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Green synthesis of silver nanoparticles using commercially available starch products

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Abstract

Silver nanoparticles (Ag-NPs) are of great interest due to their wide use in many applications. In that research work, different types of pyrodextrins (PDs), namely British gum (BG), Dexy-84, and Dexy-86, were utilized as reducing and stabilizing agents for preparing Ag-NPs. The conditions affecting synthesis of Ag-NPs such as PD type, reaction temperature and time, reaction medium pH and $AgNO_3$/PD molar ratio were studied. The results obtained exhibit that BG is the effective PD type to synthesis Ag-NPs. The optimum conditions of the synthesis reaction are: $AgNO_3$/BG molar ratio, 0.075; pH, 11; reaction time, 3h; and reaction temperature, 70 ^{O}C. Cotton fabrics treated with different finishing formulations containing the prepared Ag-NPs exhibit antibacterial properties against both the E. coli and S. aurous bacteria.

Keywords: Pyrodextrins; Green synthesis, Silver nanoparticles; Antibacterial finishing.

1. Introduction

Nanotechnology is a valuable domain dealing with design, synthesis, and applications of particles and their structure ranging from 1 to 100 nm. The nanotechnology research witness massive growth due to the novel chemical and physical properties of the nanoparticles such as, small size, large surface area and high chemical purity of these crystallites are the reasons for such unique properties [1,2].

Silver nanoparticles (Ag-NPs) are of great interest because of their applications in health care, cosmetics, biosensor materials, superconducting materials, etc [1,2]. Various chemical, physical, and biological methods including chemical reduction[3], photochemical reduction[4], biological reduction[5], electrochemical reduction [6], reduction by irradiation [7] have been used for synthesis of Ag-NPs. The most chemical approaches include the chemical reduction of silver ions (Ag^{+}) to metallic silver (Ag^{o}) using organic and inorganic reducing agents such as sodium citrate and sodium borohydride [8]. Due to the growing of the environmental awareness, it is necessary to synthesis Ag-NPs with green approaches using water as a solvent and biopolymers as reducing as well as stabilizing agents with biological and irradiation methods. The biopolymers such as cellulose, chitosan, gums and starch are renewable sources and have been proposed in addition to their derivatives for Ag-NPs synthesis [9-12].

Starch as a low cost nontoxic renewable material can be used to synthesis silver nano-particles [13]. The pure starch is a white powder insoluble in water. Modification of starch widens its applicability in the industry. Starch can be modified physically by pre-gelatinization, re-drying or extrusion, and chemically by oxidation, esterification, or etherification [14].

Dextrins are low molecular weight carbohydrates prepared by hydrolysis of starch or glycogen [15]. Dextrins are D-glucose units bridged by α-(1→4) or α-(1→6) links (Figure 1). Dextrins can be produced by enzymes like amylases[16] or by applying dry heat under acidic conditions (pyrolysis or roasting) of starches. The method of dextrinization or thermal transformation of starch was considered as the first

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method of modification of starch, published at the end of 19th century. This process was used to obtain water soluble products. Pyrodextrins are starch derivatives made by dry-roasting process in the presence of a catalyst such as hydrochloric acid. The dry-roasting process causes a principal reaction of transglycosidation resulting in more highly branched molecules of reduced molecular weight. Thus, pyrodextrins exhibit better solubility, reduced solution viscosity as well as greater solution stability than the parent starches. Controlling of the manufacturing conditions such as starch type, starch moisture content, roasting temperature, roasting time, and catalyst type, yields different products[17-20].

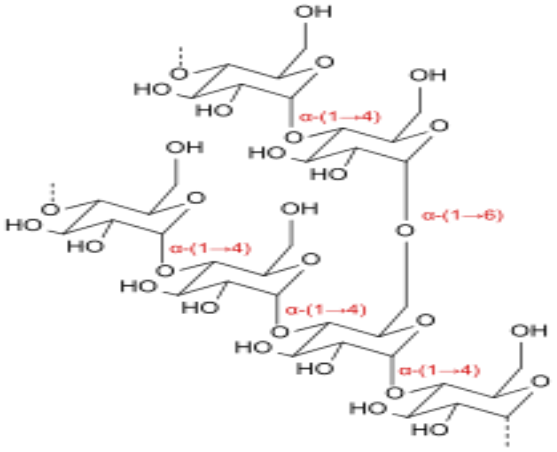

Figure 1: Dextrins with α-(1→4) and α-(1→6).

