

AG THIN FILMS FROM *PELARGONIUM ZONALE*
LEAVES VIA GREEN CHEMISTRY

Nora Elizondo-Villarreal¹, ✉, Rubén O. Torres-Barrera¹, Rodrigo Arriaga-Garza¹,
Luz-Hypatia Verástegui-Domínguez¹, Rodolfo Cortés², Víctor M. Castaño³

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Abstract. Ag thin films were successfully produced via a green chemistry method from silver nanoparticles (AgNPs) obtained from reacting an extract of *Pelargonium Zonale* leaves with silver nitrate. The ions of silver nitrate were reduced to silver atoms by reducing stabilizer-capping compounds contained in the extract of *Pelargonium Zonale* leaves. The obtained atoms nucleate in small clusters that grew into nanoparticles and finally, they formed a homogeneous silver thin film on a glass substrate. The nanostructured thin films obtained were characterized by profilometry, X Ray Diffraction, Atomic Force Electronic Microscopy, UV-Vis, and Transmission Electron Microscopy.

Keywords: silver nanoparticles, nanoparticle synthesis, *Zonale* leaves, green chemistry method, silver thin films.

1. Introduction

Nowadays, thin films containing metal nanoparticles have been used as antimicrobial coatings,^{1,2} catalysts,^{3,4} as well as electrochemical,^{5,6} photovoltaic,^{7,8} and optical,⁹ devices. These applications became possible thanks to the metal nanoparticles of small size, which impart unique properties such as absorption plasmon resonance band and high surface-area-to-volume ratio in a variety of processes that occur at nanometer scales.¹⁰ Several forms for the preparation of metal nanoparticle films on solid substrates were already reported, such as sol-gel deposition of metal ions followed by thermal treatment,¹¹ vapor-deposition methods,¹² and also, the deposition of metal nanoparticles from colloidal dispersions.¹³⁻¹⁸

The synthesis of nanostructures by this method is attracting increasing attention to an energy-efficient, hazardous waste-free approach and, above all, it opens alternative routes for the efficient, environmentally-friendly synthesis of novel compounds at the nanoscale. Accordingly, there already exist reports on the use of plants extracts, including *Rosa Berberifolia*, *Geranium Maculatum*, *Aloe Barbadensis*, *Curcubita Digitata*,¹⁹ *Chenopodium Album*,²⁰ sugar beet pulp,²¹ Pear fruit extract,²² *Tansy Fruit*,²³ *Jatropha Curcas*,²⁴ *Lemon Juic*,²⁵ fungi,^{26,27} etc., to obtain various types of isolated nanoparticles.²⁸ However, much less attention has been dedicated in the literature to the production of more complex structures, such as thin films, which involve not only the synthesis of isolated nanoparticles, but the controlled coalescence of nanostructures to be able to produce homogeneous, smooth films.

Among all the nanostructured, thin films have found practical applications in today's technology. Ag thin films have a wide range of applications in electric devices, electric circuits, photodetectors, optical coatings, gas sensors, etc.²⁹⁻³³ Accordingly, this present manuscript reports the low temperature (363–368 K), ambient pressure, environmentally-friendly synthesis of thin films of Ag with the aid of *Pelargonium Zonale* leaves,^{34,35} known popularly as geranium (Fig. 1), along with their nanostructural characterization.^{36,37}

The *Pelargonium Zonale* leaf extract works as a biochemical system for the synthesis of Ag-NPs. In plants, the abundant compounds are ascorbic acid, polyphenols, terpenoids, alkaloids, alcohols, and flavonoids. They are well known as antioxidants in plants and as well as specifically *Pelargonium Zonale* species. For this reason, they are used as reducing as well as capping, and stabilizing agents for a large amount of synthesis of silver nanoparticles.³⁸⁻⁴⁵

The biosynthesis of silver nanoparticles depends on a series of factors like time of reaction, temperature, and pH; the selected extract is very important because it contains chemical compounds that function as stabilizers, capping and bio-reducing agents and it is abundant in California U.S.A. and Mexico.⁴⁵⁻⁴⁶

