

Microwave Synthesis of Silver Nanoparticles Using Different Pentose Carbohydrates as Reducing Agents

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Abstract: A fast, green and readily reproducible microwave-based method for the production of high quality silver nanoparticles (AgNPs) in high yield is presented. Starch is used as a stabilizing agent with few pentose different reducing carbohydrates as D-ribose, D-arabinose and L-arabinose. From the UV-vis peak profile spectra of the solutions of the silver nanoparticles, the authors have investigated the size of the NPs together with the average diameter, shape, and aggregation state of the colloidal AgNPs. TEM measurements and EDX analysis have confirmed the morphology of our AgNPs.

Key words: Microwave synthesis, UV-vis spectroscopy, Mie theory, silver nanoparticles, TEM and EDX.

1. Introduction

It has been a long time since metal nanoparticles are increasingly used in various fields, among the other medical, and industrial processes, due to their unique physical and chemical properties. These include optical [1], electronic [2], and thermal, high electrical conductivity, and biological properties [3].

In particular silver nanoparticles (AgNPs) have received recognition due to its potential applications in various fields such as photonics, microelectronics, photo catalysis, lithography, and biosensor material [3-7]. In addition, silver nanoparticles are known for their antimicrobial properties and even have shown to prevent HIV binding to host cells [8, 9]. The noble metal nanoparticles have a surface plasmon resonance absorption in the UV-Visible region [10]. The surface

plasmon band arises from the coherent oscillation of conduction band electrons relative to the lattice of metal ions.

This optical property varies as a function of size, shape, and porosity of the nanostructures [11]. The chemical and physical properties of metal nanoparticles are highly dependent on their size and shape in the nanoscale regime [12]. In recent years, a number of methods have been explored to synthesize silver nanostructures. These include chemical reduction [13], polyol process [14], electrochemical [15], sonochemical [16], and biological [17, 18] methods. Among these, the most popular method for the preparation of the Ag colloid is the chemical reduction of silver salt in the presence of a stabilizing agent. The most commonly used stabilizing agents are polymers [18] and surfactants [19].

In order to minimize pollution to the environment, ecofriendly materials are being explored in the green

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Table 1 Identifications for chemicals used in these laboratory experiments.

IUPAC name	CAS number	Chemical formula
Silver nitrate	7761-88-8	AgNO ₃
D-ribose	50-69-1	C ₅ H ₁₀ O ₅
D-arabinose	10323-20-3	C ₅ H ₁₀ O ₅
L-arabinose	87-72-9	C ₅ H ₁₀ O ₅
Starch from corn	9005-25-8	(C ₆ H ₁₀ O ₅) _n

synthesis [20] of AgNPs. In one study [21] stable AgNPs were prepared by using soluble starch as both the reducing and the stabilizing agent. Other green reagents reported were β-D-glucose [22] and chitosan [23], which can act both as reducing and stabilizing agents for the preparation of Ag nanostructures.

Recently, microwave heating has been explored as a promising technique for nanoparticle synthesis [24]. The microwave irradiation generates very fast nucleation sites in the solution, which significantly enhances the reaction rate. The advantage of microwave-mediated synthesis over the conventional heating is the improved kinetics of reaction (generally by one or two orders of magnitude due to rapid internal heating [25]). Many successful reports based on microwave-assisted synthesis of AgNPs were published in recent years [26-30].

The present work reports a rapid route to obtain AgNPs with environmentally benign materials: ribose, and arabinose as the reducing agents in aqueous surfactant media via microwave-assisted heating method with starch as the stabilizing agent.

2. Experimental Section

2.1 AgNPs Preparation

The authors used for all reaction a household microwave PANASONIC, stainless steel, 1,300 W, power input 120 V, 60 Hz for different interval time.

(1) 25.0 mL aqueous solutions of AgNO₃ 1.0 × 10⁻² M, 25.0 mL of D-ribose 0.1 M were mixed together with 25.0 mL of corn starch solution (0.02% in weight) to form a total volume of 75.0 mL inside an Erlenmeyer flask. The flask was heated in the microwave oven for 2 minutes, 3 minutes, 4 minutes,

5 minutes, 7 minutes, 8 minutes using a higher power setting.

(2) 25.0 mL aqueous solutions of AgNO₃ 1.0 × 10⁻² M, 25.0 mL of D-arabinose 0.1 M were mixed together with 25.0 mL of corn starch solution (0.02% in weight) to form a total volume of 75.0 mL inside an Erlenmeyer flask. The flask was heated in the microwave oven for 2 minutes, 3 minutes, 4 minutes, 5 minutes using a higher power setting.

