Synthesis And Stabilization Of Gold Nanoparticles By Inverse Reduction Method Using Sodium Citrate And Sodium Boro Hydride As Reducing Agent



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ABSTRACT

In recent decades, observed focus largely on the preparation of nano metallic elements, including the gold because of its unique properties and many applications. In most of these researches, the Turkevich' method has been adopted. This method is based on the reduction of gold ion Au3+ to the nano gold Au0 by the addition of a reducing agent to the gold salt H[AuCl4].3H2O. In this research, a novel simple and cost-effective technique is enhanced via the addition of the gold ion (Au3+) to the decreasing agent in opposite to the previous method. This is the cause to call it "The inverse method". In this inverse method one can control the amount of gold salt solution that add and stop this addition by observing the formation of the red color, which indicates the formation of gold nanoparticles(GNPs). Many reactions were conducted to prepare stable gold nanoparticles by usingchemical reducing agents such as (sodium tri-citrate, sodium borohydride), The reactions to prepare GNPs using these reducing agents were done at temperature of 60 - 80 °C. The resulted GNPs were characterized by UV- Vis spectra, zeta potential, atomic force microscope(AFM), scanning electron microscope(SEM), and transmission electron microscope(TEM). The absorption spectrum of the red GNPs solution appeared at the range of 520-540 nm. The gold nanoparticles which formed from various reactions have different shapes but the most prominent form is the spherical, and the sizes have also been different and appeared at the range of 14.5-44 nm, which are consistent with published researches.

Introduction.

Gold is one of the metals originated in the cyclic table with 197 as an atomic number. Moreover, gold may be found in several other oxidation components (+1) and (+3) are stable in aqueous solution, gold found in metals cannot oxidize or blister in air. It has been reported to be inactive to strong acids and alkalis. Humans contain a mean of 0.35 μ g of gold (0) per gram, 2.45 mg of gold in an average 70 kg man.^(1, 2, 3) Gold is

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the symbol of nobleness and gorgeousness that makes it different from other metals. It is thought that Man found gold sometime during the Stone Age and valued it highly not just because of its magnificence, but for its good ability to resist corrosion as well. Hence, the appreciation of gold was reported to take place a long time before the progress in contemporary gold chemistry. Its crimson solution was utilized to shade ceramics and glasses. The general definition of this GNPS can also be known as the gold colloids.

The common feature shared by all kinds of gold colloids is that they have the same color that may range

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from light red purple-red to bluish-red. This impact takes place because of Plasmon resonance, a Plasmon may excite by passing an electron along a reedy metallic film or via reflecting an electron or a photon from a film. Plasmon can play a great role in the optical properties of metals, the surface Plasmon resonance can be adjusted via changing nanoparticles size or shape. This results in optical properties for particles, gold nanoparticles have different shapes such as spherical, cluster, rod, shell, star, and cube. Lots of practices have been proposed to produce metal nanoparticles. More specifically, two general tactics can be used to attain materials on the Nano scale.⁽⁴⁾ Bottom up technique in which the atoms generated from ions reduction are accumulated to produce nanostructures. On the other hand, the top down technique in which materials are detached from the bulk material, comes up with the preferred nanostructures only.

Among the well-known top down methods are photolithography and electron beam lithography. Top down methods lacks the ability eliminate huge quantities of material, whereas bottom up methods are denounced for their insufficient monodispersity which results from the necessity to capture growth at a similar point for all the nanoparticles. (5)

Other bottom up techniques include "templating, chemical, electrochemical, sonochemical, thermal and photochemical reduction techniques."(6, 7, 8, 9). Bottom up synthesis methods often utilise an agent to halt progression of the particle at the Nano scale. Capping materials like a surfactant or polymer can be employed in this method to stop accumulation and precipitation of the metal nanoparticles out of solution selection of the decrease procedure, time, and topping material regulates the shape and size. Michael Faraday was the first scholar who studied the materialization of gold solution from a scientific standpoint. (10) "Phosphorus was the agent he used for the reduction of (Au+3) ions, in the course of the last century numerous easier to handle reducers were found. Among the so-called salt reduction methods, the Turkevich route still one of the most applied procedures.(11)

Sodium citrate reduced (Au+3) ion in hot aqueous solution to give colloids of 15-20 nm. Citrate oxidation

products (acetone di carboxylate) itself can act as protecting agents, if no other stabilizer is used." (12, 13) **Experimental.**

1- Synthesis of gold nanoparticles using sodium citrate as reducing agentNa₃C₆H₅O₇.