¹ Research Center of Physical and Mathematical Sciences (CICFIM), Physical and Mathematical Sciences School, Universidad Autónoma de Nuevo León (UANL), San Nicolás de los Garza, N. L., México. Código Postal: 66455

² CICESE, Monterrey, PIIT, Apodaca, N. L., México

³ Centro de Física Aplicada y Tecnología Avanzada, Universidad Nacional Autónoma de México, Boulevard Juriquilla 3001, Querétaro 76230, México

✉ nelizond@yahoo.com; vmcastano@unam.mx

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Fig. 1. *Pelargonium Zonale* is commonly known as Geranium

The thin films were characterized by profilometry as a function of film thickness, X-Ray Diffraction (XDR), Atomic Force Electronic Microscopy, UV-VIS, and Transmission Electronic Microscopy.

The thin films show a highly homogeneous nano-scale structure. Nanostructured silver particles exhibit unique optical characteristics. In contrast to their corresponding bulk counterparts, metallic nanoparticles can absorb electromagnetic radiation, resulting in surface plasmon polaritons at the metal-dielectric interface. The resonance wavelength of metallic nanoparticles is strongly dependent on the metal, as well as the particles size and shape.⁴⁷

The synthesis of such nanocomposites typically involves the deposition of metallic nanoparticles into a dielectric matrix. The electrical properties of such metal/dielectric nanocomposites depended on the embedded metallic nanostructures.⁴⁸

This work was performed successfully by the green chemistry of the metallic film structures, which are a system of metallic nanoparticles in a dielectric medium of a glass substrate and vacuum spacing between particles at a nanometric scale.

2. Experimental

The *Pelargonium Zonale* is a common plant for its abundant and beautiful flowering. The plants were placed

in a place in some semi-shade because the weather was hot at North East of México in The State of Nuevo León.

The leaves of *Pelargonium Zonale* specie were provided from Biological Sciences School of Universidad Autónoma de Nuevo León in San Nicolás de los Garza, Nuevo León México. The classification of species *Pelargonium Zonale* is: Taxon: *Pelargonium Zonale* (L.) L'Hér, Accession: CPG 8164, Boucher: BM.

First of all, the plants were harvested in spring when it reproduces easily, healthy leaves were collected in April. The leaves were washed with deionized water four or five times and dried at room temperature at 303 K.

After that, five (5 g) of as-collected *Pelargonium Zonale* (Fig. 1) air-dried leaves were cut into small pieces, washed with deionized water, and placed into a 50 mL flask with 25 mL of deionized boiling water (Fig. 2) for 3 minutes. As a result, a green extract was obtained, which was cooled down under room conditions.

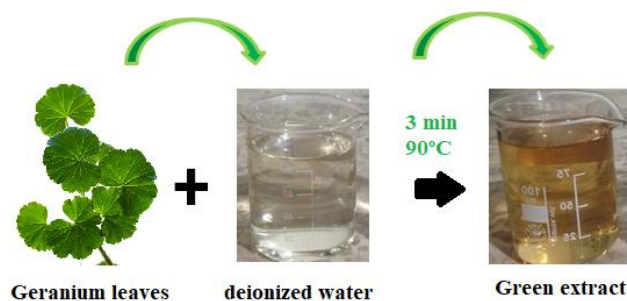


Fig. 2. Green extract of geranium

Thus, silver nitrate (AgNO_3) (99.0 % purity from Sigma Aldrich) was used to prepare a solution of 0.001 mol in deionized water.

Then, 25 mL of this solution were mixed with 1.25 ml of the *Pelargonium Zonale* extract, along with a glass plate (as a substrate for thin films) and the reaction solution was heated at 368 K until all the water was evaporated. The whole process is schematically depicted in Fig. 3. Silver nanoparticles were deposited onto glass substrates.