(3) 25.0 mL aqueous solutions of AgNO₃ 1.0 × 10⁻² M, 25.0 mL of L-arabinose 0.1 M were mixed together with 25.0 mL of corn starch solution (0.02% in weight) to form a total volume of 75.0 mL inside an Erlenmeyer flask. The flask was heated in the microwave oven for 1 minutes, 2 minutes, 3 minutes, 4 minutes using a higher power setting.

2.2 Tyndal Effect Determination

A laser pointer (Figs. 1 and 2) has been used to demonstrate the Tyndall effect for colloidal suspensions [31] and to provide a qualitative investigation of absorption and scattering components of the LSPR (localized surface plasmon resonance).

2.3 UV-vis Determination

Data were collected at room temperature with a Shimadzu UV-1280 spectrophotometer from 200 nm to 900 nm. A 1.0 mL glass cuvette with 1 cm light path was used for all scanning (Figs. 2-5)

2.4 TEM Determination

High-resolution Transmission Electron Microscope (LaB₆ TEM) JEOL JEM 2100 TEM at 200 kV was used to determine the shape and size of AgNPs. 400 mesh copper TEM grid (Ted Pella Inc.) was used for

supporting sample measurements (Figs. 6-8 and 10). TEM samples were prepared allocating 5 μ L of the sample solution onto a carbon-coated copper grid and drying in air.

2.5 EDX Determination

High-resolution EDS: Oxford (INCA 100) system JEOL JEM 2100 TEM at 200 kV was used to determine the presence of AgNPs. Samples were prepared using the same procedure adopted for TEM measurements (Fig. 11).

3. Results and Discussion

3.1 Optical Properties

Peak position and sharp intensity of the absorption spectrum from the UV-vis (Figs.3-5) has revealed the presence of AgNPs and it was confirmed by the TEM results of the Ag NPs. The shapes of the absorption curve suggest that the nanoparticles were well dispersed and spherical in shape. Figs. 3-5 show our AgNPs with a strong absorption around 400-410 nm as expected from the literature [35, 36] from all our reducing agents D-ribose, D-arabinose and L-arabinose. The solutions were observed to change from colorless to transparent yellow, and then to dark

yellow indicating the formation of larger number of NPs with increased heating time (Fig. 1). There is only one symmetric absorption peak at 410 nm, which is the characteristic surface plasmon resonance of spherical AgNPs [2]. According to Mie theory, spherical Ag NPs exhibit single surface plasmon band, while dispersed anisotropic particles such as dendrites, prism, rod and triangles exhibit two or three LSPR (localized surface plasmon resonance) bands.

3.2 Particle Morphology

TEM (Transmission electron microscopy) is a powerful tool that has been extensively used to investigate the morphologies and size distribution of the synthesized AgNPs. Figs. 6-10 and Fig. 11 show a typical TEM image and EDX image of AgNPs synthesized by microwave irradiation for 2 minutes at 1,300 W using starch and D-ribose. Particle size distribution of the prepared nanoparticles taken from a large number of particles shows the particles range in size from 15 to 80 nm with mean size of 30.5 nm. EDX data are clearly showing the presence of silver together with a small amount of carbon and oxygen due D-ribose not reduced and starch in solution. The copper signal is due to the copper TEM grid we used as sample holder.