I- Preparation stock solution of 10 mMchloroauric acid:

1.0g of HAuCl₄.3H₂O (99% HIMEDIA) was dissolved in 200 mL distilled water in 500 mL volumetric flask and then completed to 250 mL to make a 10 mM . This stock solution of gold (III) salt stored in brown bottle.

II- preparation of 1% tri-sodium citrate:

0.5g of Na3C6H5O7(99% Aldrich) was dissolved in 50 mL distilled water to make a 38 mM.

Step 1:All glassware was rinsed with pure water . 3ml of 1% tri-sodium citrate was diluted with 250 mL distilled water in 500 mL conical flask on stirring hotplate .The solution was heated to a temperature between 60-70 C0

Step2: To the rapidly-stirred solution in step 1, quickly 3ml of 10 mM gold solution gradually the citrate after was added to the solution has turned deep red this indicators the formation of nano gold particles. This solution was given the Symbol (G1)

Repeat the same process above under the same conditions of the concentration of reducing agent and the temperature. But the size of the gold solution differs, 3.6mL ,4mL ,6mL ,7mL this solution were given the symbol . G_2 , G_3G_4 , G_5 respectively.

2- Synthesis of (GNP_s) using Sodium borohydrideas reducing agent NaBH₄.

3mL of 1% sodium boro hydride (98.5 % Aldrich) Was diluted with 250 mL of distilled water in a 500 mL conical flask. The solution was heated to 60-70 C⁰ on the hot plate with magnetic stirrer . To this solution 3.6 mL of 10 mM HAuCl₄.3H₂O was quickly added with stirring . As the solution was turned to ruby red color , the temperature should be reduced to room temperature with continuing of stirring . NaBH₄ reduces the gold(III). Ruby red indicates the formation of gold nanoparticles.

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Description of Gold Nanoparticles.

"GNPswere characterized by UV-Vis spectroscopy (Shimadzu, Japan), Zeta potential analyzer (Brook haven ,USA), Atomic force microscope (AFM),(SPM AA 3000, USA), Transmission electron microscope (TEM), (Philips CM 100, Holland)".

Results and discussion.

1- Visual and UV–Vis spectrum study.

Before the addition of the reducing agent, the gold ion is in solution in the form and when we add the reducing agent, the gold atoms are shaped in the solution particles. This formulation is normally known as nucleation process. The residual melted gold atoms to nucleation sites and growth. Gold nanoparticles interaction with light is strongly, vacillating electric domains of a light ray propagating near a gold nanoparticles cooperate with the free electrons producing a concentrated oscillation of electron charge, which is in harmony with the regularity of visible light. Such resonant oscillations are normally called surface "A plasmon is a quantum of plasma plasmons. oscillation", for small 30 nm mono disperse. (14, 15) Gold nanoparticles the surface plasmon resonance phenomena results in an absorbance of light in the bluegreen part of the spectrum (450-550)nm while red light (700)nm is echoed, yielding a deep red color, bigger size of the particle, the wavelength of surface plasmon resonance associated absorption changes to longer, redder wavelengths. After that, the red light is absorbed, the blue light is echoed making a solution that looks like a pale blue or purple, the size of the particle increases more in the direction of the bulk shifting to be red instead of blue while the echoed light will change from red to blue color. Consequently, the color of the particle changes to be blue due to accumulation. The surface plasmon resonance may be adjusted via changing the Nanoparticles size and shape resulting in particles with tailored optical assets to be used in various applications. This clear we have when preparing samples and how gradient in color(purple, reddish purple, pale pink, blue, pale red, deep red) due to differences in the shape and size of the gold nanoparticles in solution. UV-Vis spectrum for the GNPs prepared from reaction chloroauric acid with trisodium citrate was (530, 520,

520 , 525 , 523) nm $\,$ for $\,$ samples G_1 , $G_2,$ $G_3,$ $G_4,$ $G_5 respectively.$



Figure 1: Colors variation during the synthesis process of gold nanoparticles (Trisodium citrate as reducing agent) Interaction equation.

