

## **SYNTHESIS AND CHARACTERIZATIONS OF SILVER NANO PARTICLES USING CHEMICAL REACTION METHOD**

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**ABSTRACT:** - In the present investigation colloidal silver nanoparticles was prepared by chemical reaction method from silver nitrate ( $\text{AgNO}_3$ ) and sodium citrate ( $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$ ) in water. The preparation method were achieved at three levels of temperature (100, 150 and 200°C) the produced silver nanoparticles characterize by UV-vis Visible Spectroscopy, Scanning Electron Microscopy (SEM) and Particle Size analyzer the result of particle size analyzer show that their size distribution to lie in the range of (58.9 -202) nm. The results show that silver nanoparticles are 58.9, 76.4 and 202 nm at temperatures of (100, 150, 200) °C, respectively can be produced.

**Keywords:** Silver nanoparticles, Chemicals reduction, UV-Vie Absorption Spectra.

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### **1- INTRODUCTION**

In recent years the chemical engineering process have witnessed increased interests of scientific community in Nanotechnology and especially nanomaterials have received much consideration because their structure and properties differ appreciably from those of molecules, atoms, and bulk materials<sup>(1)</sup>. Synthesis of silver nanoparticles is an area of considerable interest during the past decades<sup>(2)</sup>. Silver particles, which have fine size and uniform particle size distribution, have not only preferential in many fields of commercial application, but also have various potential usages in electronic, medical, the petroleum refining ,petrochemical processes and chemistry industries<sup>(3)</sup>. On the other hand, the use of silver nanoparticle is very important in separation process and purification units. Many authors used silver nanoparticle to coating the membranes filtration and filters in air and water purification<sup>(4,5)</sup>. Silver nanoparticles have synthesized with a range of different shapes, such as, spheres, discs, rods, wires, stars, prisms, right bipyramids, and cubes<sup>(6)</sup>. There are different methods to synthesize silver nanoparticles, such as conventional temperature assisted process, controlled reaction at elevated temperatures, and microwave assisted process<sup>(7)</sup> and too, There are many ways described in various literatures to synthesize silver nanoparticles. These included physical, chemical, and biological methods<sup>(8)</sup>. Chemical reduction methods have widely investigated for the synthesis of AgNPs because these techniques can be performed under easy and gentle conditions<sup>(9)</sup>. The advantage of chemical synthesis has their versatility in designing and synthesizing new materials that can be refined into the final product. The primary advantage that chemical processes has offered over other methods to lie in good chemical homogeneity, as chemical synthesis has offered mixing at the molecular level molecular chemistry can be designed to prepare new materials by understanding, how material has assembled on an atomic and molecular level and the consequent effects on the desired material macroscopic properties<sup>(10)</sup>. It has possible that the reaction rates or even the branching ratios can be regulated by varying the reaction

temperatures in the thermal reduction reactions. However, very little data has reported concerning the effect of temperature on the synthesis of silver NPs using photochemical reactions<sup>(11,12, 13)</sup>.

The present study aims to prepare of silver nanoparticle AgNPs using silver nitrate AgNO<sub>3</sub> and characterizes it using chemical reduction method and study the effect of temperatures.

## **2- EXPERIMENTAL WORK**

### **A. MATERIALS**

Silver nitrate (AgNO<sub>3</sub>) and Sodium citrate (Na<sub>3</sub>C<sub>6</sub>H<sub>5</sub>O<sub>7</sub>) were supplied by Fluke Company (Germany). All chemicals were used high purity 99.99%

### **B. PREPARATION OF SILVER NANOSTRUCTURES**

Silver colloid has prepared by using the chemical reduction method which depends on the reduction of silver nitrate (AgNO<sub>3</sub>), by sodium citrate (Na<sub>3</sub>C<sub>6</sub>H<sub>5</sub>O<sub>7</sub>). After preparing 100 ml of stock solution of silver nitrate, 25 ml of stock solution (0.005 M) silver nitrate has taken and added to a 100 ml of distilled water. The resulting 125ml solution has heated temperature of 100°C. , covered with a watch glass on a hot plate stir solution with a magnetic stir bar Once boiling, add 5mL of a 1% solution of trisodium citrate drop wise, about 1 drop per second. Wait for solution to change to a light golden color or pale yellow. Carefully remove beaker from hot plate and let solution cool at room temperature as shown in Figure 2. The process has repeated at different temperatures.

