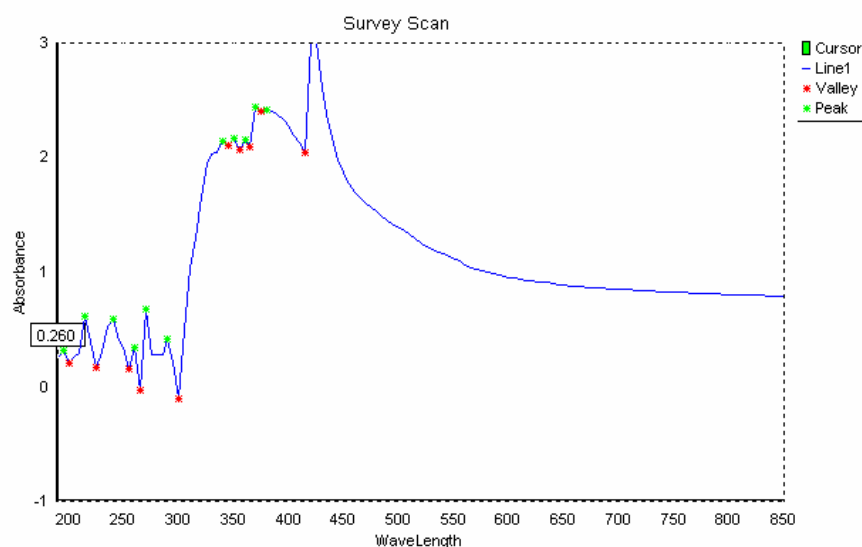


Fig.(8): UV-visible spectroscopy of colloidal gold thin film using reducing agent.

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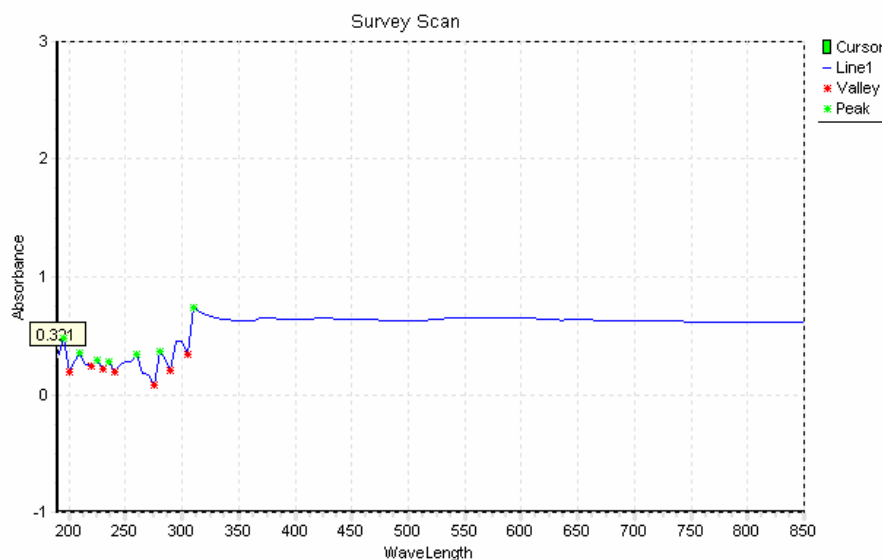


Fig.(9): UV-spectroscopy of colloidal gold thin film using reducing agent , filtering and centrifuging process.

Au thin films was examined by UV-vis spectrophotometer , Fig(8,9) shows the shifted absorption peak at 450 nm to 310nm with lower intensity related to filtering and centrifuging process for Au particles . In order to declare presence of ionic nanogold particles, fig. (10) show ionic Au solution by applying electrophoresis technique on colloidal gold .

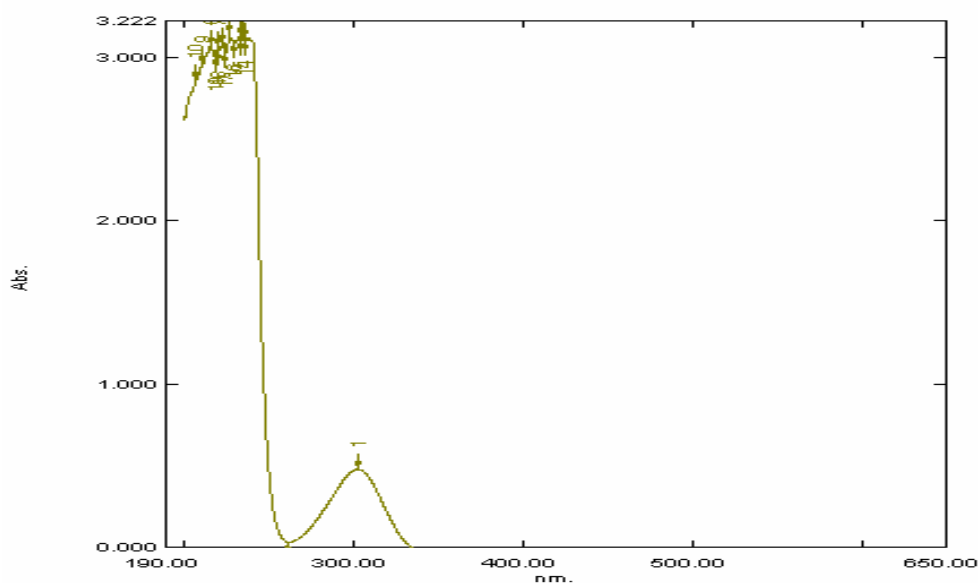


Fig. (10) : UV- visible spectroscopy of Au^+ solution .