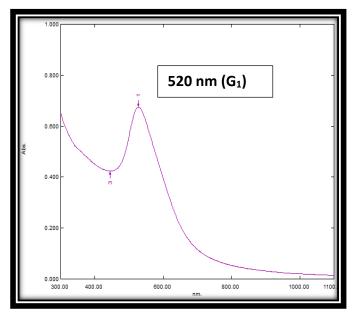
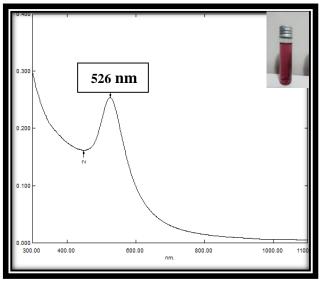
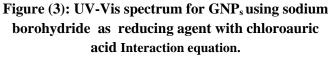
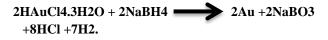


Figure 2: UV-Vis spectrum of GNPs using sodium citrate sample (G₁). 2 HAuCl₄ + 3C₆H₈O₇ 2Au+3C₅H₆O₅ (3-ketoglutaric acid)+8HCl+3CO₂

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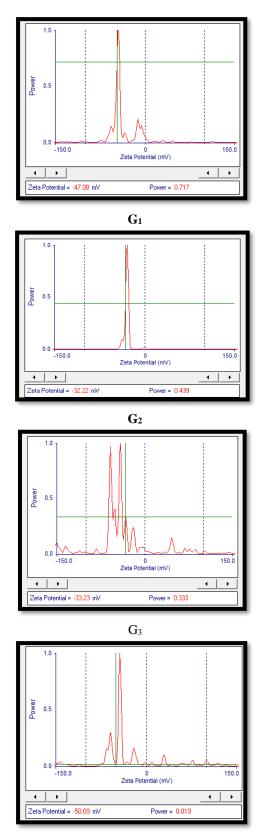
2- Zeta potential (ζ).

Zeta potential is an important pointer of the steadiness of colloidal dispersions. The magnitude of the zeta potential points to the degree of electrostatic repulsion between neighboring, correspondingly charged particles in a dispersion. For molecules and particles, which are small enough, a high zeta potential gives steadiness, the solution or dispersion will struggle against aggregation. In case the potential is small, strong powers can surpass this repulsion and the dispersion might break and flocculate. Gold Nanoparticle is considered steady if their zeta potential is more positive than +30 mV or more negative than -30 mV. ⁽¹⁶⁾. In the table (3.1) some of the solutions of Nano-stable and the value of the zeta is as high as (trisodium citrate, sodium boro hydride. Zeta potential results for samples (G₁, G₂, G₃, G₄, G₅).

Table (1):Zeta potential values to gold nanoparticles resulting from the interaction various quantity of volum chloroauric acid with trisodium citrate as

reducing agent.

symbol	Conc GNPs	Zeta potential		
G 1	0.028mg/mL	-47.08mv		
G2	0.025 mg/mL	-32.22mv		
G3	0.03 mg/mL	-33.23mv		
G4	0.045 mg/mL	-50.09mv		
G5	0.05 mg/mL	-53.55mv		



G4

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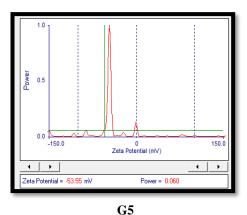


Figure (4): Zeta potential values for G₁, G₂, G₃, G₄, G₅respectively Zeta potential value (-27.01mv). for GNPS using sodium borohydride as reducing agent

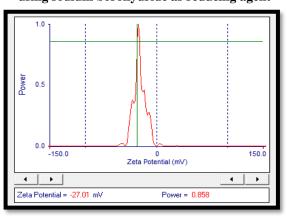
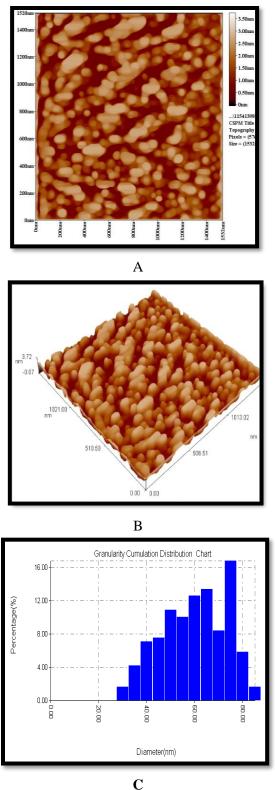


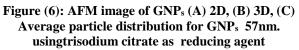
Figure (5):Zeta potential value for GNPs using sodium borohydride as reducing agent

3- AFM study.