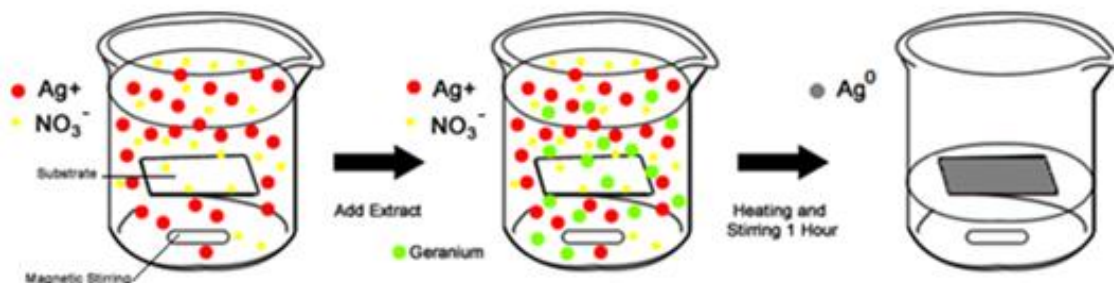


Fig. 3. Schematic process for preparing Ag thin films

This work has evidence that the thickness of Ag thin film can be controlled by the concentration and time. The experiments were carried out, varying these process parameters respectively from 0.001 to 0.007 mol of silver nitrate reagent at a constant time (1 h); and reaction time from 0.5 hours to 3.5 hours at the same concentration of the *Pelargonium Zonale* extract of 1.25 mL and a constant concentration of AgNO₃ of 0.001 mol. These experiments were performed also at 368 K.

The thin films prepared were dried at 323 K and stored for further characterizations.

3. Characterization

X-ray diffraction

Structural characterization was carried out by X-ray diffraction in a Rigaku Miniflex II Desktop diffractometer with CuK α ($\lambda = 0.15418 \text{ \AA}$) radiation at a generator voltage of 40 kV and a generator current of 17.5 mA. X-ray diffraction data were collected within the 2θ range between 2° and 90° , at a scan rate of $0.01^\circ/\text{second}$.

UV-Vis analysis

UV-vis characterization was carried out with the help of a special holder for thin films at different time and concentration intervals and the absorption maxima were scanned in a wavelength range from 300 to 800 nm at different time intervals in a Thermo Scientific Evolution 600 UV-vis apparatus.

Profilometry

Thin Film thickness measurements were performed on a DekTak mechanical profilometer.

Atomic Force Microscopy (AFM)

The surface morphology of films and roughness was measured from images collected using an Atomic Force Microscopy Agilent Technologies model 5500 SPM in acoustic mode. The visualization of two- and three-dimensional thin films was possible using this technique. The ratio between the amplitude of free oscillation and the amplitude maintained for one scan was kept constant near 50 %, minimizing the effects of adhesion and maximizing the effects of viscoelasticity. Images of an area of $0.4 \times 0.4 \text{ \mu m}$ were obtained.

Transmission Electron Microscopy

Additional characterization was performed to have enough information about the size and morphology of particles at a nanometric scale. The technique of transmission electron microscopy (TEM) was used. For the transmission electron microscopy analysis of the silver NPs, the stable silver nanoparticles were washed, diluted, and ultra-sonicated.

One drop of diluted silver nanoparticles was placed onto carbon-coated copper TEM grids and vacuum-dried. High-Resolution Transmission Electron Microscopy

(HRTEM) images were taken at an accelerating voltage of 200 kV Field Emission Gun (FEG) high-resolution transmission electron microscope (FEI Tecnai microscope G2 FEI).

4. Results and Discussion

The *Pelargonium Zonale* leaves extract works as a biochemical system for the synthesis of Ag-NPs. The abundant compounds in plants are ascorbic acid, polyphenols, terpenoids, alkaloids, alcohols, and flavonoids. They are well-known as antioxidants in plants and specifically *Pelargonium Zonale* species. For this reason, they are used as reducing as well as capping, and stabilizing agents for a large amount of synthesis of silver nanoparticles.³⁸⁻⁴⁵

As an example of a route of reduction: Ascorbic acid is present at high levels in all parts of plants. It is a reducing agent and it neutralizes, leading to the formation of ascorbate radical and one electron.

