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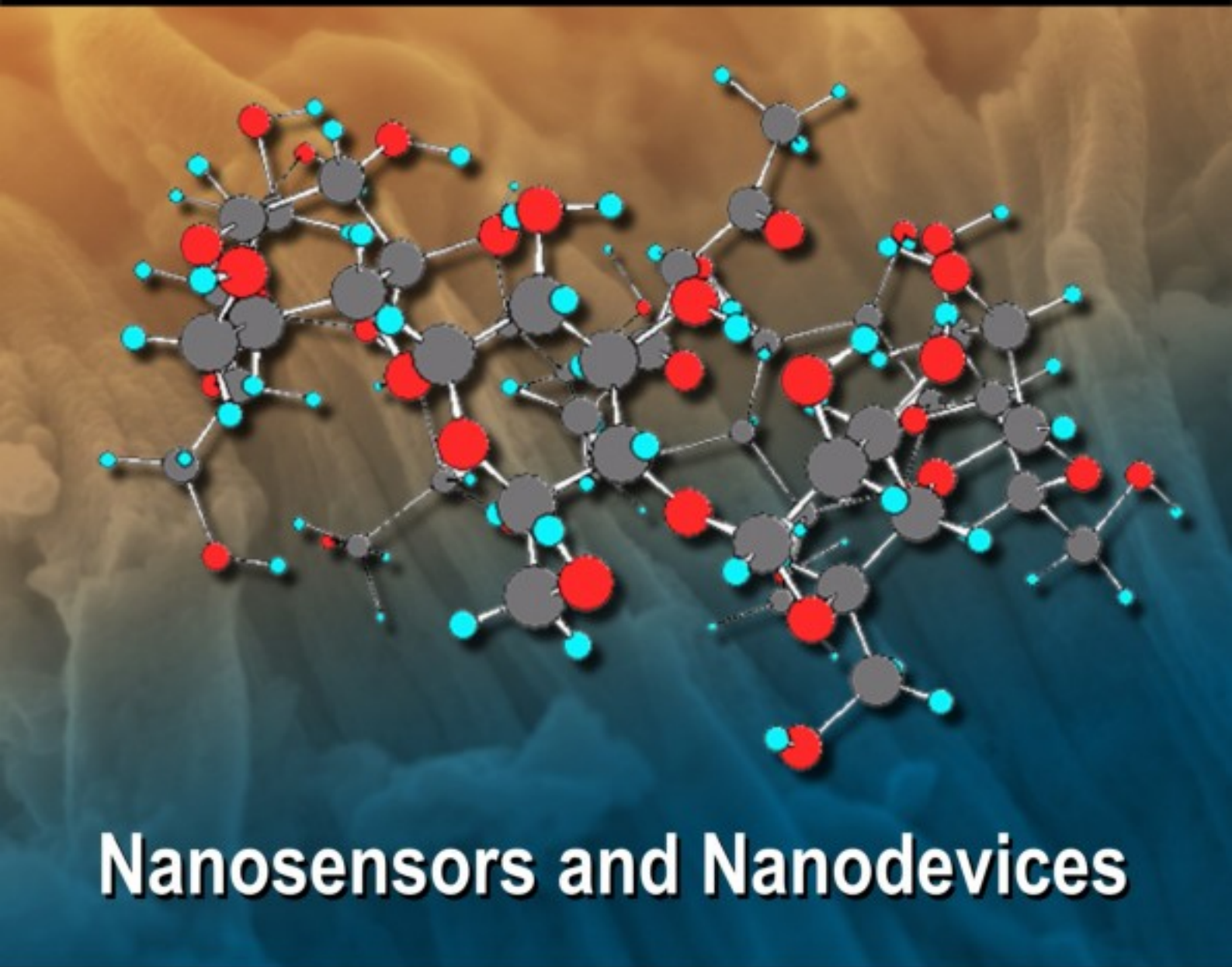

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A Facile and Green Synthesis of Small Silver Nanoparticles in β–cyclodextrins Performing as Chemical Microreactors and Capping Agents