Mechanism and Structural composition of reaction has expressed as showing in equation (1) and figure (1)



### **C. CHARACTERIZATION TECHNIQUE**

The bioreduction of Ag<sup>+</sup> ions has monitored by UV-Visible spectrophotometer (Metertech, UV/VIS SP8001) immediately after the synthesis of AgNPs by diluting a small aliquot of samples into distilled water. The morphology of silver nanostructures (AgNPs) has investigated with scanning electron microscope (SEM, the VEGA EasyProbe) and particle size analyzer. The (NanoBrook 90Plus Particle Size Analyzer) analytical has done by preparing thin films of samples in carbon copper grid and were allowed to dry by placing them in mercury lamp for 5 minutes.

## **3. RESULTS AND DISCUSSION**

It is important to mention here that many kinds of techniques can be used to characterize the nanoparticles depending on the kind of the applications. Silver nanoparticles have synthesized according to the method described in the previous section, the colloidal solution turned pale brown, pale yellow and pale red indicating that the silver nanoparticles have formed. The preparation temperature of nanomaterials plays an important role in determine the general specification of produced materials. Then, in the present investigation three samples of silver nanoparticles were prepared under three levels of temperatures (100 °C, 150 °C and 200 °C). It is important to mention here that, due to the potential of silver particles for photonic applications the optical properties of the particles have investigated by extinction measurement (UV/VIS).UV-visible spectroscopy to identify the structural characterization of silver nanoparticles (AgNPs). The Fig.3 has shown the UV-vis spectra recorded at temperature of 100- 200°C. In general, when temperature increasing, the strongest absorption band gradually shift to a higher wave length. It has noted that the increase of absorption indicated that the amount of silver nanoparticles increased. Then, it has concluded that the nanoparticles size has decreased with increasing of reaction temperature. The absorbance band was broadened and positioned at 442.28, 423.5 nm and 421.41 nm at the temperature of 100 °C, 150 °C and 200 °C, respectively for showing that silver nanoparticles have produced at a low temperature.

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Fig. 4 shows the SEM images of the prepared AgNPs at different reaction temperatures. The results have predominantly dendrite provides a clear shape in SEM micrographs with different magnifications of first method using citrate-AgNPs, which have prepared by 0,005 M of  $\text{AgNO}_3$  reduction at  $T=100^\circ\text{C}$  for 2h. It was noted that the particles with a size ranging from (80.7 to 413.2) nm with mean average of 202 nm. From Fig. 4 (b) it has noted that the agglomerations of nanoparticles have the biggest.

On the other hand, Fig. 5 shows the results of the particle size analyzer of the three samples. It has noted that the silver nanoparticles have a spherical shape dispersed with size of range 29.3–154.5 nm size for sample number 2 that has prepared at  $T=150^\circ\text{C}$ .

Also, Fig.4(c) shows SEM micrograph of spherical and irregular Ag nanoparticles using citrate-Ag NPs, which have prepared by 0,005 M of  $\text{AgNO}_3$  reduction at  $T=200^\circ\text{C}$  for 2h and the particles with a size ranging from 22.3to 99.5 nm.

In Fig. 5 the particle size distribution of the AgNPs samples have statistically has optimized and plotted in terms of volume fraction with sizes. After analyzing data, sample (1) has found in the range of 80-413.2 nm with mean average of 202 nm. On the other hand, sample (2) has found in the range of 29.3 to 154.5 with mean average of 76.4 nm. And sample (3) showed particle size in the range of 22.3 to 99.5 with mean average of 58.9 nm. Then, it has concluded that the increasing of preparation temperature will lead to small particle size diameter due to the broken of salt molecules and effect on reaction.

From the SEM images and partical size results it can be draw the relationship between the effect temperature and size of the prepared AgNps as shown in Fig. 6. The higher rate of reduction has occurred at higher temperature due to the consumption of silver ions in the formation of nuclei whereas the secondary reduction has stopped on the surface preformed nuclei. The broadening peak has obtained at low temperature shows formation of large sized nanoparticles and the narrow peak has obtained at high temperature, which indicates the formed nanoparticles have small in size and the higher rate of reduction of silver ions has occurred in the  $200^\circ\text{C}$ . Finally, it has concluded that higher temperature of  $200^\circ\text{C}$  is more favorable for production of Ag nanoparticles.