The free-electron itself reduces the Ag⁺ ions to Ag⁰ as can be seen in Scheme 1A.

The mechanism proposed for the growth of the silver nanoparticles by green chemistry is a three-step mechanism. In the first step, the silver ions interact with compounds of leaves extracts, in the second step the silver ions that are in the solution are reduced to silver atoms, which form the crystallization nuclei by the contact with the reducing agents of *Pelargonium Zonale* extract; nearby silver atoms then aggregate at close range and they form clusters which grow. These clusters are the primary nanoparticles. Finally, these primary nanoparticles interact with other nanoparticles to form larger clusters. Then the polymers of extract function act as stabilizers around the nanoparticles for the final stabilized nanoparticles, as can be seen in Scheme 1B.

During the biosynthesis of silver nanoparticles, the color changes of thin films were detected from pale yellow to blackish-brown directly by the naked eye.

The experiments were carried out without stirring to favor the formation of thin films.

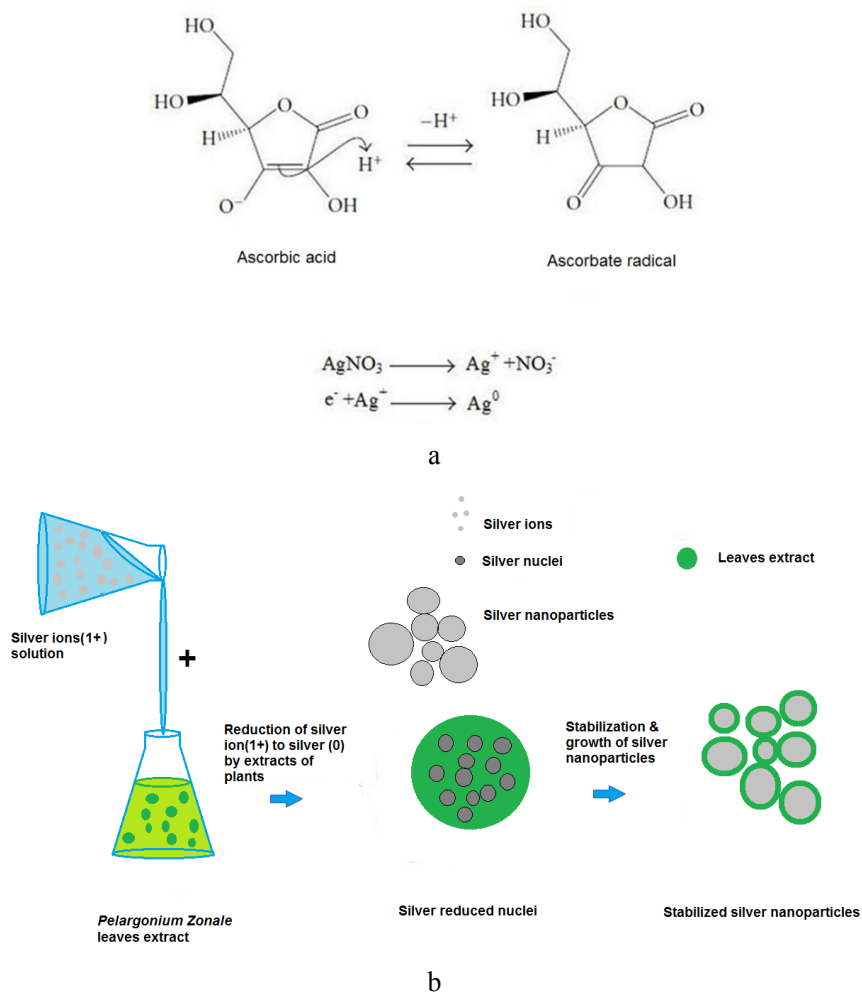
At the first 15 min of the formation of nanoparticles, an Ostwald ripening effect was observed. This thermodynamic spontaneous process occurs during the growth of nanoparticles because large nanoparticles are more energetically favored than smaller nanoparticles.⁴⁹ This stems from the fact that silver atoms on the surface of a nanoparticle are energetically less stable than the ones in the interior.