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Abstract: Small silver nanoparticles were prepared by a facile and green synthesis based on a spontaneous decomposition of a commercial silver complex at room temperature, in a water solution of β-cyclodextrins acting either as micro-cavities, in which a thermal intramolecular reduction occurs, or as additives and capping agents for, respectively, the tuning of the dimensions and the stabilization of the silver nanoparticles, without using any poisoning. The silver nanoparticles were characterized by UV/VIS spectroscopy, Resonance Rayleigh Scanning (RRS) and transmission electron microscopy (TEM). TEM of the sample showed uniform and monodispersive particle distribution in the colloids, with dimension centered at about 5 nm and not over 10 nm, dispersed into the biologically and physiologically friendly β-cyclodextrin environment. For these reasons, the synthetic route reported in this work is very promising for both its simplicity and ability to produce silver nanoparticles with reduced dimensions and high stability. *Copyright © 2012 IFSA.*

Keywords: Silver nanoparticles, β-cyclodextrins, Raylegh resonance scattering, Transmission electron microscopy, UV plasmon, Sensing.

1. Introduction

Nanoparticles are of great scientific and industrial interest since they represent a link between bulk materials and atomic and molecular structures. The bulk material has constant physical properties regardless of its size, while at the nano scale the chemical-physical properties can dramatically change

compared to the bulk [1]. In particular, noble metal nanoparticles have attracted the attention of scientists because of their technological applications in optics, electronics and in molecular biology to trigger, for example, a PCR (polymerase Chain Reaction) efficiency enhancement [2]. Metal nanoparticles typically display optical properties enabling them for analytical and sensing applications [3]. Among the optical properties, the Surface Plasmon Resonance (SPR) needs to be highlighted. For nanoparticles of gold, silver and copper, this dipole resonance occurs in the UV-Vis region making the nanoparticles useful for optical applications [4]. Because of the position and the intensity of the plasmon peak is determined by the size, the shape and interparticles spacing as well as by the dielectric properties of the nanoparticles and the local environment (i.e. solvent, capping agents, surfactants), their modification in the presence of different kinds of analytes enables the set up of various analytical strategies. In particular, SPR spectroscopy has been employed to monitor analyte-surface binding interactions with respect to the adsorption of small molecules [5], ligand-receptor binding [6], DNA and RNA hybridization [7], protein-DNA interactions [8]. In addition, also Resonance Rayleigh Scattering (RRS) was applied as analytical technique based on the Rayleigh scattering of small particles embedded in a medium or in host molecules. This technique is based on a special elastic scattering produced when the wavelength of Rayleigh scattering is close to the molecular absorption band. In this case, the frequency of the electromagnetic wave absorbed by the electron is equal to its scattering frequency. Due to the intensive absorption of light energy of the electrons, a rescattering phenomenon occurs. The RRS can provide information on molecular structure, size and geometry. The effect has been already studied for some metal nanoparticles such as gold, silver, and iron oxide, in water phase [9]. Also this technique, in which the shape, the size and the refractive index strongly affect the RRS spectrum, has been used for the study and the determination of biological macromolecules and ion-association complexes [10].

In this scenario, silver represents a very intriguing element since it could be exploited in many industrial fields, going from optoelectronics, DNA detection methodology [11] to textile coatings and a number of environmental applications, such as in food storage and healthcare industry, because of its ability to act as anti-bacterial agent and mould growth inhibitor [12].

In this perspective, quite a lot of studies in silver nanoparticles synthesis have been carried out with the aim to optimize the processes for a fine control of the Ag nanoparticles size to meet the specific applications requirements. For example, it was found that only silver nanoparticles of sizes spanning from 1 to10 nm have attached to HIV-1 virus and prevented the virus from bonding to host cells [13].

If from one side the strategies for nanoparticles size control represent a very challenging topic [14,15], also the developing of new stabilization procedures are of great interest. The stability of the nanoparticles is commonly achieved using different capping molecules such as alkylthiol [16], poly(vinylpyrrolidone) [17], organoalkoxysiloxanes [18] or SDS (sodium dodecyl sulfate) microemulsions [19]. Their capping properties avoid the nanoparticles aggregation and make them soluble in given solvents. As additional additives, agents such as formaldehyde [20], alcohol [21], or sodium citrate [22] can act as both surfactants and reductants.

However the use of reducing agent and stabilizing agents can frequently compromise the final application of the nanoparticles. In fact the reducing agents often give undesired side products poisoning the nanoparticles.