### CONCLUSION

In the present work, Ag nanoparticles collide solution has prepared successfully by the reduction of silver salt. It has concluded that the preparation temperature is important factor in determining the final Ag nanoparticles size and specifications. The best reaction temperature for Ag nanoparticles production has  $200^\circ\text{C}$ , that showed particle size with mean average of 58.9 nm. The results obtained by the proposed method have efficient, cheap, and high purity.

### ACKNOWLEDGMENT

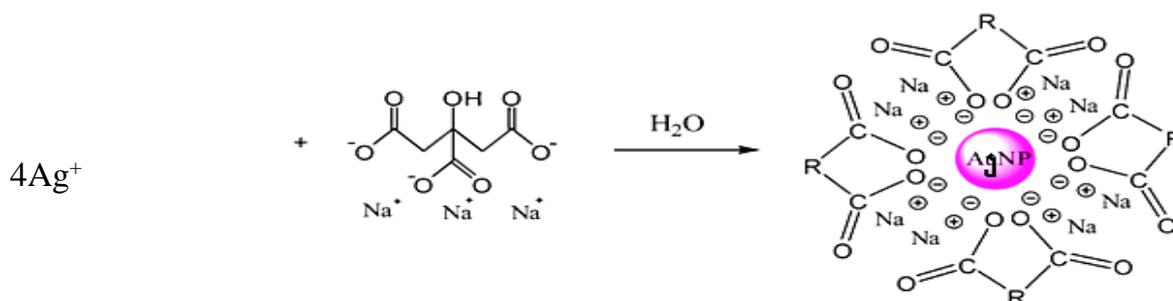
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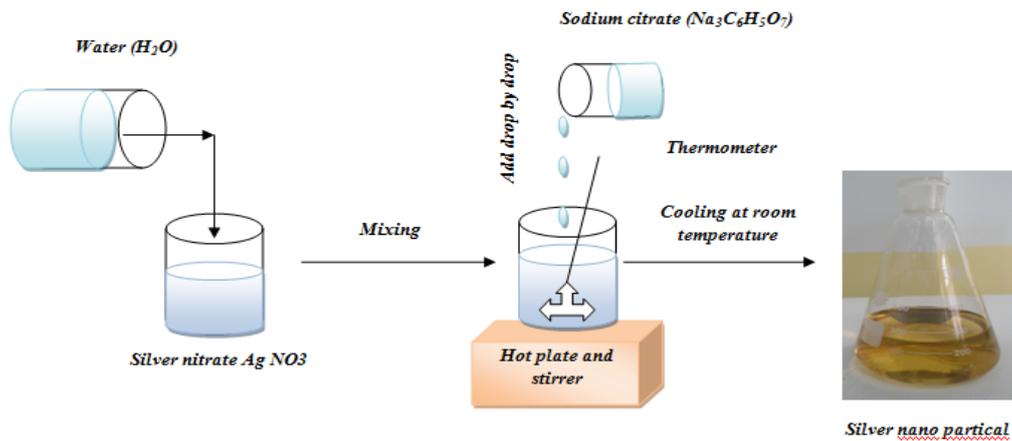
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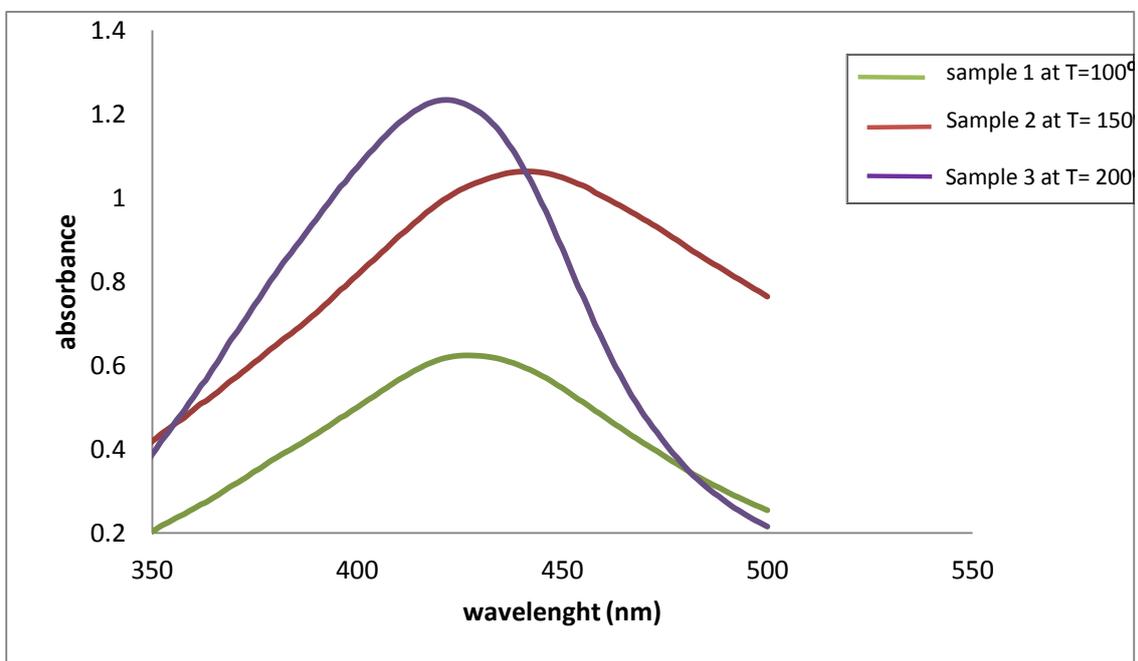
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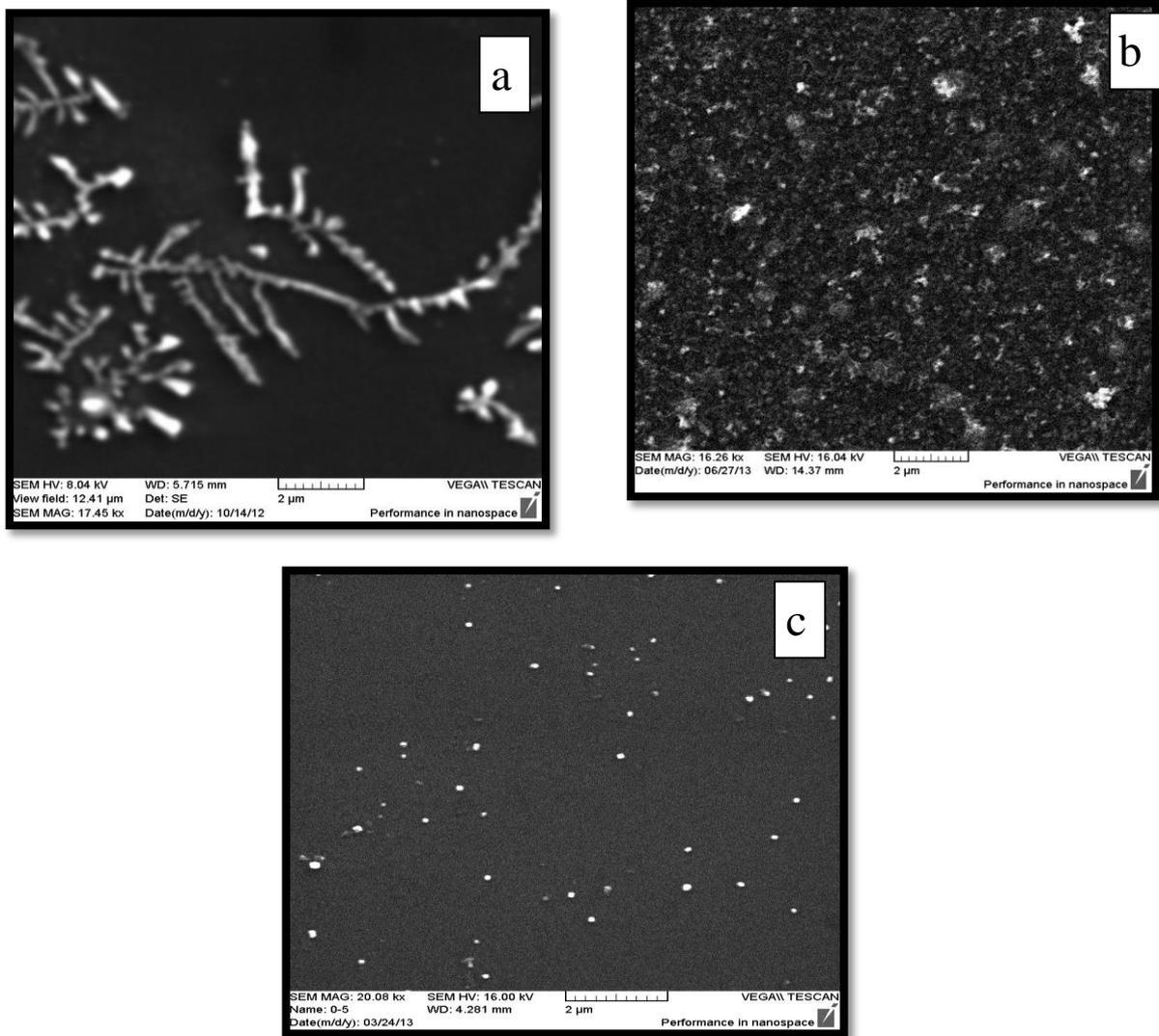
**Fig 1:** AgNps synthesis using the chemical reaction method at different temperatures <sup>(15)</sup>