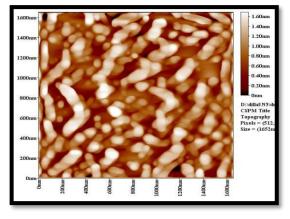
The atomic force microscope (AFM) is matched to be used in distinguishing Nanoparticles. It provides the ability of 3D visualization and both qualitative and quantitative information about several actual features such as size, roughness, morphology and surface texture. Statistics related to size, surface, and volume distributions might also be obtained. A variety of particle sizes could also be identified via the same scan, from 1 nanometer to 8 micrometers. Furthermore, the AFM has the ability to identify Nanoparticles in manifold mediums such as ambient air, controlled environments, and even liquid dispersions. ⁽¹⁷⁾ "GNPs for sodium citrate as reducing agent have a roughness surface and big particles diameter distribution, the average particle of GNPs is measured by AFM images was (57 nm), but GNPs using sodium boro hydride have a smooth surface and small particles diameter

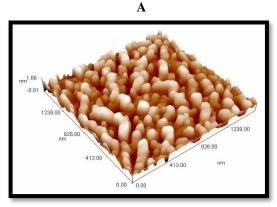
distribution. The average particle of GNP_s is measured by AFM images was (41 nm)."

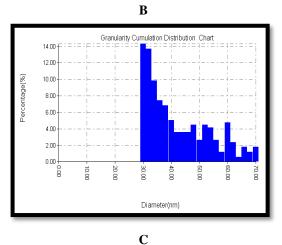








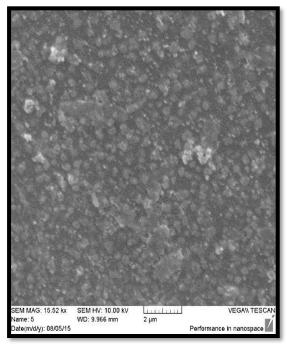




Figure(7):AFM image of GNPs (A) 2D, (B) 3D, (C) Average particle distribution for GNPs 41 nm.by sodium boro hydride as reducing agent

4- SEM study.

Detailed information related the to construction, size combined the and the nanoparticles can all be obtained via scanning electron microscopy (SEM) whose image can show high density of Au Nanoparticles. These Nanoparticles are consistently dispersed on the surface. SEM micrograph revealed the formation of mono and poly dispersed nanoparticles which ranged between 20 and 80 nm in size, different morphology of nanoparticles is obtained by SEM images. Low and high magnification images indicated that the GNP_s were spherical in nature, it was evident that the particles were < 100 nm (23, 33, 47, 52. 74) nm in diameter. The SEM analysis of GNP_s were agreement with the results of GNP_s which reported in literatures.⁽¹⁸⁾



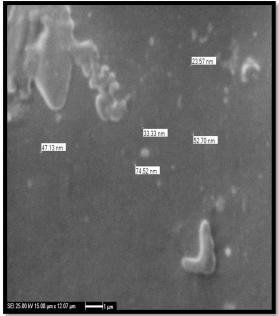


Figure (8) SEM images the size range of the nanoparticle between 20-70 nm

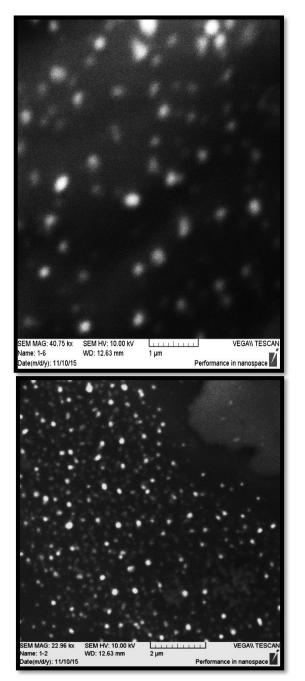
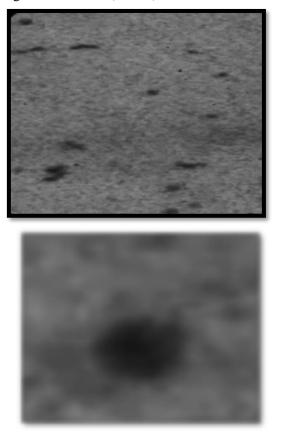


Figure (9): SEM images of GNPs using sodium borohydride as reducing agent

5- TEM study.

