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# Research Article

# Process Parameters for Green Synthesis of Silver Nanoparticles using Leaves Extract of Aloe Vera Plant

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# Abstract

In the present study, an environmental friendly synthesis of silver nanoparticles (AgNPs) has been investigated using Aloe Vera plant leaves extract act as reducing and stabilizing agent. The formation of AgNPs was confirmed by changing their color to dark brown due to surface plasmon resonance (SPR) phenomenon. The effect of process parameters on synthesis of AgNPs from Aloe Vera leaves extract such as AgNO<sub>3</sub> concentration (molarity), extract volume percent, sunlight exposure time and temperature were studied. The AgNPs obtained were characterized using UV-vis spectroscopy, Atomic Absorption Spectroscopy (AAS), Atomic force microscopy (AFM), and Fourier Transformation Infrared Spectroscopy (FTIR). UV-Vis spectroscopy showed the SPR peaks for AgNPs using Aloe Vera leaves extract were between 420-490 nm. Atomic force microscopy (AFM) showed the AgNPs are spherical in shapes with the average particle diameters between 34 - 102 nm. The best conditions for synthesis of AgNPs using Aloe Vera leaves extract were found at 5 mM AgNO<sub>3</sub> concentration, 15% extract volume percent, for 10 min of sunlight exposure and temperature at 60 °C.

Keywords: Green synthesis, Aloe Vera, silver Nanoparticles

# 1. Introduction

In recent years, metallic nanoparticles have a great attention because of the modification of properties perceived due to size effects, distribution and morphology due to present a higher surface-to-volume ratio with decreasing size of nanoparticles, this modifying the catalytic, electronic, and optical properties of the metal NPs (Elizondo *et al.* 2011; Song & Kim 2008). Silver metal nanoparticles is widely known as particular interest due to unique properties as high conductivity, chemical stability, catalytic and antibacterial (Sharma *et al.* 2009).

Nanoparticles can be synthesized using different methods including chemical, physical, and biological (Iravani *et al.* 2014). The biological method is a green chemistry method uses naturally occurring reducing agents such as biological microorganism (plants extract, fungus or bacteria) to synthesis nanoparticles (Abou El-Nour *et al.* 2010).

Green synthesis of AgNPs using plant extracts provides advancement over chemical and physical method as it is cost effective, no need to use high energy, environment friendly, and no toxic chemicals (Singhal *et al.* 2011; Panigrahi 2013).

Green silver nanoparticles have been synthesized using various natural products like coffee and tea extract (Nadagouda & Varma 2008), Hibiscus rosa sinensis leaf (Philip 2010), Cycas Leaf (Jha & Prasad 2010), garlic (Allium sativum) extract (Rastogi & Arunachalam 2011), Pomegranate peel extract (Ahmad *et al.* 2012), Aloe Vera plant extract(Chandran *et al.* 2006).

Aloe Vera is a natural plant, which is recognized to have imaginary medicinal reputation known back to thousands of years ago. It contains treasures of nutritional and antipathogenic compounds. Aloe Vera is opted for the synthesis of silver nanoparticles because of the presence of natural phytochemicals which provide natural capping and reducing agents (Yebpella *et al.* 2011).

The aim of this work is to use Aloe Vera leaves extract as low cost and ecofriendly environmental approach to green synthesis silver Nanoparticles (AgNPs). The effect of synthesis parameters such as: Silver ion concentration, Aloe Vera leaves extract concentration, and temperature been investigated. Also, the effect have of electromagnetic radiation (sunlight) on AgNPs synthesis has been studied. The AgNPs have been characterized by UV-visible spectroscopy, atomic absorption, AFM and FTIR will be used for characterization.

#### 2. Materials and Methods

### 2.1 Materials

Silver nitrate (AgNO3, 99% purity, Sigma Chemical Co., Western Australia). Aloe Vera leaves, Collected from local gardens.