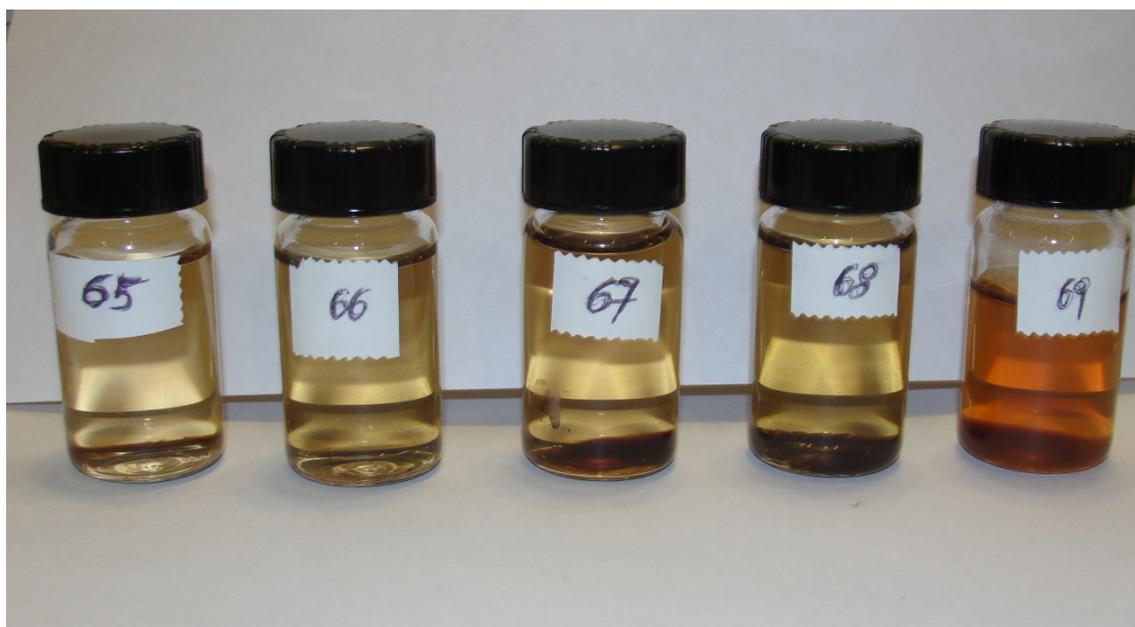


Fig. 1 Image of AgNPs synthesized by microwave irradiation from D-ribose for 2, 3, 4, 5 minutes at 1,300 W.

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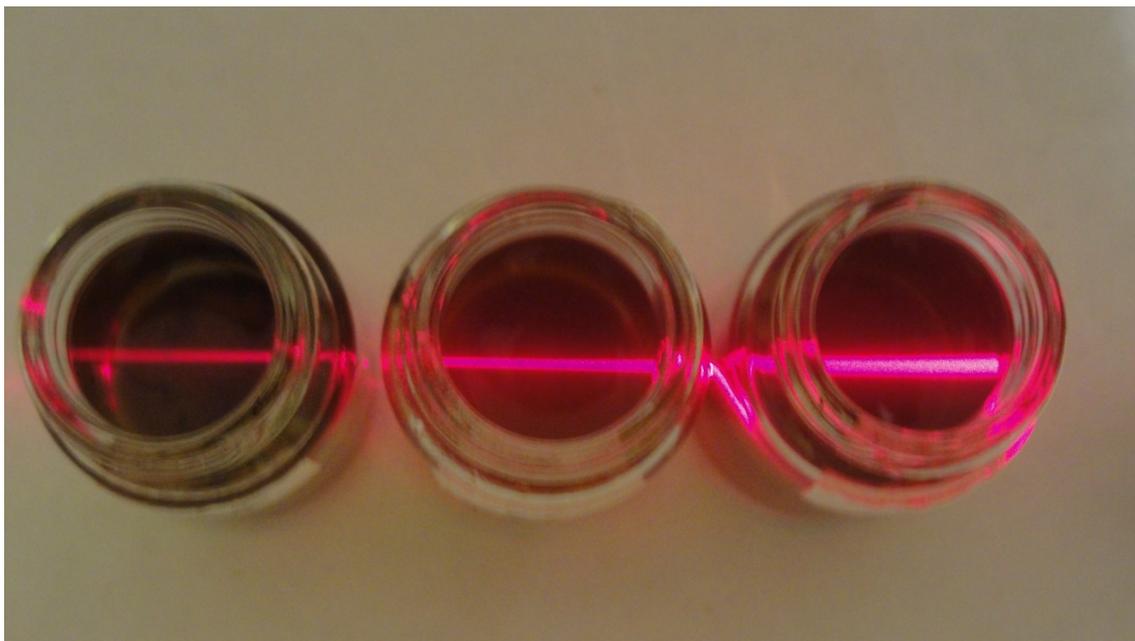


Fig. 2 Image of AgNPs synthesized by microwave irradiation from D-ribose for 2, 3, 4, 5 minutes at 1,300 W facing a laser pointer to demonstrate the Tyndall effect.