The TEM exhibited the biosynthesized gold Nanoparticles are in various forms and they are mostly circular in nature followed infrequently triangular and rare hexagonal. The particles size vary from 14 to 50 as the shape and size of some of these particles was asymmetrical. This can be because of the various stages of development. Differences in nanoparticles size and shape produced by biological schemes are frequent. "TEM is the one of the most popular characterization techniques for nanoparticles. ⁽¹⁹⁾ In this technique, a real image of nanoparticles is taken, different magnifications can be used to see a more detailed or general shape of nanoparticles. These images contain a lot of information regarding shape and size distribution, and even crystallographic structure and characteristics of nanoparticles. TEM images data clearly shows that structure and nature of reducing agent play an important role to reduce the HAuCl4.3H2O into gold Nano composites of different morphology, this method provides highly stable water soluble. Shape differently particles formats (clusters, spherical, branched chain) average size between (20 -95)nm."



- Figure (10): TEM images of GNP_S synthesized using trisodium citrate .With chloroauric acid . Size particle is 14- 24nm.
- 5-Stability for gold nanoparticles using trisodium citrate (G₁,G₂,G₃G₄, G₅) and sodium borohydride (G₆) as reducing agent with time.

Table (2) Change UV-Vis spectrum for GNP_s with increase time

increase time							
Symbol	Date 1 5/6/2015	Date 2 20/8/2015	Date 3 5/10/2015	Date 4 7/12/2016	Date 5 2/2/2016	Date 6 2/4/201	Color
G_1	530nm	525nm	524nm	523nm	524nm	524nm	Pale red
G_2	520nm	520nm	524nm	523nm	524nm	524nm	Pale red
G ₃	520nm	520nm	524nm	523nm	525nm	524nm	Ruby red
G_4	525nm	525nm	528nm	528nm	531nm	531nm	deep red
G_5	523nm	523nm	528nm	526nm	526nm	527nm	Purple
G_6	526nm	526nm	526nm	526nm			Ruby red

Trisodium citrate (0.1 mg/mL) as reducing agent with different quantities of gold solution

 $G_1 __0.048$ mg/mL for gold solution $__0.024$

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mg/mL for gold nanoparticle
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$G_2 _ 0.05 mg/mL$	0.028 mg/mL
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- G₃ ____0.06 mg/mL _____0.032 mg/mL
- $G_4 __ 0.09 \ mg/mL __ 0.048 \ mg/mL$

 $G_5 _ 0.1 \text{ mg} / \text{mL}$ _ 0.056 mg/mL

 $G_6 _ 10 \text{ mg/mL}$ Sodium boro hydride as reducing agent with gold solution (4mg/mL).

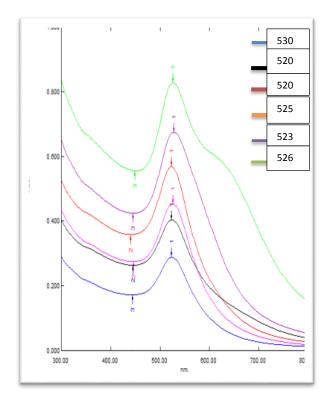


Figure (11):UV-Vis spectrum of GNP_Susing trisodium citrate with different quantities of HAuCl4.3H₂O (G₁, G₂, G₃, G₄, G₅), and sodium boro hydride as reducing agent (G₆)

