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Green Synthesis and Characterization of Colloidal Gold Nanoparticles for Optical Properties

J. Peter, M. Backialakshmi, P. Karpagavinayagam, C. Vedhi*

Department Of Chemistry, V.O Chidambaram College, Thoothukudi – 628 008, TN, India.

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ABSTRACT

Biosynthesis of gold nanoparticles using cold and hot water extract of *pedalium murex* Linn leaf. Prepared gold nanoparticle were analysed by UV-visible spectroscopy, cyclic voltammetric, XRD, SEM and TEM. UV studies shows well-built surface plasmon resonance absorption peak at 540 nm. The band gap energy of 2.95 eV and 2.90 eV achieved for gold nanoparticles prepared by cold and hot water extract respectively. The cyclic voltammetric behaviour of both types of nanoparticles was studied at different pHs. The XRD spectra for deposited thin film samples confirm the crystalline nature and highly stable gold nanoparticles. SEM image shows the nanoparticles are semi-spherical and their sizes are controlled within the range of 180 nm to 200 nm. Hexagonal, triangular, and spherical nanoparticles could be seen in the transmission electron micrographs for both types of particles. The selected-area electron diffraction patterns reveal that the sample is semi crystalline (002) and (111) phase.

1. Introduction

Biosynthesis of nanoparticles using plant extract is currently under exploitation. Plant extracts are very effective, eco-friendly and good alternative for large scale synthesis of nanoparticles. Nanotechnology is the principally attractive area of research associated with production of nanoparticles of variable sizes, shapes, chemical compositions and their possible application for human being benefits. Dimension of metallic nanoparticles was reduced it displayed extraordinary chemical, physical, thermal, optical and electronic properties [1]. Developments in the biologically inspired synthesis of nanoparticles are still in their infancy and consequently attracting the attention of material scientists throughout the world [2]. Bio-organisms are used to synthesize inorganic materials include magneto tactic bacteria (synthesizing magnetite nanoparticles), diatoms (synthesizing siliceous materials) and S-layer bacteria (producing gypsum and calcium carbonate layers) [3].

Nanotechnology is a multidisciplinary science comprising various aspects of research and technology. Nanoparticles are metal particles in the size range of 1 – 100 nm and form building blocks of nanotechnology [4-5]. In recent times, many methods have been designed to synthesize nanoparticles such as physical method, chemical method and biological methods [6-8]. The physical and chemical methods involve the use of strong chemical reducing agents such as sodium borohydride and weak reducing agents like sodium citrate, alcohols, use of gamma rays and UV rays, etc. [9]. A number of bacteria like *bacillus subtilis* [10], *pseudomonas stutzeri* [11], thermonospora sp. [12], shewanella algae [13], *lactobacillus* strains [14], etc. have been studied for the synthesis of metallic nanoparticles. Yeast have also been explored for the biosynthesis of nanoparticles including candida glabrata [15], *schizosaccaharomyces pombe* [16], MKY3 [17] etc. A number of plants like medicago sativa [18], *pelargonium graveolans* [19], *azadirachta indica* [20], triticum [21], *cinnamomum camphora* [22], and *capsicum annum* [23] have been used for the fabrication of metal nanoparticles. The synthesis of nanoparticles by fungi, and their subsequent application, particularly in medicine are studied under myconanotechnology. Myconanotechnology is the interface between 'Mycology' and 'Nanotechnology' and has considerable potential, partly due to the wide range and diversity of the fungi.

Bacillus subtilis 168 were able to reduce Au^{3+} ions to gold nanoparticles with a size range of 5 – 25 nm inside the cell walls [24]. Shewanella algae were found to reduce Au^{3+} ions forming 10 – 20 nm gold nanoparticles extracellularly with the assistance of hydrogen gas [25]. Fungi and actinomycete were also used to synthesize nanoparticles intra- or extracellularly. However, the biosynthesis of gold nanoplate extracellularly is still scarce [26]. Prokaryote bacteria *rhodopseudomonas capsulata* recognized as one of the ecologically and environmentally important microorganisms, commonly existing in the natural environment, were investigated for reducing Au^{3+} ions at room temperature with a single step process. Especially gold nanoplates were formed under the lower starting pH [27].

The microbial enzymes or the plant phytochemical with antioxidant or reducing properties are usually responsible for reduction of metal compounds into their respective nanoparticles [28, 29]. A novel approach for the synthesis and characterization of gold nanoparticles using *ginkgo biloba* [30], fresh water algae *chlorella pyrenoidusa* in optimum pH and temperature [31], *camellia sinensis* [32] were studied. The biological preparation of gold nanoparticles from flowers, fruits, microorganisms and characterization of gold nanoparticles from different sources and applications of the gold nanoparticles in medicine through antibacterial activity against bacteria and fungi was discussed [33].