In the experiments, *Pelargonium Zonale* extracts and silver nitrate reacted in the solution and the solutions

started turning brown in 30 min (without stirring) and then they turned dark. This is because the vibrant colors of plasmonic nanoparticles occur by the conduction electrons on the surface of each nanoparticle vibrating when they are excited by light at a specific wavelength. These vibrations result in extremely specific bright colors that can be tuned by changing the nature of the metallic particle (silver), particle size, shape, and composition.⁴²

The nanostructured Ag thin films obtained are depicted in Fig. 4. It is interesting to point out that the thickness of the Ag thin films can be easily controlled by the silver nitrate concentration, as observed in Fig. 4.

It must be pointed out that the thickness of resulting nanostructured thin films was controlled stably by the change of AgNO₃ solution concentration in time, as can be seen in Figs. 5 and 6.



Scheme 1. a) Ascorbic acid reduction mechanism of silver ions to obtain Ag⁰ NPs (a); mechanism of nucleation, stabilization, and growth of silver nanoparticles (b)

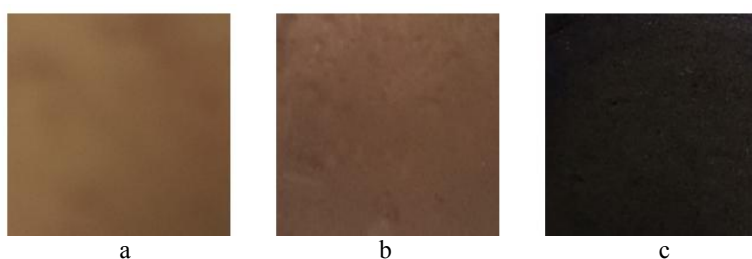


Fig. 4. Ag thin films: with half concentration of silver nitrate (0.001 mol) (a); with a concentration of silver nitrate of 0.002 mol (b); with twice the concentration of silver nitrate of 0.004 mol (c)

It is evident from Fig. 5 that when the concentration of silver nitrate (AgNO_3) solution increases, the thickness of silver films could be controlled and it increases with some regularity at a constant time of one hour.

When the time increases, the thickness of silver films could be controlled and it increases also at a constant molar concentration of 0.001 mol (Fig. 6).

Fig. 7 depicts the X-ray diffraction pattern of Ag thin film, showing reflections at 38.52° , 44.49° , 64.70° , and 77.63° , corresponding precisely to the [111], [200], [220], and [311] Ag crystalline planes. Scherrer's equation confirms that the broadening of the X-ray reflections corresponds to an average crystallite size of a few nm (below 6 nm).

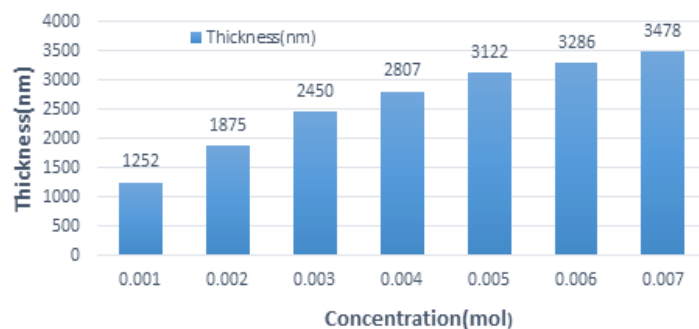


Fig. 5. The thickness of nanostructured silver thin film versus concentration of silver nitrate