Recently, extensive research has been carried out for developing and manufacturing of high added value textile goods having durable multifunctional properties such as anti-crease, antimicrobial, softness, UV-protecting, and self cleaning properties to cope with customer needs taking in consideration comfort, economic and ecological concerns [21–31].

The current work is undertaken with a view to use a sustainable and eco-friendly reducing and stabilizing agent such as pyrodextrins namely British gum, Dexy-84, and Dexy-86 for the synthesis of Ag nanoparticles. Different factors affecting the synthesis of Ag nanoparticles were also studied.

2. Experimental

2.1. Materials

Mill scoured and bleached plain-weave cotton fabric (143 g/m^2) was used. Three pyrodextrins (PDs), namely British Gum (the corn starch was heated for along period of time), Dexy-84 (the corn starch was heated for 2h in the presence of an acid), Dexy-86 (the corn starch was heated for 5h in the presence of an acid), were kindly supplied by the Egyptian Company for starch products, Mostrod, Cairo, Egypt. Hostapal® CV-ET (a non-ionic wetting agent based on alkyl aryl polyglycol ether, clariant). Silver nitrate, citric acid (CA), sodium hypophosphite (SHP) and sodium hydroxide were of laboratory grade chemicals.

2.2. Methods

2.2.1. Preparation of silver nanoparticles

A known concentration of silver nitrate aqueous solution (0.05–0.125mmole) was added dropwise to an aqueous solution of the PDs at specific concentration (1 mmole) and pH (8-13). The temperature was kept constant (50 – 90 ^{O}C)for a certain time (30 – 240 min) with continuous stirring.After short time, the reaction medium turned into a brownish yellowcolor indicating the formation of Ag-NPs. The extent of Ag-NPs formationwas followed by evaluating the UV -Vis spectra.

2.2.2. Loading of cotton fabric with Ag-NPs as an antibacterial finishing agent

The potential application of the prepared Ag-NPs as an antibacterial finishing agentwas studied by padding cotton samples of 30X30 cm^2 in finishing formulation containing 1 or 2 g/L of the synthesised Ag-NPs, 40 g/l citric acid as crosslinker and 40 g/l of sodium hypophosphite as a catalyst. The padded samples were then dried at 80 ^{O}C/3 min and cured at 180 ^{O}C/90 sec followed by washing at 50 ^{O}C for 15 minutes and drying before testing.

2.3. Testing and analysis

- The aldehyde group content was determined using the rapid quantitative alkali consumption method as reported elsewhere[32].
- The formation of Ag-NPs was indicated by ultraviolet-visible (UV–Vis) spectroscopy using T80 spectrophotometer. In that concern, the

reaction medium has an absorption in the range of 420–450 nm due to the Surface Plasmon Resonance band of the Ag-NPs.

- The antibacterial activities of an untreated control and Ag-NPs treated fabric samples were assessed according to the bacterial count method as reported elsewhere [33] against the following bacteria strains:

(1) Gram-positive bacteria: *Staphylococcus aureus (S. aureus).*

(2) Gram-negative bacteria: *Escherichia coli (E. coli*).

According to that method, a liquid culture was prepared by mixing 0.5 g peptone and 0.3 g beef extract in 100 ml water. 1 cm diameter of the crosslinked film samples were cut and put into 10 ml of liquid culture, to which 10 µL of microbe culture was added and the tested samples were then incubated for 24 h at 37 °C. From each incubated sample, 100 µL of solution was taken, diluted and distributed onto an agar plate. All plates were incubated for 24 hours and the colonies formed were counted. The percentage reduction was determined as follows:

% Reduction in CFU (Colony Forming Units) = (C – A) / C X 100

Where, C and A are the colonies counted from the plate of the control and treated samples, respectively.