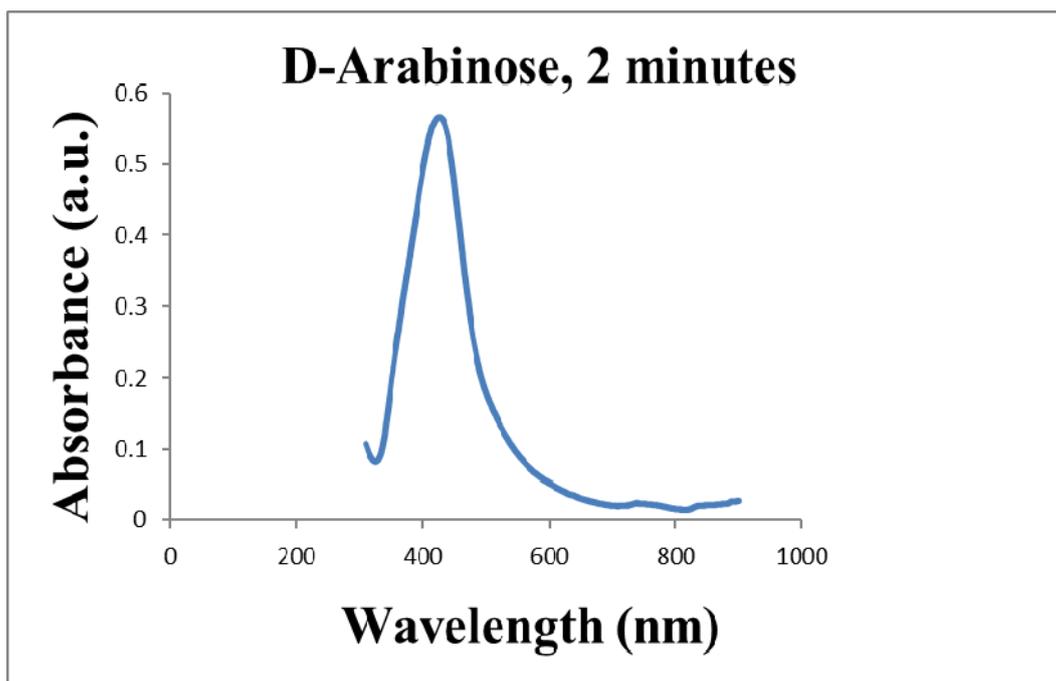


Fig. 3 Representative UV-vis spectra for silver nanoparticles synthesized from D-arabinose in domestic microwave after 2 minutes.

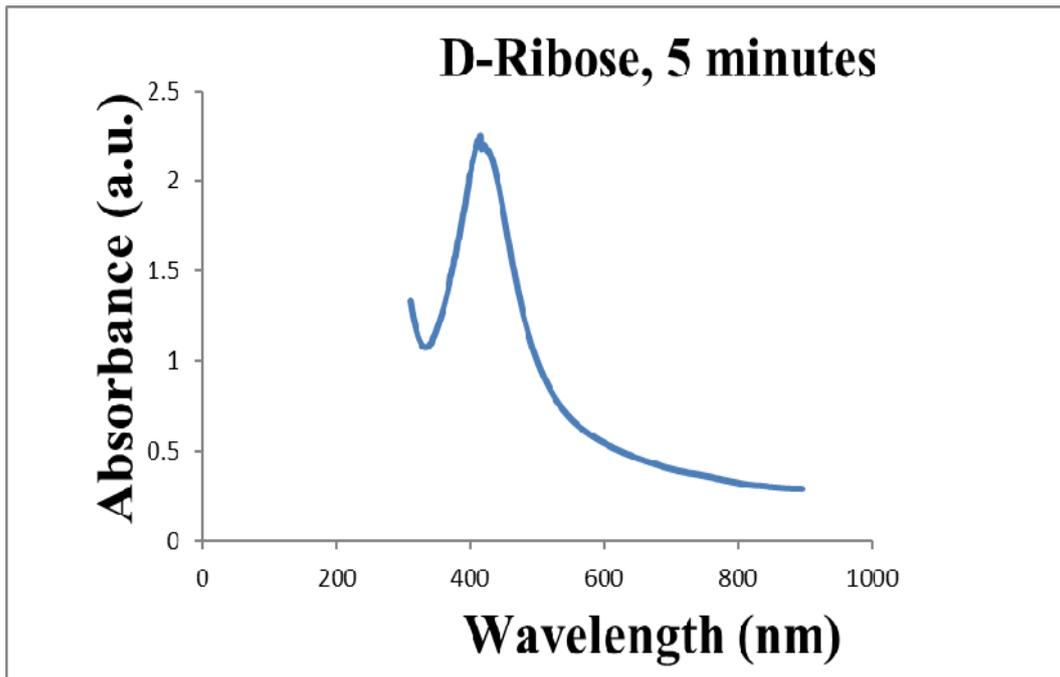


Fig. 4 Representative UV-vis spectra for silver nanoparticles synthesized from D-ribose in domestic microwave after 5 minutes.