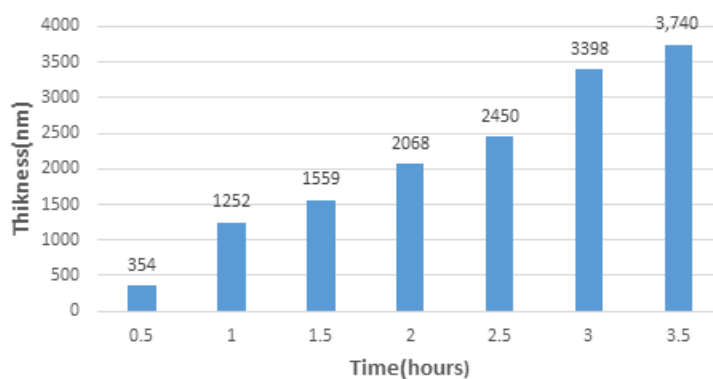


Fig. 6. The thickness of nanostructured silver thin film versus time of deposition

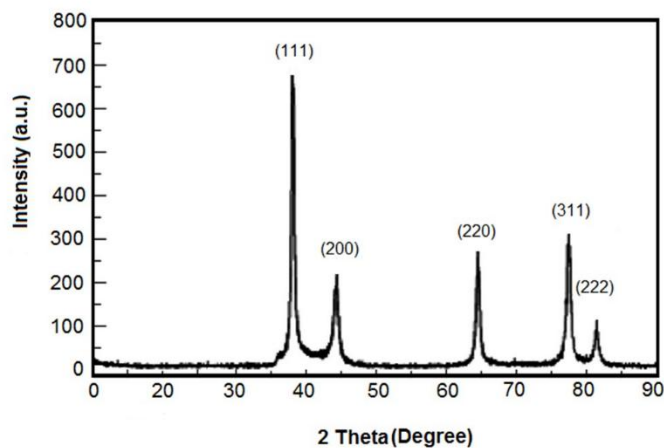


Fig. 7. X-ray diffraction pattern of Ag thin film of silver nanoparticles biosynthesized by treating silver nitrate with *Pelargonium Zonale* leaf extract

The UV-VIS spectra show the presence of pure Ag in the range of 250-550 nm, with the common plasmon peak of silver nanoparticles at 445 nm. The maximum plasmon peak of the sample is at 425 nm, as shown in Fig. 8, which was consistent with Rasheed *et al.*⁴²

UV Vis Spectroscopy confirmed the bio-reduced samples from Ag^+ to Ag^0 .

Atomic Force Microscopy reveals that the roughness of the films was below 50 nm, the roughness average of the film was 5.7496 nm, and the root mean square (RMS) roughness was 7.4847 nm, which demonstrates the capability of the technique for producing uniform, smooth Ag films as showed in Figs. 9A and 9B.

The process of silver nanoparticles deposition to prepare thin films consisted of three steps: the ions of silver nitrate first were reduced to silver atoms by reducing stabilizer-capping agents contained in the extract of *Pelargonium Zonale* leaves. The obtained atoms nucleate in small clusters that grew into nanoparticles and finally, they formed a homogeneous thin film with silver nanoparticles on a glass substrate.

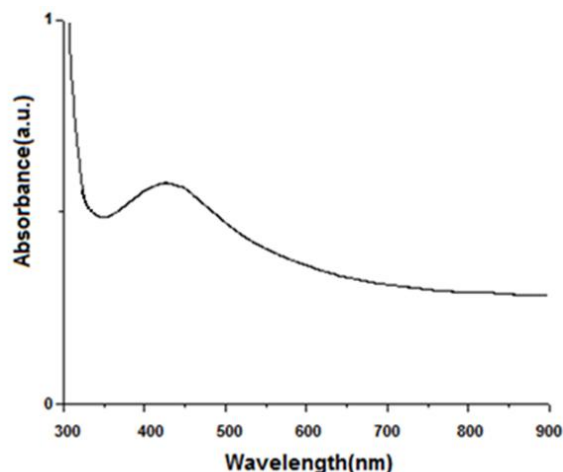


Fig. 8. UV-vis spectrum of the Ag thin film

The particle size was determined with greater precision by transmission electron microscopy in Figs. 10A and 10B.