As a class of water-soluble and non toxic cyclic oligosaccharides, the cyclodextrins (CDs) have been extensively investigated in host-guest chemistry for the construction of versatile supramolecular aggregations thanks to their special hydrophobic cavities [23]. Cyclodextrins represent an intriguing and powerful class of molecules since they can allow (i) nanoparticle-size control, (ii) in water solubilisation of metal complexes elsewhere not soluble, iii) potential nanoparticles delivery in biological medium. The cyclodextrin has been found to perform as a micro chemical reactor acting

also as size control agent. Green aspects of this synthesis come from the exclusion of any harmful reducing agents during the process, such as sodium borohydride ($NaBH_4$) for its toxicity, or hydroxylamine hydrochloride and any special capping or dispersing agent [24]. A similar approach to green chemistry has been reported for Ag nanoparticles formation from Ag(acac) in deionised water [14].

This paper reports the synthesis of silver nanoparticles (Ag NPs) with average size of about 5 nm, using a green one-step route. This synthetic protocol involves a commercial betadiketonate silver complex, the 2,4-pentandionato of silver(I) [Ag(acac)] in β-cyclodextrin aqueous medium, and it is based on a simple and spontaneous thermal intramolecular reduction reaction of the Ag(acac) complex included into the cyclodextrin cavity. Here the reaction occurs spontaneously via a thermal intramolecular electron transfer from the coordinated molecules to the metal centre, with the reduction of the silver(I) ion to metallic silver and consequently release of the free protonated ligand. The thermal kinetics of the silver reduction was followed by UV-vis spectral changes while the aggregation phenomena between cyclodextrin and both the initial Ag complex and the Ag nanoparticles later were evaluated by RRS. Size evaluation and characterization of the silver nanoparticles were carried out by Transmission Electron Microscopy (TEM).

2. Material and Methods

The (2,4-pentanedionato) silver(I) complex (Aldrich), namely Ag(acac), was reagent grade and purchased by Sigma-Aldrich.

The Ultraviolet-Visible absorption spectra were recorded with a UV-Vis Jasco V-560 spectrophotometer. The kinetics of Ag(acac) degradation was spectrophotometrically followed in quartz cuvettes, in solutions of concentrations ranging from 1.3×10^{-4} to 7×10^{-4} M. The complex solutions were prepared by dissolving Ag(acac) in 10^{-2} M β-cyclodextrin (β-CD) water solution; all the operations were performed into a bag box deaerated by means of a nitrogen flow. As soon as the complex powder was completely solved, within a couple of minutes, 3 mL of solution were putted in a quartz cuvette and sealed under nitrogen atmosphere and then UV-visible spectra and synchronous scan acquisition, for the RRS investigation, were performed at pseudo-zero time and followed during the time. For TEM analysis the solution 2.3×10^{-4} was maintained in the glove bag under nitrogen atmosphere for an hour and then some drops of this solution evaporated on a copper grid for the eventual TEM investigation. TEM analysis was carried out using a TEM JEOL 2010 F.

For the RRS spectra, a SPEX Fluorolog 111 instrument, equipped with a cuvette holder, was used.

Calculations were performed at the level of the PBE formalism [25]. The standard all-electron 3-21G basis was used for all atoms [26]. Molecular geometry optimization of stationary points used analytical gradient techniques. All calculations were performed using G09 [27] code on IBM-SP systems

3. Results and Discussion

The complex Ag(acac) is a deep-brown powder. This color is probably due to a stacked arrangement of the molecules [28] that keep it stable in the solid phase. When dissolved in water or acetonitrile, it gives colorless solutions because of the dilution and the molecules solvation and monomerization. It is well known that the Ag^+ ion shows a pronounced tendency to exhibit linear, 2-fold coordination [29], therefore when a chelate oxyanion such as the $acac^-$ (acetylacetonate) coordinates it, the monomeric

molecule in solution shows instability due to the formation of monodentate chelation, triggering the thermal reduction of the Ag^{+} ion.

The Ag(acac) β-cyclodextrin solutions were prepared by adding a β-cyclodextrin aqueous solution (10-2 M) to the solid complex, operating under nitrogen atmosphere, in order to avoid possible backreactions. In Fig. 1 it was reported the UV-vis absorption spectra of the acac- in solid line, and the protonated free ligand (Hacac), in dotted line, while in Fig. 2 it was reported the kinetics of degradation of a solution about 2.3x10-4 M, followed by UV-visible spectra.