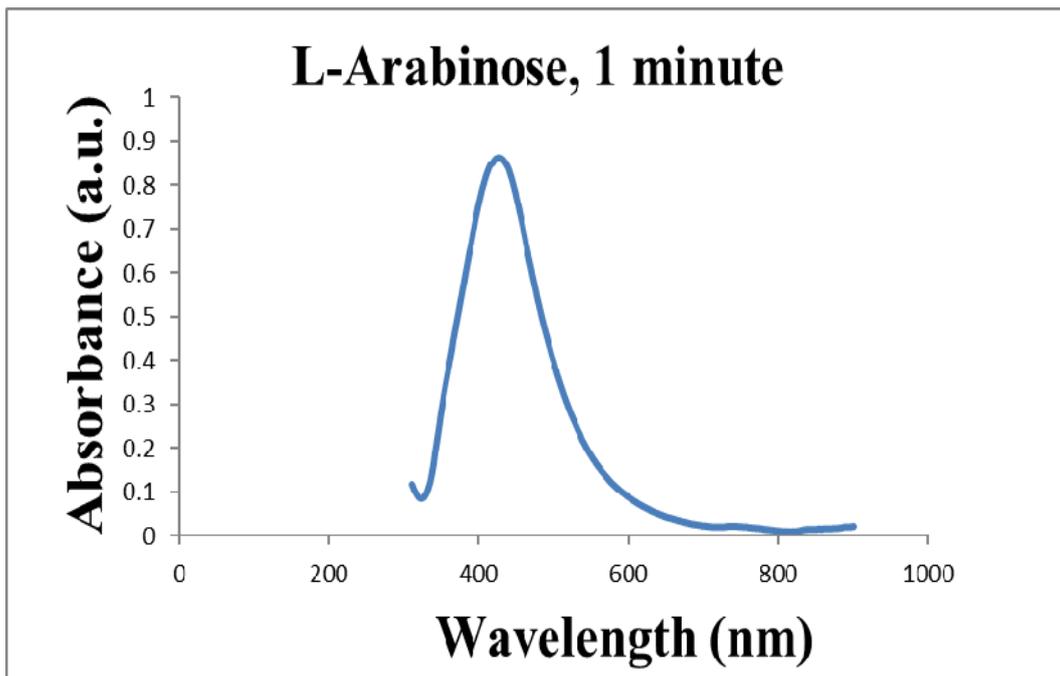


Fig. 5 Representative UV-vis spectra for silver nanoparticles synthesized from L-arabinose in domestic microwave after 1 minute.

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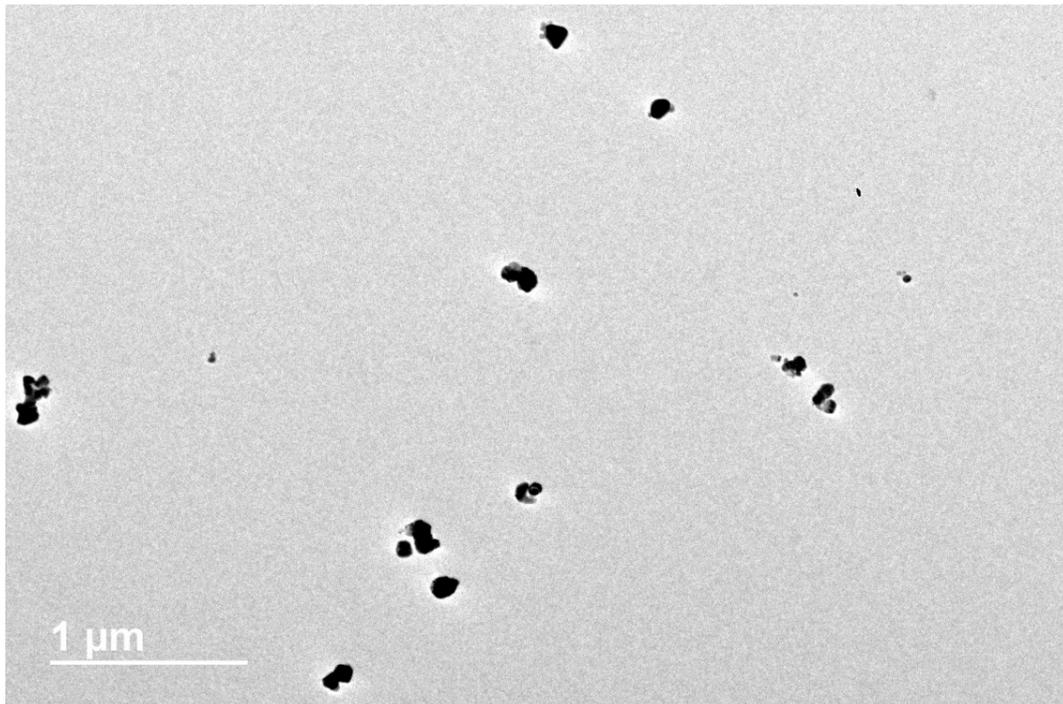


Fig. 6 TEM image of AgNPs synthesized by microwave irradiation from D-ribose for 1 minute at 1,300 W.