- The Ag-NPs size distribution was determined using Zeta sizer nano series (NanoZS), size range of 0.6 - 6000 nm, Malvern Instruments, UK.
- Durability to wash was carried out by subjecting the fabric samples to 5 laundering cycles. Each laundering cycle consists of washing (10 min at 50 ^{O}C using 2 g/l nonionic surfactant followed by rinsing and air drying at ambient conditions [23].

3. Results and discussion

The UV-Vis spectroscopy is commonly used technique to characterize the metallic nanoparticles due to surface Plasmon resonance (SPR) phenomenon[10].The characteristic surface plasmon resonance vibration peak in UV-Vis spectrum of Ag-NPs is around 420 nm[10]. Different nanoparticles sizes will absorb different wavelengths of light and will broaden the absorbance peak in UV-Vis spectrum. The effect of the reaction time and temperature, pH of the reaction medium, $AgNO_3$ concentration and pyrodextrin type on extent of the Ag-NPs formation will be discussed as follows.

3.1. Factors affecting Ag-NPs formation

3.1.1. Reaction time

Figure 2 shows the UV-Vis absorption spectra of Ag-NPs colloidal solutions prepared at different reaction time intervals. It is clear that, increasing of reaction time from 30 to 240 min results in a progressive enhancement in the absorption intensity as well as narrowness of the Ag-NPs Plasmon band; almost without affecting the maxima absorption peak of Ag-NPs at 430 nm. This matter indicates a successful reduction of Ag+ to Ag0and formation of Ag-NPs using BG as a reducing and stabilizing agent [11,34-36]. Moreover, the wide band at earlier reaction time, i.e. 30 min, reflects a presence of a wide distribution of Ag-NPs size[11, 36].

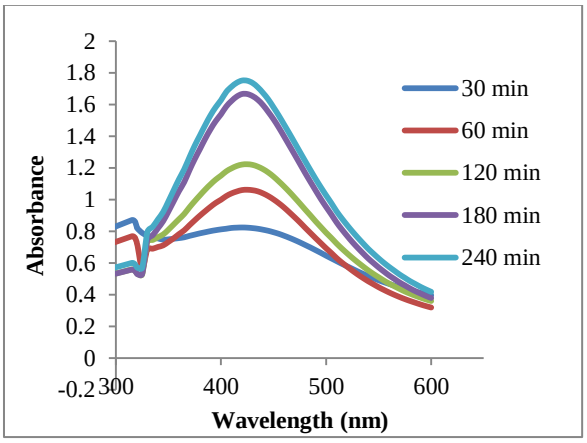

Figure 2: UV–Vis absorption spectra of Ag-NPs synthesized at different time intervals, $AgNO_3$/BG molar ratio, 0.05; reaction pH, 9; reaction temperature, 80 ^{O}C.

3.1.2. Reaction temperature

Figure 3 illustrates the effect of raising the temperature on UV-Vis absorption spectra of their colloidal Ag-NPs solutions. It is clear that raising the reaction temperature from 50 to 70 ^{O}C is accompanied by a gradual enhancement in the absorption intensity as well as broadness in the Ag-NPs plasmon band. It seems that increasing the

temperature up to 70 ^{O}C leads to a gradual enhancement in the reduction rate of Ag^{+} ions in their colloidal solutions to form Ag-NPs of different sizes[2, 34-36].

The further increasing in the reaction temperature, beyond 70 and up to 90 ^{O}C, decreases the absorption intensity of Ag-NPs Plasmon band but keeps the band at 430 nm. This may be a direct consequence for decreasing of the extent of Ag^{+} ions reduction[11] and/or aggregation of the synthesized Ag-NPs [11] that ultimately reduces the absorption intensity of Ag-NPs Plasmon band.