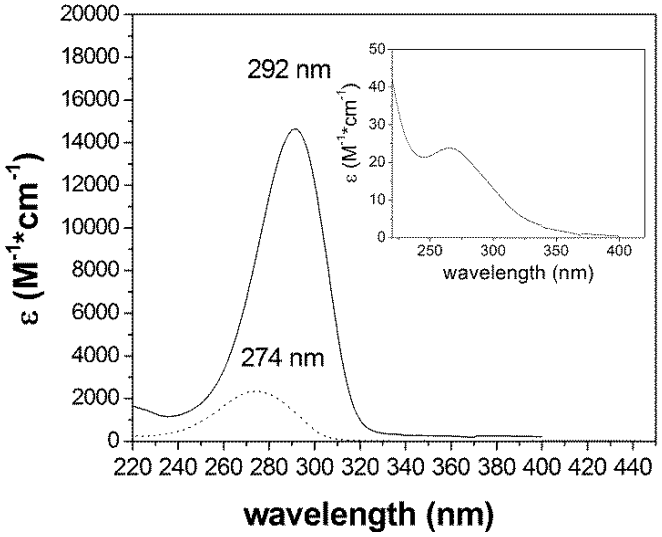


Fig. 1. UV-vis absorption spectra in epsilon, in water β-cyclodextrin 10^{-2} M, of $acac^{-}$ (from sodium acetilacetonate) (solid line) and Hacac (dotted line). Inset: spectrum in epsilon of β-cyclodextrin in water.

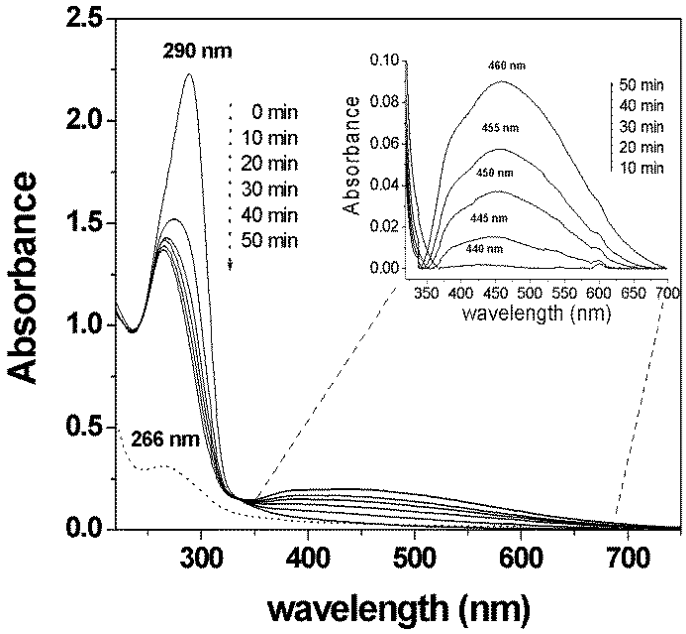


Fig. 2. UV-Vis kinetics in β-cyclodextrin solutions of Ag(acac) 2.3×10^{-4} M at various times. The dash-dot line is the absorbance spectrum of an aqueous solution of β-cyclodextrin 10^{-2} M, while the dot line is the spectrum of Ag(acac) at 1080 minutes . Inset: Kinetics of silver plasmon peak formation (425 nm).

The absorption spectrum at zero time is the electronic spectrum of Ag(acac) that exhibits a band due to the intra-ligands (IL) π_L to π^*_L transitions ($\varepsilon_{\lambda 292\ nm}$ = 14000 M^{-1} cm^{-1} in β-cyclodextrin), as found in other complexes with betadiketonate ligands [30], while the protonated ligand (Hacac) shows absorption at 274 nm with a molar absorptivity of 2200 M^{-1} cm^{-1}

