



## Solid state synthesis of starch-capped silver nanoparticles



A. Hebeish, Th. I. Shaheen, Mehrez E. El-Naggar\*

Textile Research Division, National Research Centre (Affiliation ID: 60014618), Dokki, Cairo, Egypt

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### ABSTRACT

The present research addresses the establishment of a technique which is solely devoted to environmentally friendly one-pot green synthesis of dry highly stable powdered silver nanoparticles (AgNPs) using starch as both reductant and stabilizing agent in the presence of sodium hydroxide. It is believed that the sodium hydroxide can improve the reduction potential of starch. Thus when the alkali treated starch is submitted to addition of silver nitrate ( $\text{AgNO}_3$ ), the alkali treated starch induces the well-established dual role of starch; reduction of silver ions ( $\text{Ag}^+$ ) to AgNPs and capping the as-formed AgNPs to prevent them from further growth and agglomeration. Beside assessment of AgNPs formation, structural and morphological characteristics of AgNPs are investigated by making use of UV–vis spectroscopy, transmission electron microscopy (TEM), dynamic light scattering (DLS), zeta potential, FT-IR and X-ray diffraction (XRD) analysis. Research outputs signify (a) the absorbance around 410–420 nm in the UV–vis spectra of AgNPs appears most, probably owing to the presence of nanosized silver particles and the intensity of this peak increases by increasing  $\text{AgNO}_3$  concentration; (b) that highly stable AgNPs with well-dispersed particle are successfully prepared using the present research-based innovation; (c) that the size of AgNPs does not exceed 30 nm with sphere-like morphology even at the highest  $\text{Ag}^+$  concentration employed during synthesis operation; (d) that the XRD and FT-IR confirm the successful preparation of pure AgNPs without noticeable impurities; (d) and that the one-pot synthesis of powdered AgNPs in large scale is clean and easily operated and easily transportation which may be applied as per demands of industries such as textile and painting industry.

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### 1. Introduction

Nowadays, the research area of noble metal nanoparticles has witnessed tremendous growth because of the unique chemical and physical properties of the intermediate state of such matter [1–5]. By virtue of their small size, these crystallites nanoparticles exhibit novel material properties that largely differ from the bulk materials [6]. Of these noble metal nanoparticles, silver nanoparticles (AgNPs) find wide spread applications in the industry confirmed by around 70% of silver produced in the World is used for industrial applications [7]. Some of AgNPs applications are of particular interest due to their remarkable antimicrobial activity which render them feasible unique applications such as antimicrobial activity in textiles [8–11], optical sensor [12], conductivity [13,14], sensor technology [15], cancer therapy [16], catalytic effect [17] and so on. These applications relies on strongly dependent on the size, shape, and morphology of AgNPs.

Several synthetic methods are reported for the synthesis of AgNPs. among these methods mention is made of the following: chemical reduction [18] of  $\text{Ag}^+$  with or without protecting agent, thermal decomposition [19], photochemical reduction [20,21], sonochemical reduction [22], Fungus-mediated synthesis [8,9,23], in situ generation in polymer film, and microwave-assisted reduction have been reported for AgNPs synthesis [24].

Most of the various synthetic methods are able to produce AgNPs in a liquid state and the formed nanoparticles dispersed in solvents are stable only at relatively low concentration of metal salts.

The essential advantage of liquid state synthesis is the formation of uniform AgNPs having certain particle size through selective synthesis. However, colloidal AgNPs tend to agglomerate due to their high surface energy. That is why protecting agents are used to prevent such agglomeration of the colloidal particles via surrounding the particles and thereby stabilizing them [25].

The weaknesses of the liquid state synthesis include the following drawbacks: 1) it is not suited for mass production at low cost. 2) It is necessary to use a very low concentration of metal salt to guarantee dispersion stability of the formed AgNPs. 3) A large amount of dispersion media is inevitably used along with a very large container for mass production and transportation of AgNPs, thereby

\* Corresponding author.

E-mail addresses: [Mehrezchem@yahoo.com](mailto:Mehrezchem@yahoo.com), [melnaggar@ncsu.edu](mailto:melnaggar@ncsu.edu) (M.E. El-Naggar).

causing an increase in industrial cost. 4) Morphology change of the synthesized AgNPs can occur during the process of evaporating the solvent during preparation of a solid sample from a liquid phase. 5) The conventional liquid state synthesis method of AgNPs is not suitable in terms of commercial mass production. Therefore, there is still an environmental benign need for method for mass production of AgNPs at low cost.

Hebeish et al. report for the first time a method for preparation of AgNPs in the powder form from aqueous solution using ethanol as precipitating agent. However the precipitated method is not economically cost-effective and needs longer times and lots of chemicals [26]. Debnath et al. report for the first time a solid-state high speed vibration milling method for the synthesis of AgNPs. In this method poly (vinylpyrrolidone) is used for the reduction of the  $\text{Ag}^+$ . The synthesis is performed at room temperature without using any surfactant to direct the anisotropic growth of AgNPs [27].