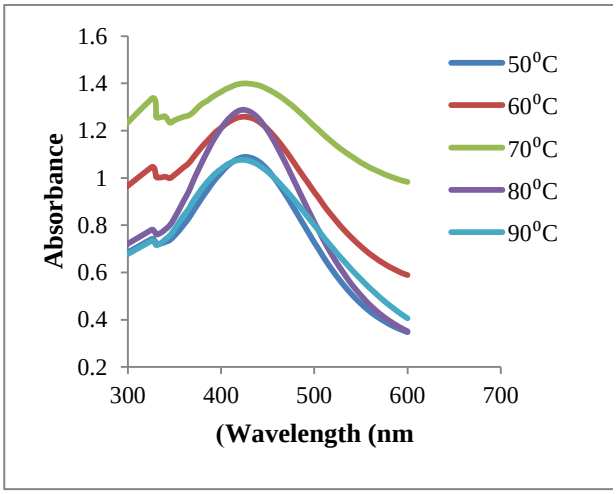

Figure 3: UV–Vis absorption spectra of Ag-NPs prepared at different temperatures; $AgNO_3$/BG molar ratio, 0.05; reaction pH, 9; reaction time, 3h.

3.1.3. Reaction pH

Figure 4demonstrates the impact of pH on UV-Vis spectra of silver colloidal solutions prepared at different reaction pHs. It is well seen that increasing pH of Ag-NPs synthesis reactionfrom8to 13 results in a gradual growing in the absorption intensity and narrowing of the Ag-NPs plasmon band as well as shifting the absorption peak to longer wavelength, i.e. from 420 to 455 nm. This could be attributed to the gradual increasing in the magnitude of HO^{-} ions in the reaction medium and the subsequent partial hydrolysis and/or oxidation of BG chains[11, 37, 38], that results in a higher extent of reducing species formation and consequently a higher extent of Ag^{+} ions reduction to form Ag^{0}.

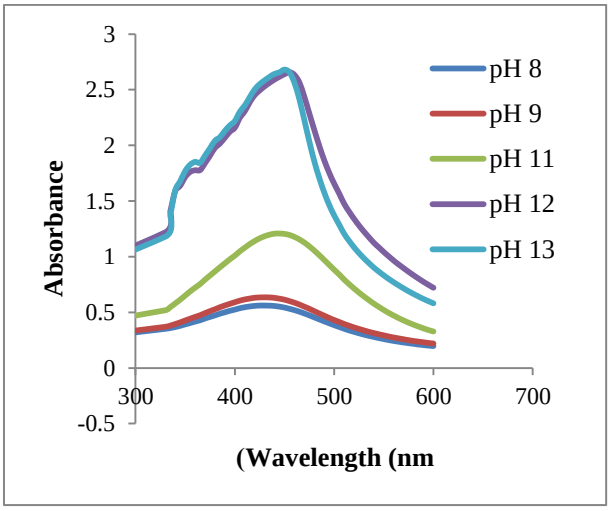

Figure 4: UV–Vis absorption spectra of Ag-NPs prepared at different reaction pHs, $AgNO_3$/BG molar ratio, 0.05; reaction time, 3h; reaction temperature, 70 ^{O}C.

On the other hand, the particles size distribution of Ag-NPs prepared at pH 11, 12 and 13 are shown in Figure 5. It is clearly seen that the average particles size of the prepared Ag-NPs can be arranged in the following ascending order with respect to the pH of the reaction medium: pH 11 (2.7 nm) < pH 12 (6.5 nm) < pH 13 (28.2 nm).

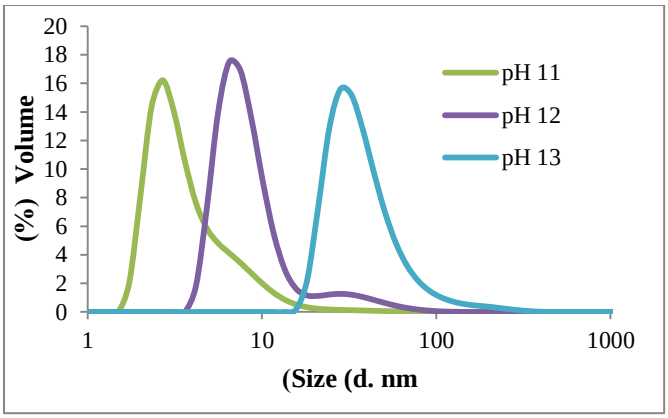

Figure 5: Particle size distribution of Ag-NPs prepared at different reaction pHs, $AgNO_3$/BG molar ratio, 0.05; reaction time, 3h; reaction temperature, 70 ^{O}C.