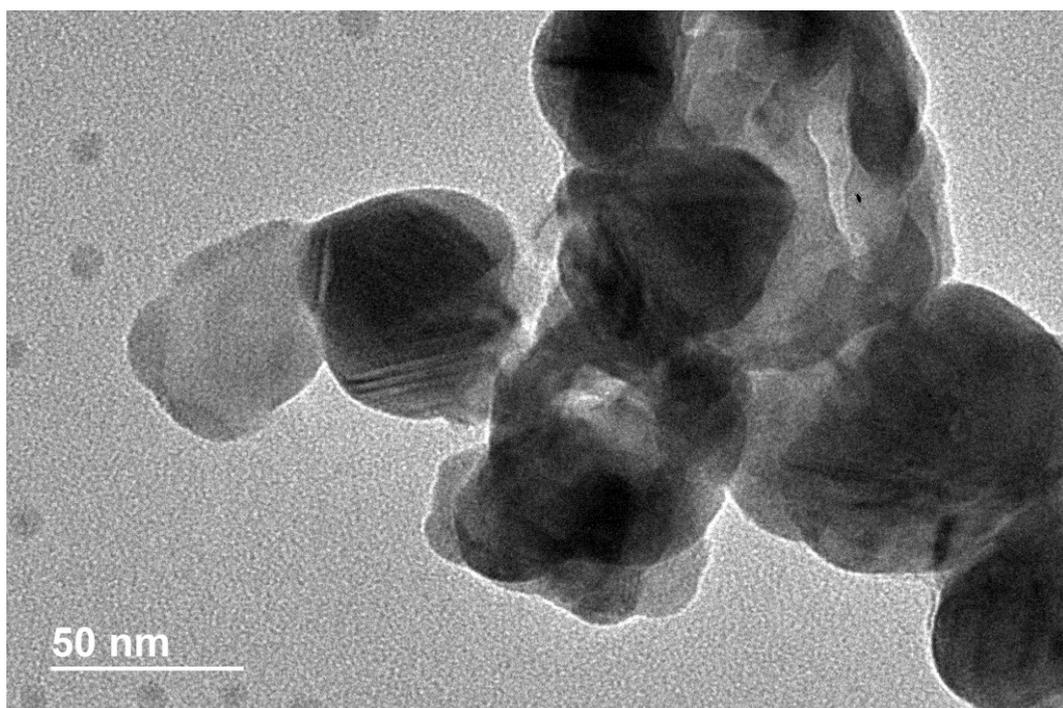


Fig. 7 TEM image of AgNPs synthesized by microwave irradiation from D-ribose for 2 minutes at 1,300 W.

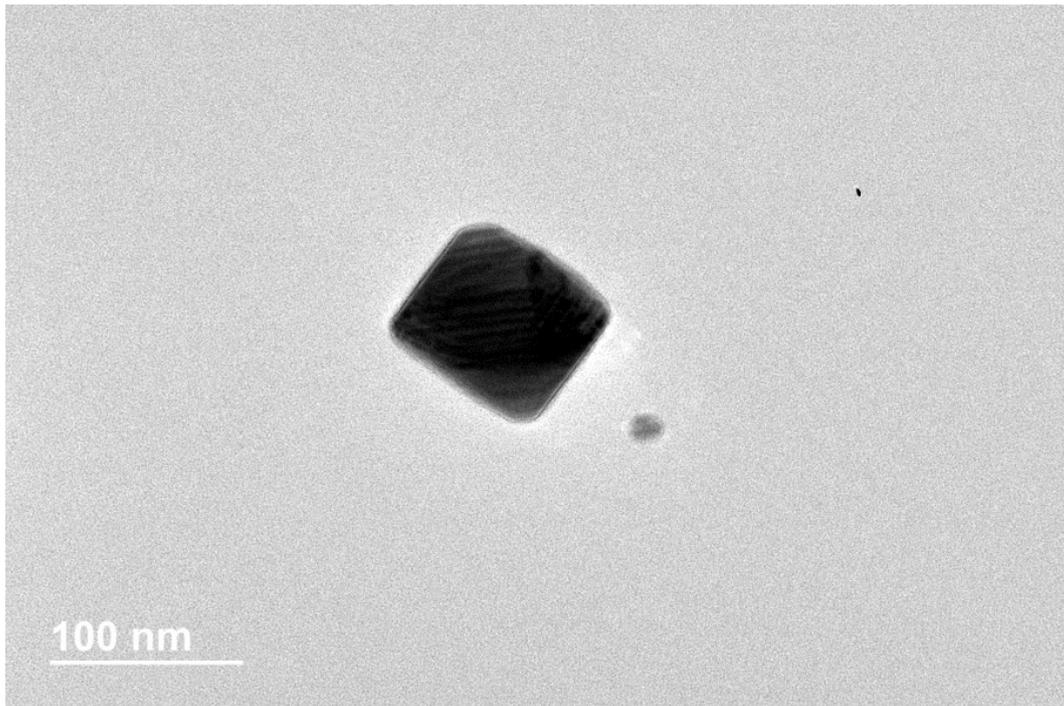


Fig. 8 TEM image of AgNPs synthesized by microwave irradiation from L-arabinoose for 2 minutes at 1,300 W.