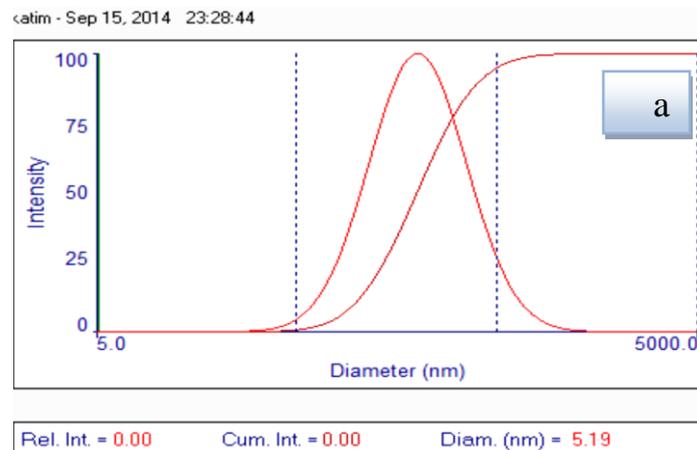
**Fig. 2:** showing the process of the preparation of silver nanoparticles

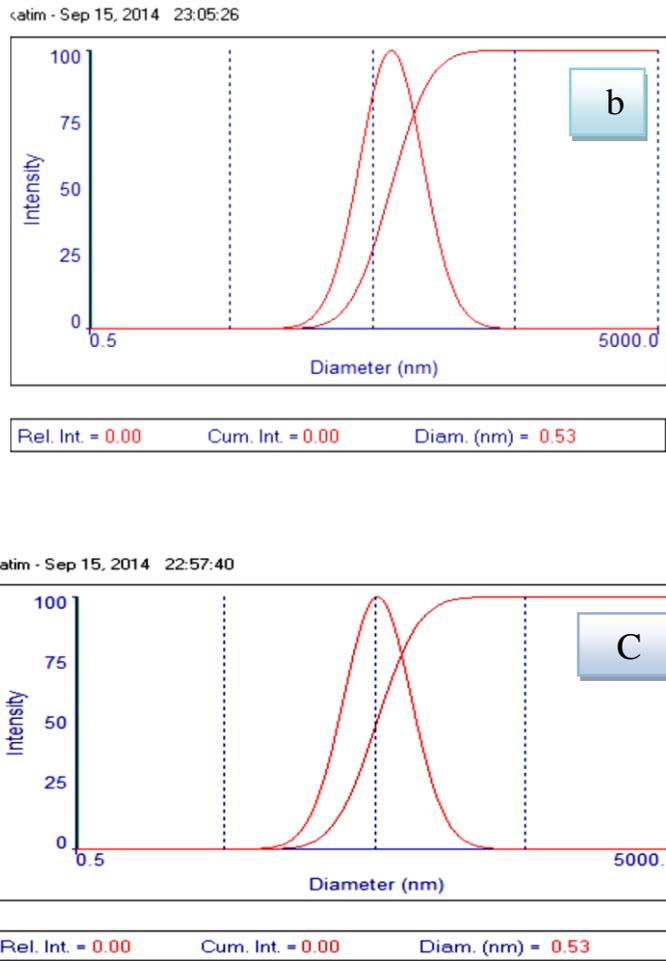


**Fig. 3:** UV-Vis absorption spectrum of silver nanoparticles synthesized by chemical method at different Temperatures (100, 150 and 200)°C.

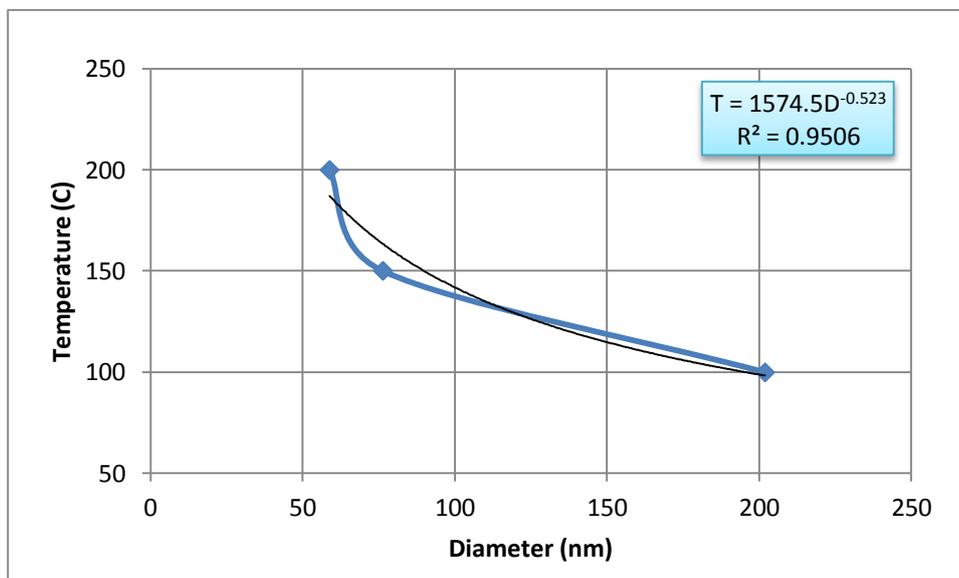


**Fig.4:** (a, b& c) SEM images of the prepared AgNPs using chemical reaction method at different temperatures (a: 100°C, b: 150°C and c: 200°C ), respectively





**Fig.5:** Particle Size Analyzer distribution of the prepared Ag NPs using chemical method at different temperature (a: 100°C, b: 150°C, 200°C)



**Fig. 6:** The effect of temperature on Ag NPs diameter

## تحضير وتشخيص الفضة النانوية بالطريقة الكيمياوية وبدرجات حرارية مختلفة

ختام سالم شاكر

مدرس مساعد/ الجامعة التكنولوجية

### الخلاصة:

تم الحصول على الفضة النانوية الغروية من خلال طريقة التفاعل الكيمياوي باختزال نترات الفضة من قبل سترات الصوديوم ( $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$ ) في الماء. وشخصت الفضة النانوية التي حصل عليها بطريقة التحليل الطيفي للأشعة فوق البنفسجية ، المجهر الإلكتروني (SEM) ، وقيست الفضة النانوية بواسطة استخدام جهاز محلل حجم الجسيمات وتوزيع حجمها لقد تبين ان الحجم الحبيبي يقع بين (58.9- 202) نانومتر .وتشير النتائج إلى أن معدل جزيئات الفضة المحضرة يتراوح ما بين (58.9، 76.4 و 202 ) نانومتر عند درجات حرارة (100، 150، 200) م° و على التوالي.

**كلمات البحث:** الفضة النانوية ، التفاعل الكيمياوي، الأشعة فوق البنفسجية والمرئية واطياف الامتصاص.