3.1.4. Silver nitrate to British Gum molar ratio

Figure 6 shows that increasing of $AgNO_3$/BG molar ratio results in an enhancement in

the absorption peaks and leads to a gradual shifting of the absorption peak to higher wavelengths, i.e. from 430 to 470 nm which may be attributed to the aggregation and agglomeration of the prepared Ag-NPs[39].

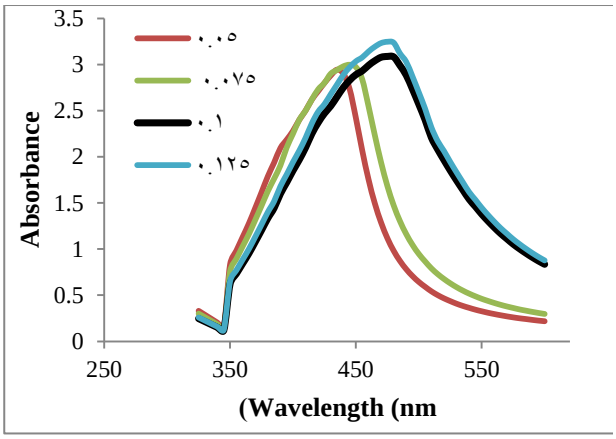

Figure 6: UV–Vis absorption spectra of Ag-NPs prepared at different AgNO3/BG molar ratios, Reaction pH, 11; reaction time, 3h; reaction temperature, 70 ^{o}C.

On the other hand, Figure 7 indicates that the particles size distribution of the prepared Ag-NPs is depending strongly on the concentration of silver nitrate as a precursor. As shown from Figure 6, the average particles size of Ag-NPs prepared at different $AgNO_3$/BG molar ratios can be arranged in the following ascending order:

0.075 (6.5 nm) <0.1 (7.8 nm) < 0.125 (8.7 nm) < 0.05 (10.1 nm)

The results of Figure 6 suggests that the greater availability of HO^- ions in the reaction medium at lower Ag^+ concentration, i.e. at molar ratio of 0.05,induces a faster reduction rate of Ag^+ ions as well as agglomeration of $Ag^0$to form large Ag-NPs. Beyond that ratio and up to molar ratio of 0.125, increasing Ag^+ may result ina gradual balancing between the HO^- and Ag^+ ions and causes a progressive formation and agglomeration of Ag-NPs.

3.1.5 Pyrodextrin type

The effect of the PD type, viz. BG, Dexy-84 or Dexy-86, on the UV-Vis absorption spectra of the prepared Ag-NPs colloidal solutions are shown in Figure8.Obviously, Figure7 clarifies the following orders:

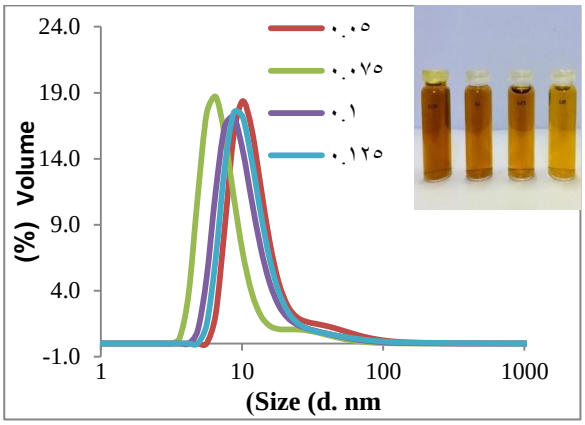

Figure 7: Particle size distribution of Ag-NPs prepared at different AgNO3/BG molar ratios, Reaction pH, 11; reaction time, 3h; reaction temperature, 70 ^{O}C.

the absorption intensity of such PDs decreases in the order:

BG >> Dexy-86 > Dexy-84,

- the absorption peak width increases in the order:

BG < Dexy-86 < Dexy-84, and

- the absorption peakwavelengthdecreases in the order:

- Dexy-84 (415 nm) > BG (420 nm) > Dexy-86 (440 nm).

Keeping the other parameters constant, the variation in such properties could be ascribed to the differences among these PDs in their preparation conditions that were mentioned before in the experimental part.