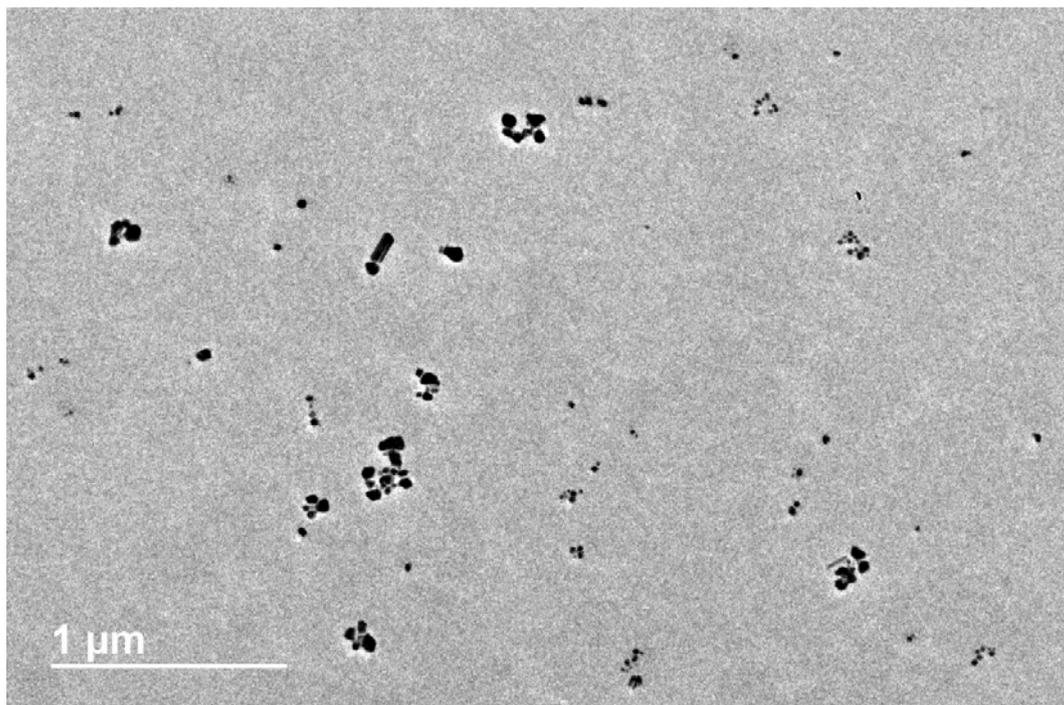


Fig. 9 TEM image of AgNPs synthesized by microwave irradiation from D-arabinoose for 3 minutes at 1,300 W.

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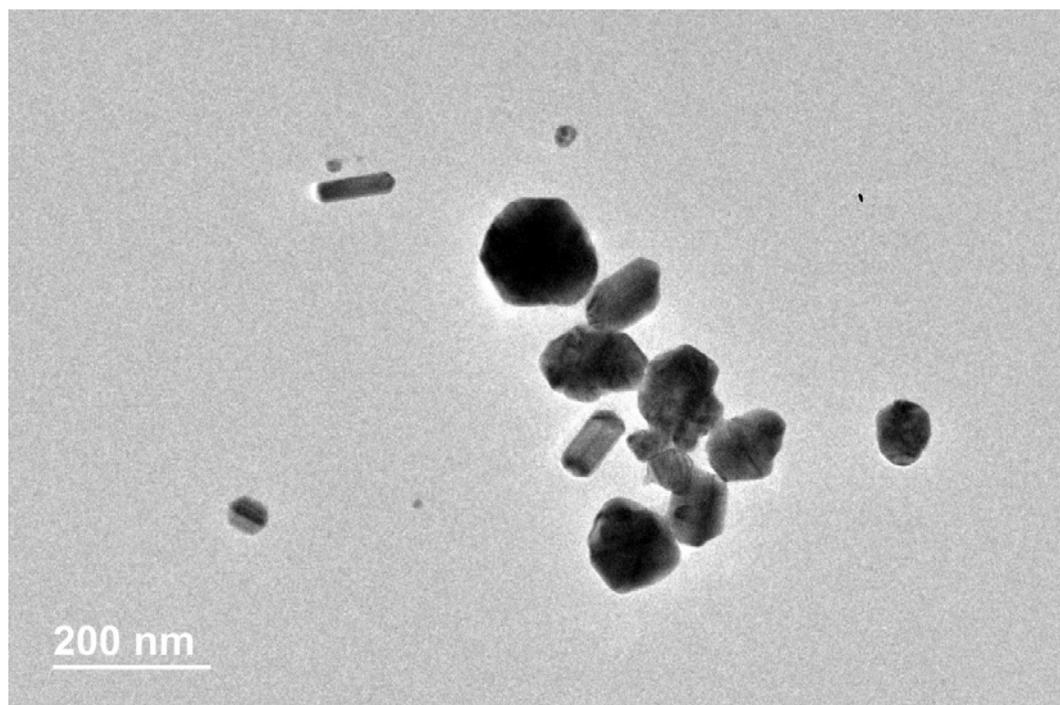