As shown in Fig. 2, unexpectedly, it was found that when the complex starts to solubilise, a chemical reaction occurs, as proven by changes in the electronic absorption spectra observed in the first minutes. The typical silver plasmon absorption band grows during the reaction time while the band at 292 nm decreases with formation of a maximum at about 274 nm, corresponding to the Hacac that shifts below 270 nm along the time, because of a spectral bands combination with the scattering in the UV region attributed to the Ag colloid formation. The addition of Ag(acac) to the water β-cyclodextrin solution quite immediately provoked the appearing of a slight yellow color due to the formation of the absorption band centered at about 425 nm, typical of the silver surface plasmon resonance [31], reaching an apparent plateau value within 60 minutes, with a constant absorption value in the UV region. After 18 hours, the plasmon absorption peak reached a maximum value, while no differences were observed in the UV region where absorption can undoubtedly attributed to the contribution of free cyclodextrin, free Hacac and Ag colloid scattering. Taking into account the absorption of β-cyclodextrin (dash-dotted line in Fig. 2) and the Ag plasmon absorption, the ligand release is computed as quantitative. Unfortunately, zero time spectrum slightly deviates from the real time zero because while the Ag(acac) complex inclusion in β-cyclodextrin starts, the concomitant reduction reaction occurs, so evaluations cannot be very precise.

Parallel to the UV-Visible spectra at various times, synchronous scan spectra (Fig. 3) for the Resonance Rayleigh Scattering investigation were recorded after each UV-visible measurement.

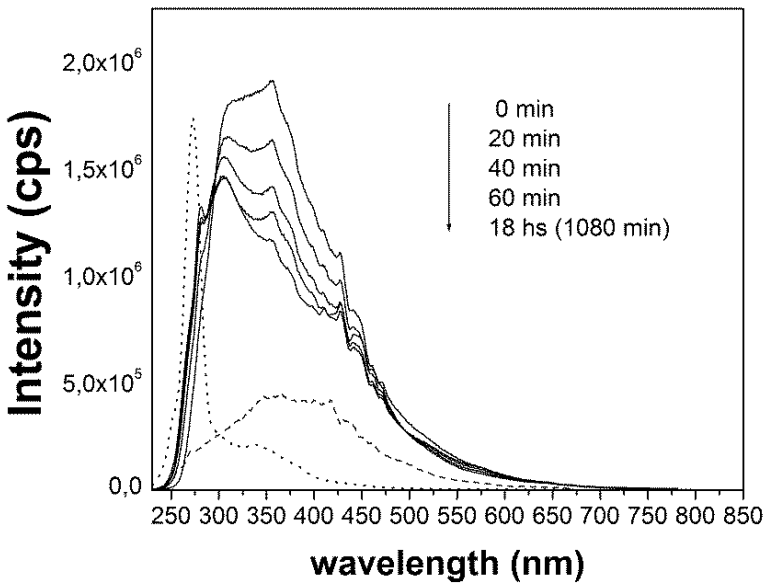


Fig. 3. RRS spectra of Ag(acac) complex in β-cyclodextrin solution along the time. The dotted line is the spectrum of β-cyclodextrin 10^{-2} M while dashed line is the spectrum of pure Ag nanoparticles in water.

The RRS spectrum showed the excitation and emission wavelength synchronously in the form of $\Delta\lambda$=0, thus the synchronous scan investigation was performed by using the spectrofluorometer in λ_{exc}= λ_{em} condition.

The dotted line in Fig. 3 represents the spectrum of a solution of β–cyclodextrin 10^{-2} M while the dashed line is the RRS spectrum of Ag nanoparticles, in water, produced according to the procedure reported in literature [14]. At pseudo zero time, the spectrum of the β–cyclodextrin is not present, while a broadband span over the entire range from 250 nm to 600 nm. This is attributable to the complex inclusion phenomenon determining the inclusion of the metal complex into the cyclodextrin, as reported in literature for other inclusions involving β–cyclodextrin [32]. While the reduction reaction occurs bringing to the metal nanoparticles formation, the broad band decreases in scattering intensity and begins to change the spectrum profile, appearing slightly split in two components: one more defined component in the UV part, around 320 nm, attributable to β-cyclodextrin, (pure spectrum is in dotted line), and a second attributed to the scattering of the metal colloid (see dashed line as reference of RRS spectrum of pure naked Ag nanoparticles in water). It is possible to observe two isosbestic points centered at 310 nm and 520 nm confirming that an equilibrium process is occurring.