# 2.2 Preparation of Silver Nitrate Solution

Different molarities (1, 3, 5, 7 and 9 mM) of silver nitrate aqueous solution were prepared. The silver nitrate aqueous solution was used as precursor to synthesis AgNPs.

### 2.3 Preparation of Aloe Vera Leaves Extract

Fresh green leaves were collected from Aloe Vera plant and then washed extensively with distiller water for several times to remove dust particles, impurities or any other stakes. The leaves were then chopped into small pieces. A 10 g of chopped Aloe Vera leaves were boiled with 100 ml of distilled water at 80 oC for 10 minute (Pattanayak *et al.* 2013) in a 250ml flask to obtained Aloe Vera leaves extract. The aqueous leave extract was then filtered using Chm filter papers (No. 2042-150, with 3-2  $\mu$ m pore size) to obtained Aloe Vera extract with a yellow color to be used as reducing and capping agents in the AgNPs synthesis.

#### 2.4 Silver Nanoparticles Synthesis

Silver nanoparticles were synthesized by adding a well known volume of plant extract to AgNO3 aqueous solution at known molarities then mixed using hand mixture. The mix solution was allowed to react in sunlight for a specific time. Samples were then monitored for the formation of AgNPs by monitoring the change in color of the mix solution.

# 2.5 Effect of Silver Nitrate Concentration on AgNPs Synthesis

A 10 mL of Aloe Vera leaves extract is added to 90 ml of aqueous solution of AgNO3 of different concentration (1, 3, 5, 7 & 9) mM in a 250 ml flask. The solution was allowed to react at room temperature under sunlight for 10 minutes. The sunlight intensity is 95800 ± 500 LUX (as measured by Note3 Samsung mobile at IOS 800). The color of the solution mixture of silver nitrate and Aloe Vera leaves extract was changed within 10 minutes from yellow brown to deep brown color under sunlight. This indicates the reduction of Ag+ ions to Ago nanoparticles (Awwad *et al.* 2013).

2.6 Effect of Aloe Vera Extract Concentration on AgNPs Synthesis

Different volume ratio (5, 10 and 15 %) of Aloe vera leaves extracts was added to the best molarity of AgNO3

aqueous solution. A 5, 10 and 15 ml of Aloe Vera leaves extract was added to 95, 90 and 85 ml of AgNO3 solution respectively and waited for 10 min for the color change under sunlight at room temperature. The Aloe Vera leaves extract and AgNO3 solution mixture was then characterized using UV, AFM and Atomic absorption.

# 2.7 Effect of Sunlight Exposure on AgNPs Synthesis

Process Parameters for Green Synthesis of Silver Nanoparticles using Leaves Extract of Aloe Vera Plant

The best volume of plant extract were added to the best molarity of AgNO3 solution at room temperature and waited for the color changed under sunlight at different time (5, 10, 15 min) for each sample. The Aloe Vera leaves extract and AgNO3 solution mixture was then analyzed using UV, AFM and Atomic absorption.

# 2.8 Effect of Temperature on AgNPs Synthesis

The effect of temperature on AgNPs synthesis was studied at (25, 40, 50, 60, 70 and 80) oC using water bath r. The best volume of Aloe Vera leaves extract was added to the best concentration of AgNO3 to synthesis AgNPs at constant sunlight exposure time of 10 min and wait for color change. Each sample was then taken out from the bath and water cool down to room temperature. The Aloe Vera leaves extract and AgNO3 solution mixture was then characterized using UV, AFM and Atomic absorption.

# 3. Analysis Methods

# 3.1 UV-Visible Absorption

The synthesized silver nanoparticles by reducing silver metal ion solution with Aloe Vera leaves extract were initially characterized using UV-Visible Spectrophotometers (Jenway 6310 / Bibby Scientific Limited / UK).

# 3.2 Atomic Absorption Spectrometry

The initial concentration of Ag+ in the prepared AgNO3 solution and the final concentration of Ag+ in the prepared nanosilver solution were also measured using the Atomic absorption (Nov AA350 Analytik Jena AG, Germany). The difference between the initial and final concentration of Ag+ will give the concentration of Ag0 nanoparticles.