On the other hand, despite the BG lower content of the aldehyde groups compared to Dexy-86or Dexy-84, as shown in Table 1, it provides the highest intensity with narrowest Ag-NPs absorption band suggesting that BG has the highest content of amylose chains among the foregoing pyrodextrins[40] keeping in mind that the amylose chains are long linear d-glucose macromolecules and can protect, i.e. stabilize, the growing Ag-NPs compared to the amylopectin branched macromolecules of the short side chains[41].

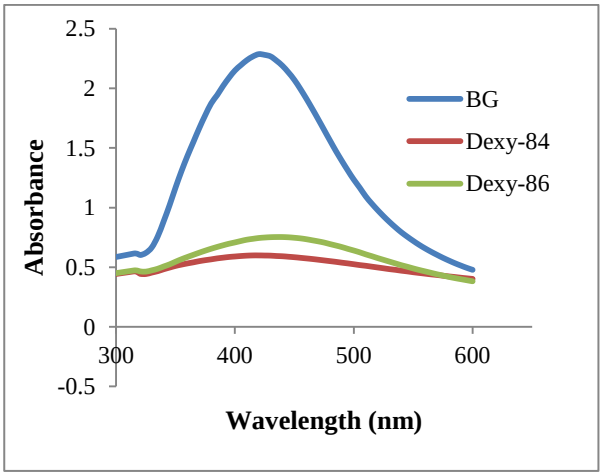

Figure 8: UV–Vis absorption spectra of Ag-NPs prepared by different PDs, $AgNO_3$/BG molar ratio, 0.075; reaction pH, 11; reaction time, 3h; reaction temperature, 70 ^{O}C

Table 1: The aldehyde groups contents of the nominated PDs

PD type	(%) Aldehyde content
BG	5.4
D-86	6.7
D-84	5.58

3.2. Application of the synthesized Ag-NPs as antibacterial finish

To illustrate the impact of that synthesized Ag-NPs as antibacterial finish, they were incorporated in different finishing bathes of cotton fabric containing citric acid as a crosslinker. Table 2 shows the antibacterial properties of the finished fabric. It is obvious that: i) finishing a cotton fabric in presence of citric acid as a crosslinked imparts antibacterial properties to that fabric confirming the antibacterial properties of citric acid[42], ii)inclusion of the synthesized Ag-NPs as antibacterial finishing the finishing bath with a concentration of 1 g/L, in addition to citric acid, is accompanied with an enhancement in the antibacterial properties of the finished fabric reflecting the ability of Ag-NPs to destroy the bacteria via formation of Ag-ions, in the presence of the moisture, that binds to and thereby inactivate the bacterial DNA and/or generation of oxygen radicals that oxidize the molecular structure of bacterial cells [43-49], iii) increasing the synthesized Ag-NPs concentration in the finishing bath to 2 g/l in effectively promotes the antibacterial properties of finished fabric, and iv) the antibacterial properties of the finished fabric samples are durable to 5 washing cycles.

Table 2: Antibacterial properties of treated cotton fabrics

Treatment type	1. % Reduction	
	G +ve	G –ve
Citric acid	(43) 48	(49) 53
Citric acid + 1 g/L of Ag-NPs	(75)81	(54) 62
	(93) 99.9	(79) 87
Citric acid + 2 g/L of Ag-NPs		

The Ag-NPs were prepared using BG; [Citric acid], 40 g/l; [SHP], 40 g/l; wet pick up, 100%; drying, 85 ^{O}C/5 min; curing, 180^{O}C/90 sec. Values in parentheses indicate the antibacterial properties after 5 laundering cycles.

4. Conclusion

PDs were used to synthesis Ag nanoparticles. These substrates act as both reducing and stabilizing agents. BG is the effective PD type for Ag-NPs synthesis. The optimum conditions of the synthesis reaction are: $AgNO_3$/BG molar ratio, 0.075; pH, 11; reaction time, 3h; and reaction temperature, 70 ^{O}C. Cotton fabrics treated with different finishing formulations containing the prepared Ag-NPs exhibit antibacterial properties against both E. coli and S. aurous.

"The authors declare no conflict of interest."
The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

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