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According to atomic force microscopy image , as show in fig.(11,12) , declare the agglomeration of nano particle gold and with higher magnification we can see the particle size distribution less than 50nm . Fig. (12,c) show the surface morphology for small area of nano Au on glass base dispersed unhomogenously on the area . The nano gold particle shape seems semi spherical , and that confirm the particle size range since different shapes obtained according preparation method parameters .

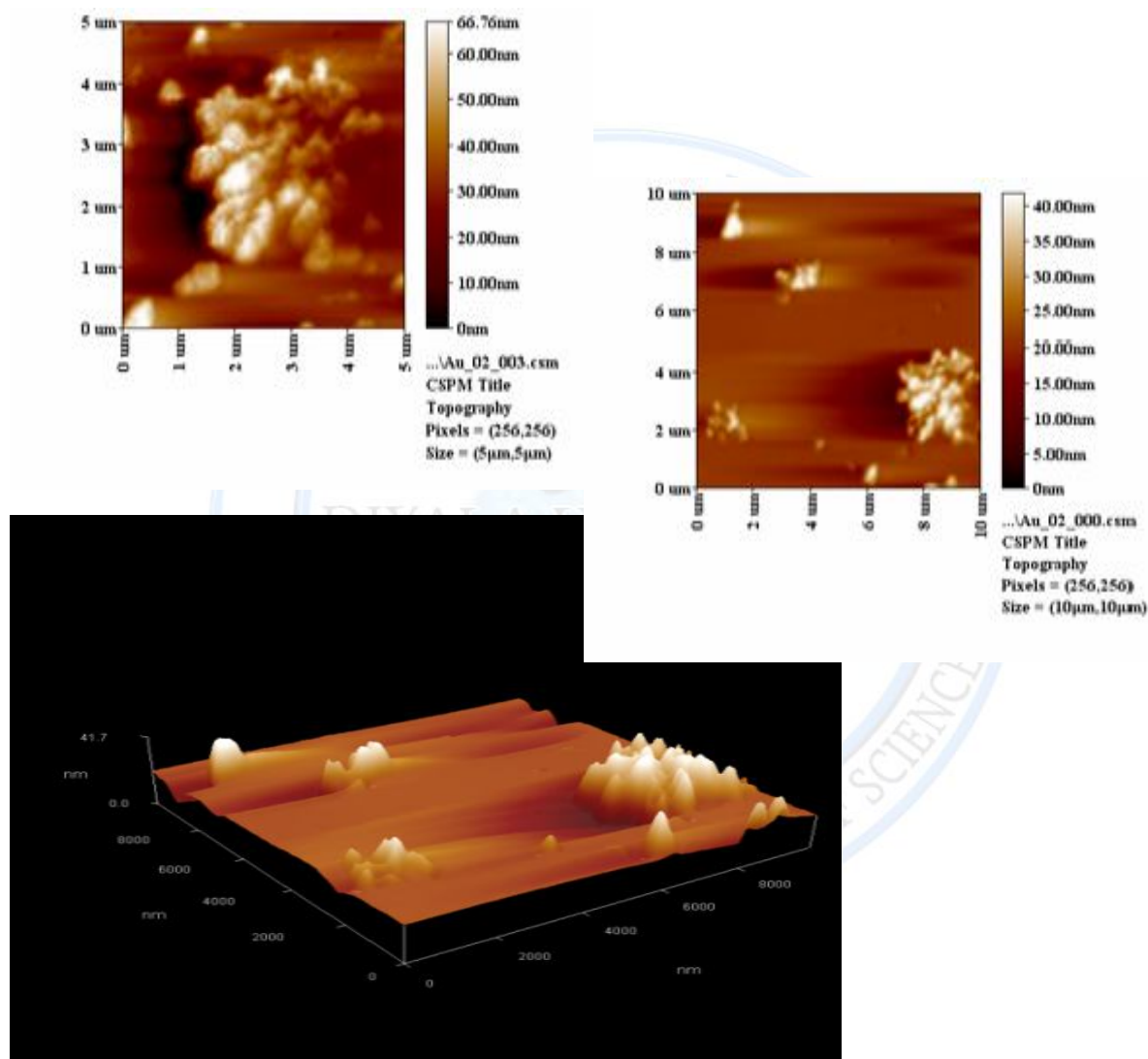


Fig.(11,12) : AFM images with 3D image of Au nano-powder ,prepared by electrochemical method.

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Conclusions

Nano colloidal gold has turned out to play an important role in various fields of nanoscience . Besides their particle size , nano gold particles become of scientific , and technological interest because of their stability to air which means that they can be used even in nanosized form for many applications . UV-vis spectrophotometer analysis show well defined absorption peak for ionic nano gold suspension peak at 310 nm after electrophoresis process . High concentration of NaCl caused little peak diffraction of N_{Au} and could be improved by electro phoresis technique and reduction reaction .

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