# 3.3 Atomic Force Microscopy

A thin film of the prepared sample of AgNPs was deposited on a silica glass plate by dropping few drops of the AgNPs on the plate and allowed to dry at room temperature in the dark (to avoid nanoparticles diameter growth due to temperature and/or light). The deposited film on silica glass plate was then scanned with the Atomic Force Microscopy (CSPM-5500, Karaltay (Beijing) Instruments Co. Ltd., China).

#### 3.4 Fourier-Transform Infrared Spectroscopy

The Aloe Vera leaves extract was analyzed using Fouriertransform infrared (FTIR) Spectroscopy, (IRTracer-100 / Shimadzu Co. / USA). A several drops of freshly prepared Aloe Vera leaves extract were sandwiched between two silica glass plates such that no gas bubbles are trapped.

#### 4. Results and Discussion

#### 4.1 FTIR of Aloe Vera Leaves Extract

The identification of specific compounds and functional groups in the plant extract will help in the physiochemical characterization of materials with reference to pertained biochemical and biological activities. The FTIR spectra for Aloe Vera extract show several peaks and signify various functional groups (Figure 1). The broad band at about 3452.58 cm-1 can be attributed to bond – OH groups. The band at about 2356.89 can be attributed to C=N stretching. A carbonyl group is present at 1735.93 bands. A functional carbonyls group composed of a carbon atom double-bonded to an oxygen atom C=O. The absorption bands at 1635.64 cm-1 it for N–H bond (Amine I). The peak at 1562.34 cm-1 is attributed to secondary amine groups. The peaks at 1458.18 and 1396.46 cm-1 are both related to the symmetric bending of CH3. The peak at 470.63 cm-1 correspond to stretching vibration of amine groups (Nejatzadeh-Barandozi & Enferadi 2012; Ray & Dutta Gupta 2013; Vitro *et al.* 2014).

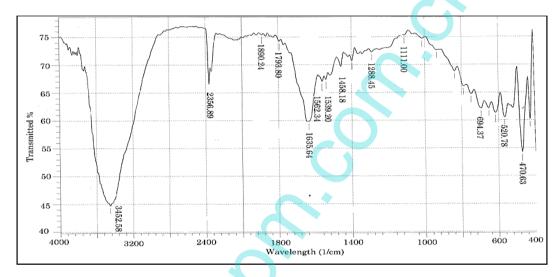
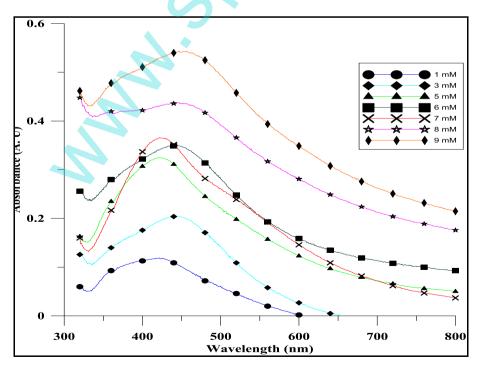


Figure 1 FTIR of Aloe Vera leaves extract



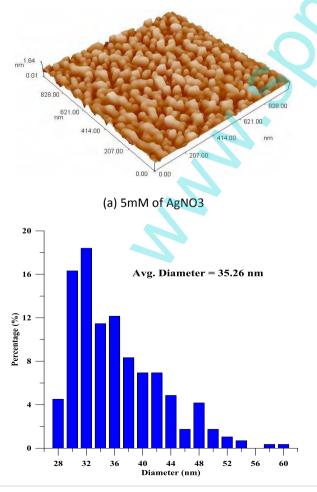
**Figure 2** UV–Visible absorbance spectra of AgNPs suspension at different AgNO<sub>3</sub> concentration using Aloe Vera leaves extract

#### 4.2 Effect of AgNO<sub>3</sub> Concentration (Molarity)

A 10 mL of Aloe Vera leaves extract was added to 90 mL of  $AgNO_3$  aqueous solution at different concentration (1, 3, 5, 7 & 9) mM with 10 min exposure to sunlight at room temperature. The solution was then characterized using UV-Vis and AFM.