As regard the chemical mechanism, it is reasonable to take into account the involving of a thermal spontaneous metal reduction. In particular, two main reaction pathways can be invoked to explain the silver formation and the free ligand release. The first is typical for many cases of thermal spontaneous reduction of metal(I) betadiketonates complexes to form metal and free ligand. In particular, monovalent metal acetylacetonate complexes such as Ni(acac) and Pt(acac) [33] undergo to a spontaneous thermal reduction process. In this case, Ag^+ is reduced to metallic silver by $acac^-$ ligand acting as electron donor, with formation of a radical acac• that, in turn, abstracts a hydrogen atom from cyclodextrin cage, as suggested in Fig. 4, when the inclusion complex between one β-cyclodextrin molecule and a single silver complex molecule occurs. The release of the free ligand involves an H-abstraction reaction by the ligand-centred radical, very efficient due to the availability of 14 hydrogen atoms in the β-cyclodextrin, bonded to secondary carbons and placed really close to the radical center [34].

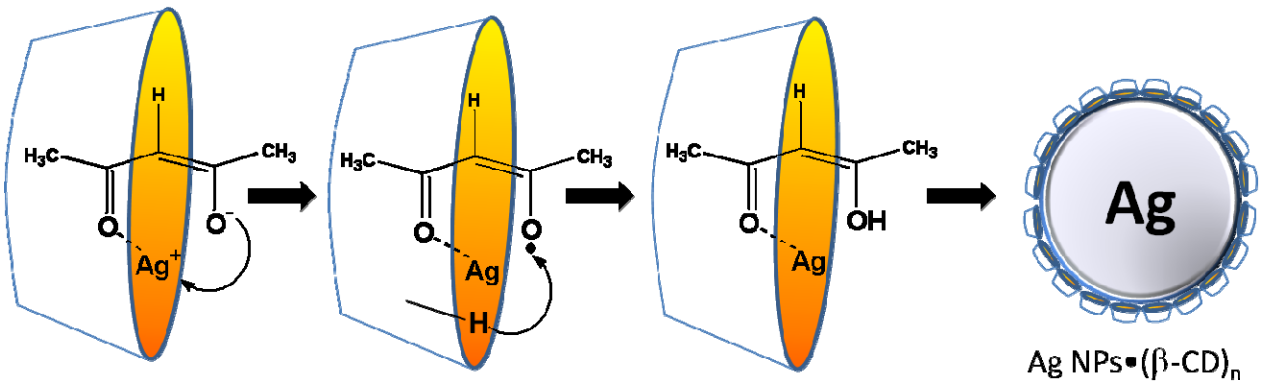


Fig. 4. Reaction mechanism for complex inclusion and Ag nanoparticles formation.

Once reduced to metal, the silver particles aggregate within the β-cyclodextrin microenvironment, allowing solubilization and preventing agglomeration. Since the internal diameter of the β-cyclodextrin cavity is 0.78 nm and the average size of the silver nanoparticles is around 5 nm, spanning from 1 to 10 nm (Fig. 5a and 5b), it can be ruled out that sizes are mainly driven by the inclusion but it is reasonable that a process of metal nanoparticles surrounding with β-cyclodextrin molecules occurs, stabilizing small nanoparticles.

The inclusion phenomena as well as the Ag(acac)/β-cyclodextrin ratio were confirmed by DFT modeling. The calculations evidenced the arrangement of just one Ag(acac) molecule within the β-CD cavity, with the ratio 1:1. As shown in the side view of the inclusion complex (Fig. 5), part of the

Ag(acac) molecule is outside the β-CD ring while the Ag^+ ion is within. The inclusion of the Ag(acac) into to the β-CD moiety resulted thermodynamically favored (ΔE≅80 Kcal/mol) while the free ligand Hacac inclusion process was found less stabilizing, although still advantaged (ΔE≅35 Kcal/mol). The stabilizing energy difference between the two inclusion configurations is due to the specific interaction between the Ag^+ ion and the oxygen in the β-CD cavity.