In this present work, we wish to report green synthesis of gold nanoparticles using cold and hot water extract of *pedalium murex* Linn leaf. Gold nanoparticles were characterized by UV-visible spectroscopy analysis, cyclic voltammetric studies, X- ray diffraction studies, SEM, TEM techniques.

2. Experimental Details

2.1 Materials

Reagents of $HAuCl_4$, 0.1 M H_2SO_4 (pH 1.0), B.R Buffer (pH 4.0), 0.1M KCl (pH 7.0), B.R Buffer (pH9.2) and 0.1 M NaOH (pH 13.0) were analytical grade obtained from Merck (India) Ltd. and used as received without further purification. All the solutions are prepared by ultra-pure deionized water.

2.2 Methods

UV-visible spectroscopy analysis was carried out by a UV–visible spectrophotometer Jasco V-530 between 200 and 1100 nm, possessing a scanning speed of 400 nm/min. Cyclic voltammetric studies were done through computer controlled CH Instruments, performed using a single-

*Corresponding Author.
Ph.: +91 4612310175, +91 9092368104; Fax: +91 4612310275
Email Address : cvedhi@rediffmail.com

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compartment cell with three electrodes, at room temperature. The surface morphology was studied by computer controlled JEOL JSM-5600 LV. The computer controlled XRD system JEOL IDX 8030 was used to record the X-ray diffraction of samples. The exact nano meter size of the particle was characterized by computer controlled PHILIPS CM200 Operating voltages: 20-200kv Resolution: 2.4 Å Transmission Electron Microscopy was used.

2.3 Preparation of Pedalium Murex Linn Extract

Plant material was harvested from in the neighborhood villages and cleaned using water. Exactly 5 grams of cleaned leaf taken in a corning bottle, added 100 mL of deionized cold water and shaked well for 60 minutes in electrical shaker to form highly viscous residual solution. The solution was filtered in ordinary filter paper and used for preparation of Au nanoparticles. Similarly hot water extract also prepared and used to preparation of Au nanoparticles.

2.4 Biosynthesis of Gold Nanoparticles

5 mL of cold water/ hot water leaf extract was added into10 mL of $1x10^{-3}$ M aqueous $HAuCl_4$ solution, stirred in a magnetic stirrer. After 10 min the solution was turn into violet colour then continuously stirred for 6 hours, the pale violet colour solution was observed. The precipitated gold nanoparticles were used for further characterization.

3. Results and Discussion

3.1 UV–Vis Analysis of Gold Nanoparticles

The size and shape of nanoparticles was examined through UV–Vis spectroscopy. The analysis was carried out between 200 nm and 1100 nm, possessing a scanning speed of 400 nm/min. A 0.2 mL of the suspension was diluted in a 2 mL of deionized water and spectrum measured at room temperature. Fig. 1 shows the UV-Vis absorption spectrum of colloidal gold nanoparticle synthesized from cold water (a) and hot water (b) extract of *pedalium murex* L. Spectrum shows a steady increase in the absorbance. A characteristic peak of gold nanoparticles maximum at 540 nm in cold water extract and 549 nm in hot water extract. From these spectral studies identified the formation of stable nanoparticles of gold. The maximum absorption showed the appearance of a single and strong surface plasmon resonance band absorption peak centered at about 540 nm and 549 nm, which indicated that these particles are isotropic in shape and uniform in size. Transmittance spectra of gold nanoparticles coated plate given in Fig. 2. Maximum transmittance of 92% was recorded for cold water extract used gold nanoparticles and 94 % for hot water extract used gold nanoparticles. Since the Au particles shows good optical properties.

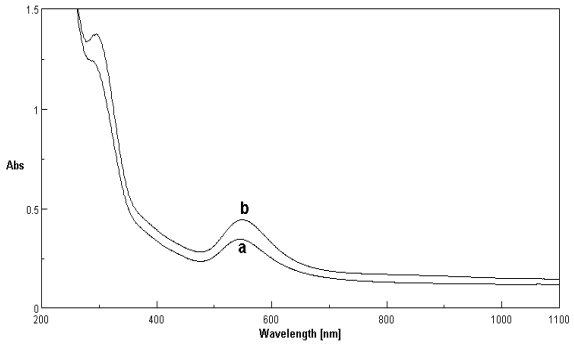

Fig. 1 UV-VIS absorption spectrum of colloidal gold nanoparticle synthesised from cold water (a) and hot water (b) extract of *pedalium murex* L.