Figure 2 shows the UV–visible spectra of AgNPs at different AgNO3 concentration. Increasing AgNO3 concentration resulted in gradual increasing of absorbance peak between 422 – 447 nm for (1, 3, 5, 7 & 9) mM AgNO3 which are the characteristic of Ag nanoparticle, due to its surface plasmon resonance absorption band. The color intensity increases with increasing of AgNO3 concentration. As the concentration of the AgNO3 increases, the SPR band was shifted from 422 at 1mM to 447 nm at 9mM with simultaneous broadening of the band due to the increase of AgNO3 particles size. The rate of formation of AgNPs was found to be slower at lower AgNO3 concentration and hence causing weaker absorbance.

In present investigation, a single SPR band was observed for each (1, 3, 5, 7 & 9) mM AgNO3 revealing spherical shape of silver nanoparticles. The number of SPR peaks increases with decreasing the symmetry of the nanoparticle. Thus, spherical nanoparticles, disks, and triangular nanoplates of silver show one, two, and more peaks, respectively (Sosa *et al.* 2003).



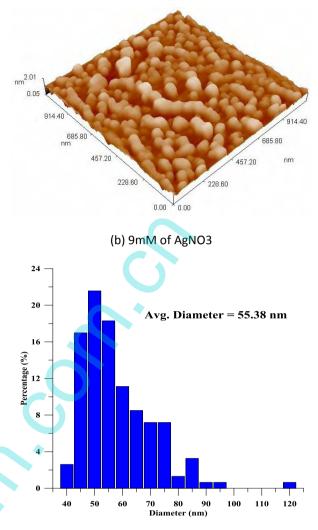


Figure 3 AFM images with nanoparticles size distribution of AgNPs synthesized using Aloe Vera plant extracts: a) 5mM AgNO<sub>3</sub> and b) 9mM AgNO<sub>3</sub>

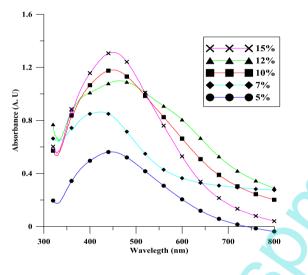
Figure 3 show the AFM images for AgNPs at different AgNO3 concentration (1, 5, 7 & 9) mM. AFM was used to analyse the particle morphology (shape, size) of AgNPs in Three- Dimensional view. Figure 3 show the AFM images for AgNPs at different AgNO3 concentration (1, 5, 7 & 9) mM.

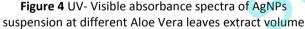
The 3D images results of AFM indicate the formation of homogeneous distribution of silver nanoparticles and no agglomeration was observed as shown in Figure 4. The Granularity Accumulation Distribution (GAD), which gives the particles size distribution of AgNPs, showed the average particle diameter of AgNPs is 34.08, 35.26, 40.22 and 55.38 nm at 1, 5, 7 and 9 mM respectively. A small change in the average particle size between 1-5 mM is observed. However, the average particle size increases rapidly with increasing in the molarity after 5mM due to the availability of more Ag+ which lead to an increase in the size of silver nanoparticles. At 9 mM, the SPR peak was observed to be broad with maximum absorbance and shifted toward longer wavelength. The shift to longer wavelengths is associated with increasing in particle size (Gunalan et al. 2013). This increase in particle size, the

broadening of the peak, and the maximum absorbance can be explained by the fact that particles are polydispersed and no sedimentation of the particles.