Geckeler et al. report to have invented a method whereby mixing a silver salt is mixed with water soluble polymer which exerts the dual action of both reducing agent and a protecting agent to produce a solid mixture; milling of this solid mixture by a high-speed vibration milling process leads to formation of AgNPs within the water dissolved polymer. The as formed AgNPs can be easily and simply produced in a solid state through high speed vibration thereby reducing costs for industrial production and transportation of AgNPs. They also report that the synthesized AgNPs can be used for a long time for more than one year in the solid state without aggregation [25]. Zhang et al. report a method for preparation AgNPs in the solid-state reactions at ambient temperature using ascorbic acid as reducing and stabilizing agent by grinding  $\text{AgNO}_3$  and ascorbic acid (AA) for about 30 min without adding any solvent and organic protectors [28].

In our present work starch is used as reducing agent to convert  $\text{Ag}^+$  to AgNPs and stabilizing agent for the formed AgNPs. Starch is carbohydrate polymers, insoluble in water. It is converted to water soluble by the addition of sodium. Addition of NaOH to starch under mechanical grinding promotes immediate and rapid swelling of the starch granules. The swelling starch has the ability to be act as stabilized agent for the resulted silver nanoparticles. On the other hand, the presence of reducing group in C6 positions of glucose unit can act as reductant. All these reactions have been occurred without the addition of organic or aqueous solvents. The ultimate effect of this is enhancement of the reduction efficiency of starch as reducing agent as well as starch capability in capping AgNPs. Thus we present, herein, a research-based work for synthesis of powdered AgNPs using a system referred to as one-pot green synthesis.



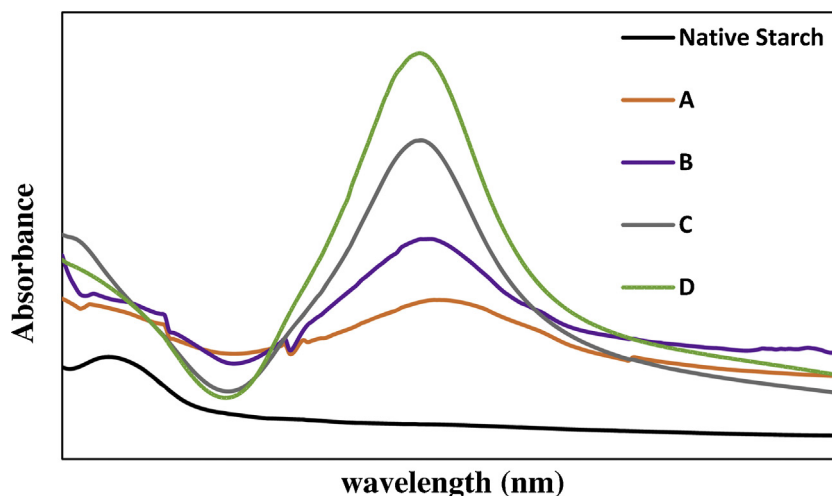
**Fig. 1.** Photographic photo of the powdered AgNPs prepared at different concentrations of  $\text{AgNO}_3$ , viz, (0 mM, 0.1 mM, 0.5 mM, 1 mM and 2) for St-ONa, AgNPs 1, AgNPs 2, AgNPs 3 and AgNPs 4 respectively.

The so called technique is performed according to a strategy which comprises: (1) unique preparation of alkali treated starch via allowing starch–sodium hydroxide grinded materials to interact in the solid state, (2) addition of  $\text{AgNO}_3$  dissolved in the least amount of water/isopropanol mixture to the as-prepared alkali treated starch, (3) giving time for reduction of  $\text{Ag}^+$  to AgNPs as well as for capping and or encapsulation of the latter with alkali treated starch and (4) during this time of synthesis at the high temperature used, water is evaporated, aided by evaporation characteristic of isopropanol. Dependence of AgNPs formation on  $\text{AgNO}_3$  concentration is studied visually as well as by using UV–vis spectroscopy. Also studied are the characteristic structural and morphological features of AgNPs using world class facilities.

## 2. Materials and methods

### 2.1. Materials

Starch was supplied by Glucose and Yeast Co., Egypt. Silver nitrate ( $\text{AgNO}_3$ ) was purchased from Sigma–Aldrich Co., USA. Isopropanol was obtained from Across Co., Germany. NaOH was purchased from Sigma–Aldrich Co., USA. All chemicals reagent are of analytical grade and all were used as received without purification. Deionized water was used for all dilutions and characterization.



**Fig. 2.** UV–vis spectra of the powdered AgNPs prepared at different  $\text{AgNO}_3$  concentrations; 0.1 mM, 0.5 mM, 1.0 mM and 2.0 mM coded as A–D, respectively.

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