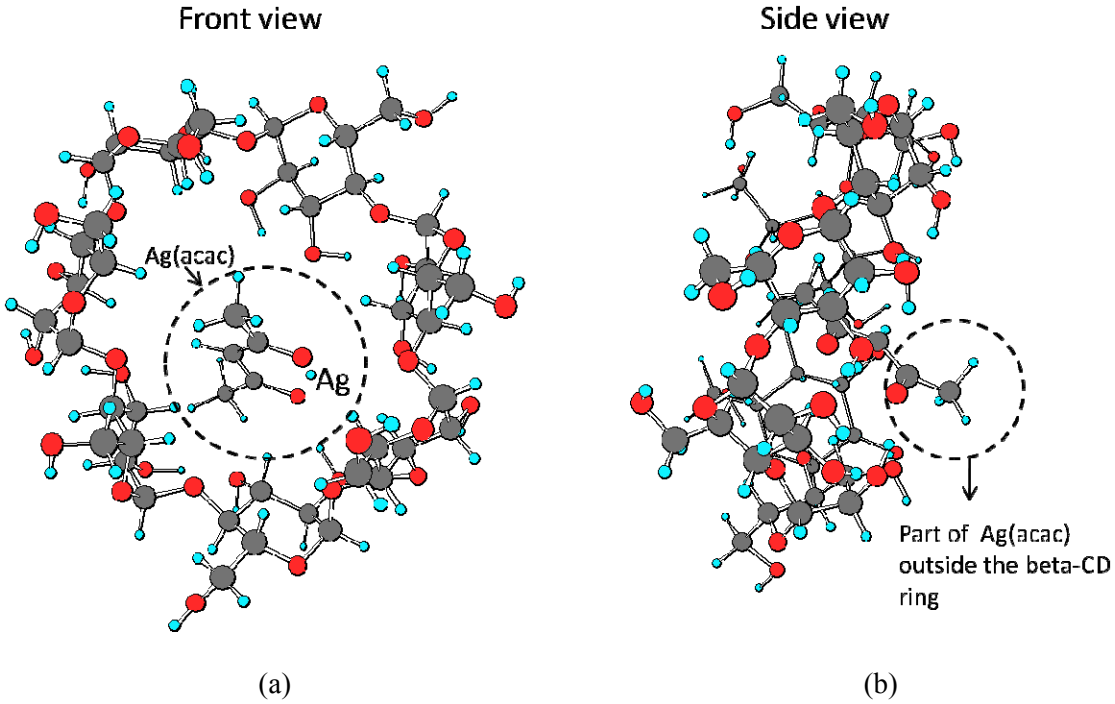
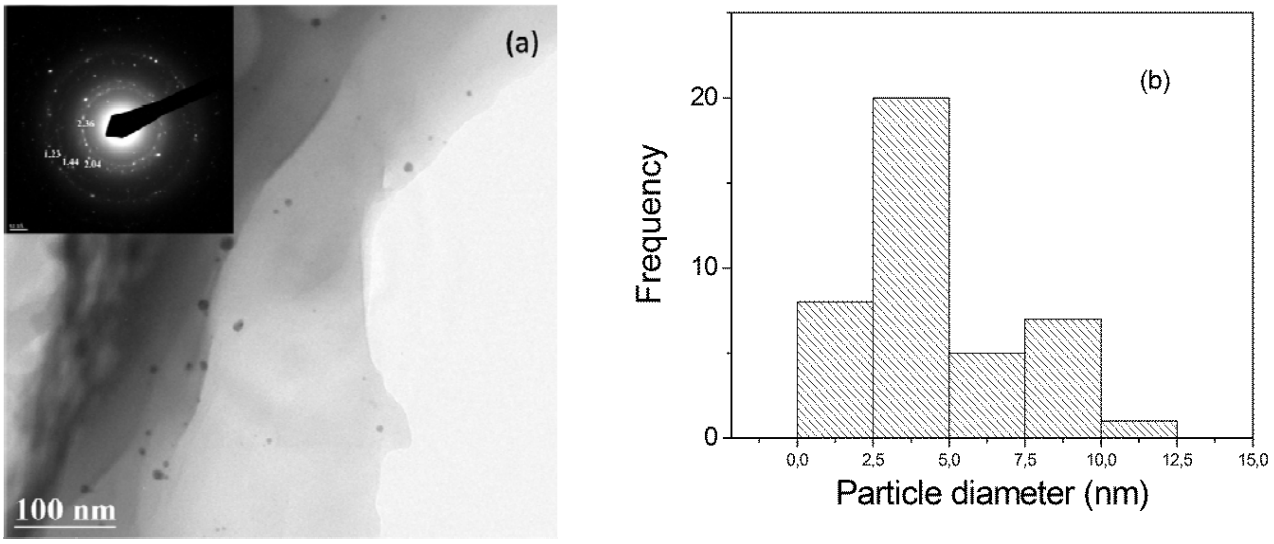


Fig. 5. Inclusion of Ag(acac) in a β-CD molecule.