#### 4.3 Effect of Aloe Vera Plant Extract Volume

A 5, 7, 10, 12 and 15 mL of Aloe Vera leaves extract were added to 95, 93, 90, 88 and 85 mL of AgNO3 solution (5mM) respectively under sunlight exposure for 10 min at room temperature. Figure 4 shows the UV–visible spectra of AgNPs at different Aloe Vera leaves extract volume ratio. The SPR band for 5 mL extract was centered at 477 nm. At higher extract ratio 10 and 15 mL, the SPR band is also at 447 nm with an increase in the absorbance due to more the formation of more AgNPs caused by the availability of more functional groups in the leaf extract.





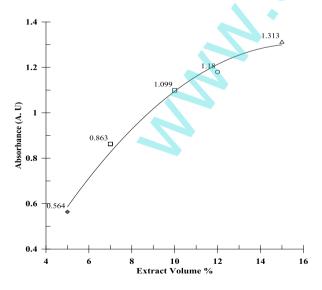
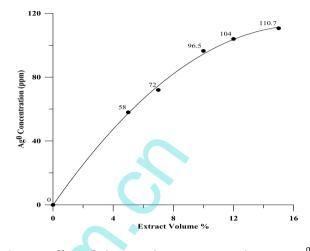


Figure 5 Absorbance as function of Aloe Vera leaves extract volume

Figure 5 shows the absorbance as function of Aloe Vera leaves extract ratio. The absorbance increases with

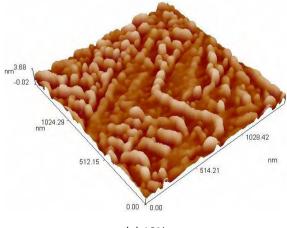
increasing the extract volume for (5, 7, 10, 12 and 15%) of Aloe Vera leaves extract as a result of increasing the concentration of nanoparticles due to the increasing of reduction sites as confirm in Figure 6.



**Figure 6** Effect of Aloe Vera leaves extract volume on Ag<sup>0</sup> concentration

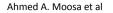
The AFM images of AgNPs synthesized by different Aloe Vera plants extract volume are shown in Figures 7. The image topography of AgNPs clearly indicates the formation of nanoparticles with no agglomerations. The average nanoparticle diameter of AgNPs was found to be 68.31 nm, 60.27 nm and 51.64 nm at 5, 10 and 15mL Aloe Vera extract volume respectively. The AgNPs particles size decreases with increases of Aloe Vera plants extract volume. This is in agreement with other researchers (Dubey et al. 2010). The increase in the Aloe Vera extract volume means increasing in the reduction sites and this will increase the rate of formation of more AgNPs and this will decrease the size of AgNPs. The AFM images are supported by UV- Visible absorbance spectra of AgNPs suspension, Figure 4 at 10% and 15% where the absorbance are very high due to the formation of more AgNPs.

Therefore, the best extract volume percent for synthesis of AgNPs using Aloe Vera leaves extract was taken at 15% extract volume based on UV-Vis spectra, amount of AgNPs, best efficiency and acceptable particle size.

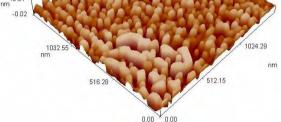


(a) 10%

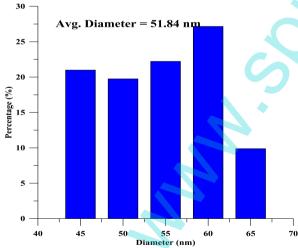
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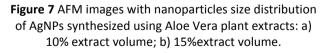


Avg. Diameter = 60.27 nm  $\begin{pmatrix} 25 \\ -20 \\ -$ 









# 4.4 Effect of Sunlight Exposure Time Using Aloe Vera Leaves Extract

Figure 8 shows UV–Visible absorbance spectra for samples at different sunlight exposure time of 5, 10, 15 min. At exposure time (5 min), the absorbance is very small with SPR peak at 440 nm with broad band. At 10 min, the absorbance is maximum with SPR at 446 nm and with a sharp peak. The color intensity increased with the

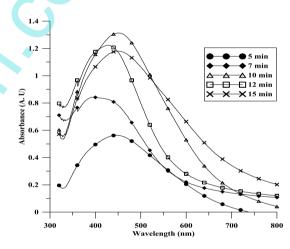
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duration of exposure time. At 15 min, the SPR band is very broad and is shifted to 447 nm with decreasing in the absorbance.