This peaks usually occurs between 520-530 nm depending on the diameter of gold nanoparticle, and occurs at longer wavelength for larger gold nanoparticles, changing the concentration of gold nanoparticles just changes the absorbance magnitude (peak height) but not the wavelength of the peak. ⁽²⁰⁾Stability of GNP_s was investigated by measuring the absorption spectra of the gold nanoparticle solution prepared at various times. The sample was stored at room temperature in a transparent vial and UV-Vis spectra of the sample were taken after the formation of final ruby red gold nanoparticle solution. For example, sample (G₁) plasmon absorbance at 530nm was taken two weeks, two, four, six months and one year. The results indicate that there is small obvious difference in position of absorption peak during the initial two months from plasmon absorbance at 530nm to 524nm. After six months, the position of the peak has a slight blue shift (525 to 524nm) suggesting the formation of smaller particles without any aggregation. Even after one year

the plasmon absorbance remained at same wavelength and no aggregation. But samples (G_2, G_3, G_4, G_5) . Table (2) show a slight red shift suggesting the formation of bigger particle. Often the peak will broaden or a secondary peak will form at longer wavelength (due to the formation of aggregation).UV-Vis spectroscopy can be used as a characterization technique that provides information on whether the nanoparticles solution has destabilized over time. Unaggregated GNPs will have a red color in solution. If the particles aggregate, the solution will appear blue/purple and can progress to a clear solution with gold nanoparticle precipitates. (21, 22) As the particles increase in size, the absorption peak usually shifts toward the red wavelengths. Increase the absorption indicates that the amount of gold nanoparticles increases. The stable position of absorbance peak indicates that new particles do not aggregate. One can understand that since the gold Nano particles possessed a negative charge due to the adsorbed citrate ions, a repulsive force worked along particles and prevented aggregation. Sodium boro hydride is a strong reducing agent reduces gold solution and act reducing and stabilizing agent give it ruby red color for gold nanoparticle was stable for a period of time more than six months . Negative charge consisting of reducing agent will surround the gold Nano particles for remain stuck in solution.

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4- Conclusion:

This paper defines the facile and rabid combination of gold nanoparticles via a novel biochemical method. The proposed method or the so called the reverse method was utilised via adding the Au3+ solution to the reducing agent while heating and stirring. The merits of this method are its simplicity and ease, hence, it can be used by graduates as well as undergraduate. This method offers a control on the quantity of the gold, the salt and the reducing agent which makes this method as easy technique to follow-up the gold Nano-particles establishment via the red color of solution. In a nutshell, the paper has examined the employment of orchid and Arabic gum as a reducing and stabilizing agent for the synthesis of AuNPs in an aqueous medium. The UV-Vis, zeta potential, AFM and TEM results illustrate that as synthesized AuNPs are polydisperse nature, quasi-spherical and hexagonal shape with an average size ranging from 14 –95 nm.

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تحضير واستقرارية جسيمات النانو ذهب بواسطة طريقة الاختزال العكسي باستعمال ثلاثي سترات الصوديوم والصوديوم بورو هيدرايد كعوامل مختزلة.

عدوية جمعة حيدر ظلال عبد الغفور شبيب عبد الاله ثابت محمد

E.mail:

الخلاصة:

تضمن البحث طريقة جديدة وسهلة واقتصادية قد طورت من خلال إضافة حامض الاورك الى العامل المختزل المغلي لهذا السبب سميت بالطريقة العكسية في هذه الطريقة ممكن السيطرة على كمية ملح الذهب المضاف وإيقاف الإضافة عند تشكل جسيمات النانو ذهب من خلال تحول لون المحلول الى اللون الأحمر الياقوتي الذي يشير الى تكون جسيمات النانو ذهب حضرت الجسيمات النانوية المستقرة باستعمال ثلاثي سترات الصوديوم والصوديوم بورو هيدر ايد كعوامل مختزلة وفي درجة حرارة تفاعل (60-60) م⁰ تم دراسة خواص النتائج عن طريق التقنيات التالية طيف الاشعة فوق البنفسجية المرئية ، جهد زيتا ، مجهر القوة الذرية ، المجهر الالكتروني الماسح و المجهر الالكتروني الناقل . طيف الامتحال النانو ذهب الأحمر بين 520- 540 دانومتر وقد كانت الجسيمات الناتجة مختلفة الاشكال ولكن الشكل الأكثر وضوحا هو الكروي ومعدل حجوم الجسيمات الناتجة هو بين 14.50 بنومتر وقد كانت متطابقة مع البحوث المنشورة سابعاً .