The second possible pathway consists in a reductive reaction involving the direct participation of a coordinated water molecule [14]. Coordination of a water molecule to bis-coordinated Ag complexes is not uncommon [35]. Literature reports suggest that the reduction potential of water molecules in the first coordination sphere may be significantly lower than that of free water [36] and, in some cases, this may bring to the reduction of the metal centre and oxidation of coordinated water to hydrogen peroxide. Although possible, it is reasonable that this mechanism is not favored in presence of hydrogen donors such as β-cyclodextrin, switching to the predominance of the first suggested mechanism.

In order to characterize the nature and the morphology of the silver nanoparticles, a TEM analysis (Fig. 6) was carried out on some evaporated drops of reacted Ag(acac) 2.3×10^{-4} M solution.

The TEM images evidenced a quite spherical shape for Ag nanoparticles. The diffraction ring pattern of the polycrystalline sample indicates that the structure of the silver nanoparticles is fcc as in the bulk material. The very small dimensions are quite well in agreement with the absence of a quadrupole, assigned at 420 nm [37], only observable with the increasing of the nanoparticles size. The total extinction at 30 min is in agreement with the behavior of nanoparticles below the 30 nm of diameter, for which the only interaction way is the plasmon absorbance. For higher dimensions, the plasmon absorbance is combined with scattering and the correlation between UV-Vis spectrum and size is more complex.



(a) (b)

Fig. 6. (a) TEM analysis of a dried 2.3×10^{-4} M solution reacted till 30 minutes. Inset: electron diffraction; (b) nanoparticles dimensions (diameter) distribution.

4. Conclusions

This paper reports the synthesis of small silver nanoparticles in aqueous medium in presence of β-cyclodextrins acting as microchemical reactors and capping agents. They are promising for sensing activity and applications in materials industry. Silver nanoparticles can be prepared in a single step by spontaneous decomposition of a commercial silver complex in water and β-cyclodextrins, at room temperature. Water is used as benign solvent, while β-cyclodextrins act, on the one hand, as micro-reactors into which the inclusion of the silver complex occurs, followed by a spontaneous intramolecular reduction and, on the other hand, as capping agents for controlling the particle size and stabilizing the nanoparticles in solution. Silver nanoparticles, produced by means of the method here reported, are not poisoned with reductants and stabilizing agents. Moreover, the nanoparticles are capped with biocompatible molecules and all side products are easily removed under vacuum evaporation, enabling their use in a biological medium. This approach to the nanoparticle production appears promising in the field of metal nanoparticles engineering as well as in the developing of nanoparticles-containing new materials.

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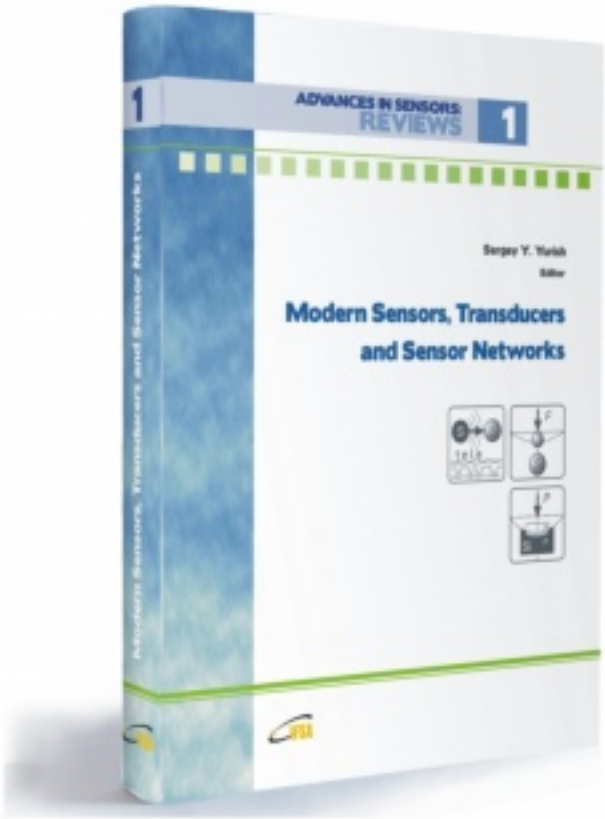

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