Figure 9 shows the reduction efficiency of  $AgNO_3$  by Aloe Vera leaves extract as function of sunlight exposure time. The maximum formation efficiency was 21% at 10 min of sunlight exposure time. This is in agreement with the SPR sharp peak at 10 min obtained from UV–Visible absorbance (Figure 8).

AFM images for AgNPs synthesis using Aloe Vera extract at different exposure time are shown in Figure 10 (a, b). The image shows spherical nanoparticles shape with no agglomeration and good nanoparticles size distribution. Large nanoparticles density was observed at 10 min of sunlight exposure, which is in agreement with the SPR sharp peak at 10 min obtained from UV–Visible absorbance. AFM shows the average nanoparticles diameter of 78.05 nm, 51.84 nm and 101.56 nm at 5 min, 10 min and 15 min respectively.

Therefore, the best time for sunlight exposure for synthesis of AgNPs using Aloe Vera leaves extract was taken at 10 min based on UV-Vis spectra, amount of AgNPs, best efficiency, acceptable particle size, and the best sunlight exposure time.



**Figure 8** UV–Visible absorbance spectra of AgNPs at different sunlight exposure time using Aloe Vera leaves

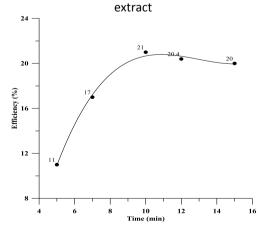


Figure 9 Reduction efficiency as function of sunlight exposure time for Aloe Vera leaves extract

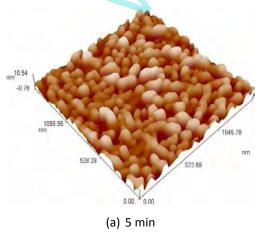
#### 4.5 Effect of Temperature using Aloe Vera Leaves Extract

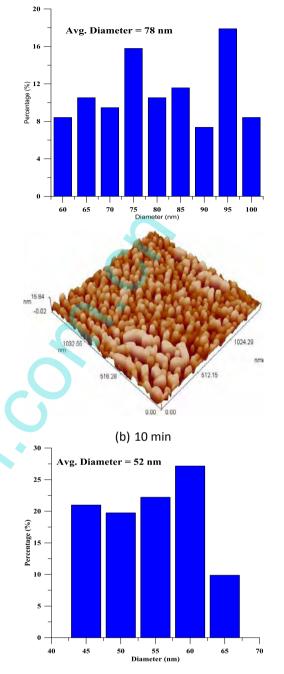
The UV-Vis spectra for AgNPs suspension at different temperature are shown in Figure 11. The absorbance peak for AgNPs occurs at 460 nm at room temperature, 40 and 80°C. The absorbance peak is 480 nm for synthesis at 50 and  $60^{\circ}$ C with a shift toward the longer wavelength. At 70°C the absorbance peak is at 450 nm which is narrow peak with an increase in absorbance due to increasing number of nanoparticles formed as a result of reduction of silver ions present in the aqueous solution. Figure 12 shows the AgO concentration in solution was increased initially from 110.5 ppm at room temperature then reaches maximum concentration of 152 ppm at temperature 60°C. The concentration was then decreased sharply with increased of temperature after 60 oC. This is in agreement with the maximum absorbance that occurs at 60°C.