Fig. 10 TEM image of AgNPs synthesized by microwave irradiation from D-arabinoose for 40 s at 1,300 W.

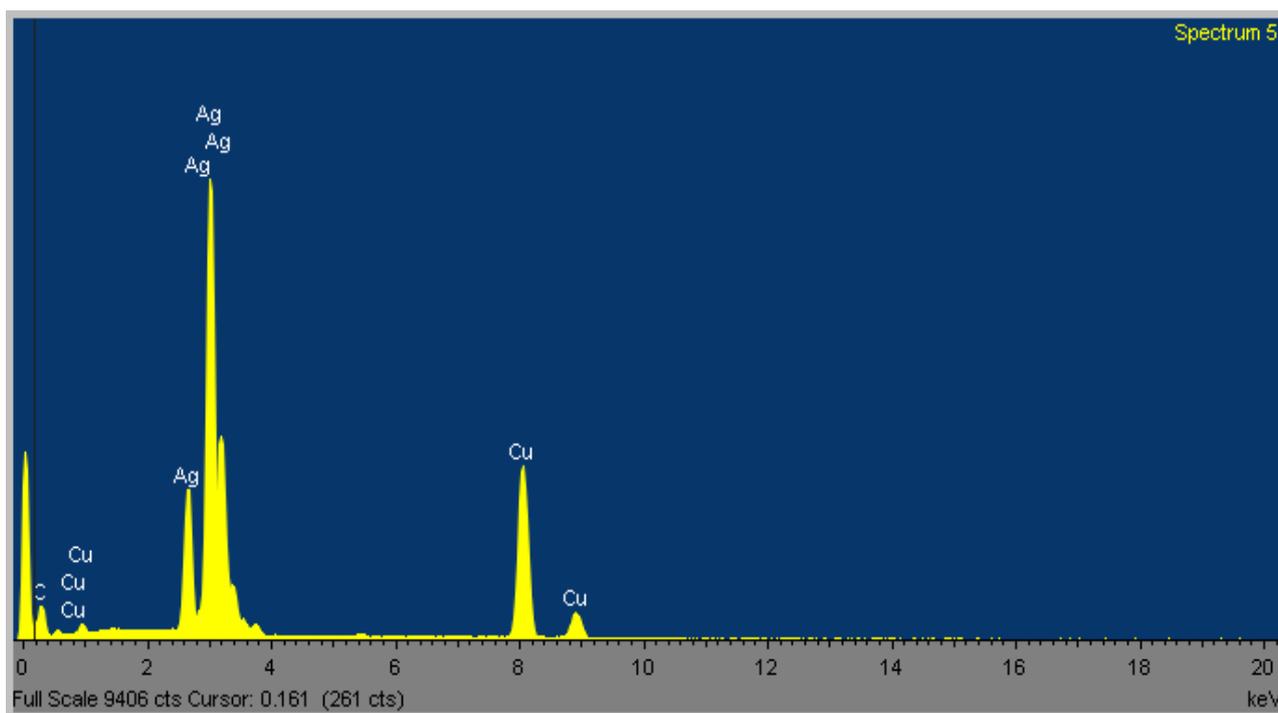


Fig. 11 EDX image of AgNPs synthesized by microwave irradiation from D-ribose for 2 minutes at 1,300 W.

4. Conclusions

AgNPs were synthesized under microwave irradiation using ribose and arabinose as the reducing agents and starch as the stabilizing reagent. Microwave radiation can achieve a complete conversion after only 1 minute when L-arabinose is used, between 2 and 8 minutes with D-arabinose and between 2-5 minutes using D-ribose. Silver colloidal solution was characterized by UV-visible spectroscopy, SEM and EDX. The UV-visible spectra showed that the synthesized samples have an absorbance peak at 410 nm. Increasing reaction time is leading to a shift of the absorbance peak towards 420-430 nm. TEM images confirm a uniform particle size around 20-30 nm.

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