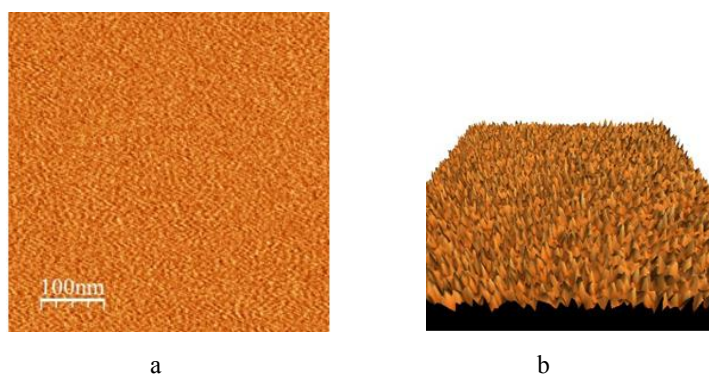


Fig. 9. AFM images of Ag thin film, 2D view in an area (a) of $0.4 \mu\text{m} \times 0.4 \mu\text{m}$ and Ag thin film, 3D view (b)

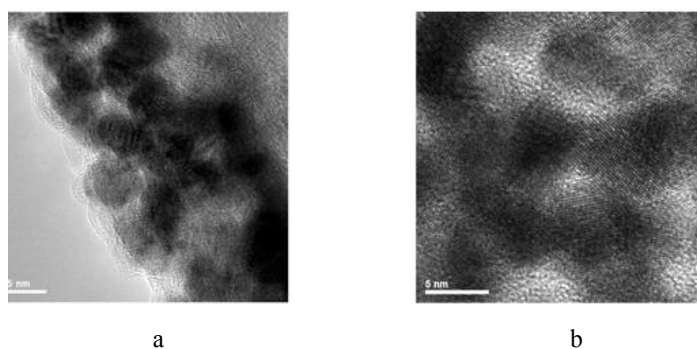


Fig. 10. High-Resolution TEM images (a) and (b) of silver nanoparticles synthesized via a green chemistry method with an extract of *Pelargonium Zonale* leaves and silver nitrate at a magnification of five nm at 368 K

Figs. 10A and 10B show that the silver nanoparticles crystallized in the face-centered cubic (fcc) symmetry of bulk silver.

The nanometric scale particles had a quasi-spherical shape and a size approximately of six nm in diameter, which coincides with the calculated diameter by a Scherrer's equation in this work.

5. Conclusions

The present green method for thin films preparation using silver nanoparticles leads to a significant reduction of the silver thin film costs, as it avoids high temperatures, vacuum, or other processes traditionally used in the fabrication of thin films. Besides its low cost, it combines the ease of fabrication and high efficiency. This method allowed the production of homogeneous nanoscale-level smooth Ag thin films, in which the thickness of the film can be controlled easily by the time and concentration of the biosynthesis reaction. The morphological characteristics of the resulting nanostructured films were adequate for exploration in the electronics industry.

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ТОНКІ ПЛІВКИ Ag З ЛИСТЯ *Pelargonium Zonale* ЗА ДОПОМОГОЮ ЗЕЛЕНОЇ ХІМІЇ

Анотація. Тонкі плівки Ag успішно одержано за допомогою методу зеленої хімії з наночастинок срібла, отриманих через реакцію екстракту листя *Pelargonium Zonale* з нітратом срібла. Іони нітрату срібла були відновлені до атомів срібла через відновлення речовинами-стабілізаторами, які містяться в екстракті листя *Pelargonium Zonale*. Отримані атоми нуклеюються в невеликі кластери, які виростають у наночастинки, і, нарешті, утворюють однорідну тонку плівку срібла на скляній підкладці. Отримані наноструктуровані тонкі плівки були охарактеризовані за допомогою профілометрії, дифракції рентгенівських променів, атомно-силової електронної мікроскопії, УФ-спектроскопії та трансмісійної електронної мікроскопії.

Ключові слова: наночастинок срібла, синтез наночастинок, листя *Zonale*, метод зеленої хімії, тонкі плівки срібла.