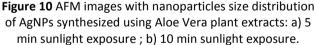
AFM images for AgNPs synthesis using Aloe Vera leaves extract at different temperature are shown in Figure 13 (a, b,). The images show a spherical nanoparticles shape with no agglomeration but at 80 oC there are some aggregations of nanoparticles. The higher particle density occurs at 60 oC. This is in agreement with UV–Visible absorbance spectra of AgNPs suspension at 60 oC where the SPR peak is very narrow which indicates uniform nanoparticles distribution. The average nanoparticles diameter is 78.89, 82.41, 85.76, 90.53 and 101.64 at 40, 50, 60, 70 and 80°C respectively.

The particles diameter distributions for 40, 50, 60 and 70°C are 50 - 130 nm, while for sample at 80°C the range is 80-175 nm with existing of 1.12% of particles with diameter 175 nm. This is in agreement with UV–Visible absorbance spectrum at 80°C, Figure 11, with the broadening peak area of the band and lower absorbance indicates decrease in interparticle spacing due to particle aggregation (Desai *et al.* 2012).

Therefore, the best condition for synthesis of AgNPs using Aloe Vera leaves extract was taken at temperature 60 oC for synthesis of AgNPs using Aloe Vera leaves based on UV-Vis spectra, amount of AgNPs, best efficiency, acceptable particle size, and the best sunlight exposure time.







#### Conclusions

Silver nanoparticles were synthesized by green method using Aloe Vera leaves extract. AgNPs synthesis under sunlight is very efficient method compare with that in the dark. The green synthesis of AgNPs method provides a simple, efficient, environmental ecofriendly and cost effective route for the synthesis of nanomaterials with tunable optical properties directed by particle shape. The synthesized silver nanoparticles was confirmed by the change of color of Aloe Vera leaves extract and characterized by UV-Vis spectroscopy.

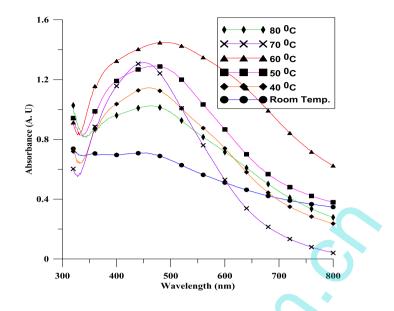


Figure 11 UV–Visible absorbance spectra of AgNPs suspension at different temperature using Aloe Vera leaves extract

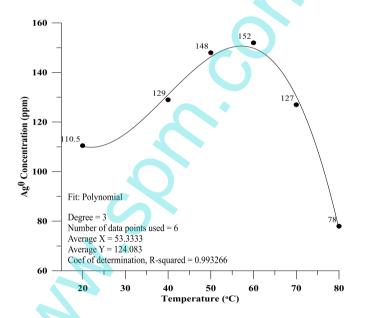
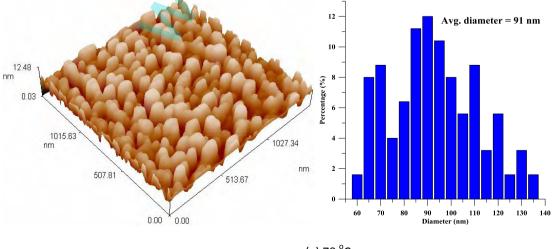


Figure 12 Ag<sup>0</sup> concentration as a function of temperature using Aloe Vera leaves extract



(a) 70 °C

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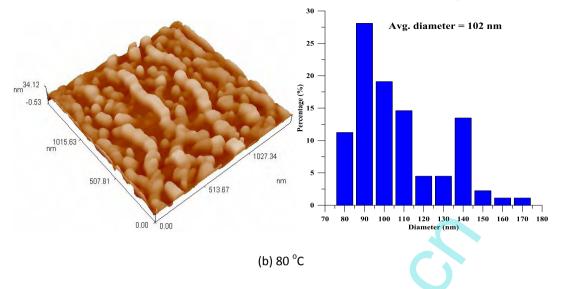


Figure 13 AFM images with nanoparticles size distribution of AgNPs synthesized using Aloe Vera plant extracts: a) 70 °C ; b) 80 °C

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