The band gap energy can be determined using the tauc relation. It is a convenient way of studying the optical absorption spectrum of a material. According to the tauc relation, the absorption coefficient α for direct band gap material is given by,

$$\alpha h\nu = A(h\nu - E_g)^m$$

where *A* is the optical constant, *α* is the absorption coefficient, E_g is the optical band gap and *m* is an index which assumes the values ½, 3/2, 2 and 3 depending on the nature of electronic transition responsible for the reflection.

For determining the bandgap energy we used the tauc plot. Tauc plot has the photon energy ($h\nu$) on the X axis and a quantity $(\alpha h\nu)^2$ on the Y axis and extrapolating the linear portion of the curve to the X axis yields the bandgap energy of the material. Fig. 3 shows the tauc plot of gold nanoparticles. The band gap energy of 2.95 eV and 2.90 eV achieved for gold nanoparticles prepared by cold and hot water extract respectively.

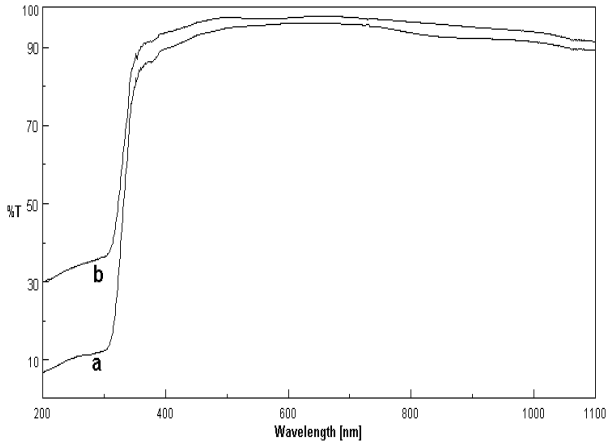

Fig. 2 UV-Vis transmission spectrum of colloidal gold nanoparticle coated plate cold water (a) and hot water (b)

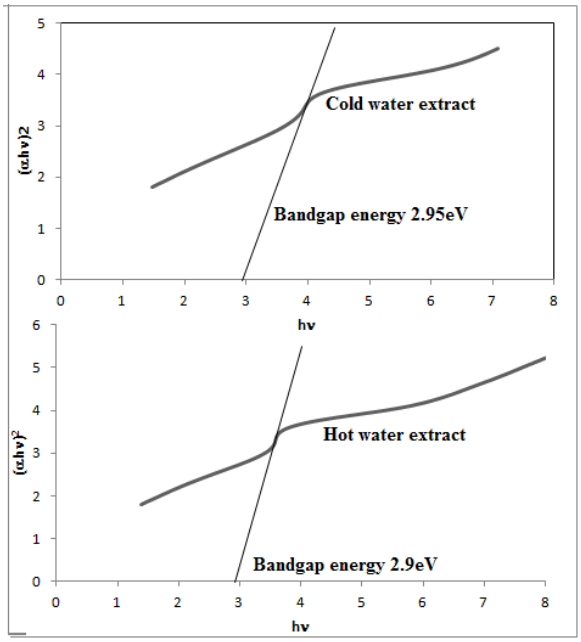

Fig. 3 Tauc plot of gold nanoparticles

3.2 Electrochemical Studies of Gold Nanoparticle

Cyclic voltammetric studies of colloidal gold nanoparticle were carried out at different selected pH from 1.0 to 13.0 in aqueous media. For preliminary studies of cyclic voltammetric behaviour of colloidal gold nanoparticle was coated on GCE and analysed in five selected pHs 1.0, 4.0, 7.0 9.2 and 13.0.

Fig. 4 shows the cyclic voltammetric behaviour of colloidal gold nanoparticles coated on GCE, studied in different pH 1.0, 4.0, 7.0 9.2 and 13.0 at scan rate 100 mV/s. At pH 1.0, one cathodic peak at 0.3 V, 0.25 V and one anodic peak at 0.95 V, 1.05 V was observed for cold water and hot water extract of *pedalium murex* L. used colloidal gold nanoparticles respectively. Similarly other four pHs also shows one cathodic and one anodic peak. As increases the pH of peak response of particles was decreased. Hence gold nanoparticles are more active in acidic medium. Electrochemical behaviour data of gold nanoparticles in different pHs were listed in Table 1.

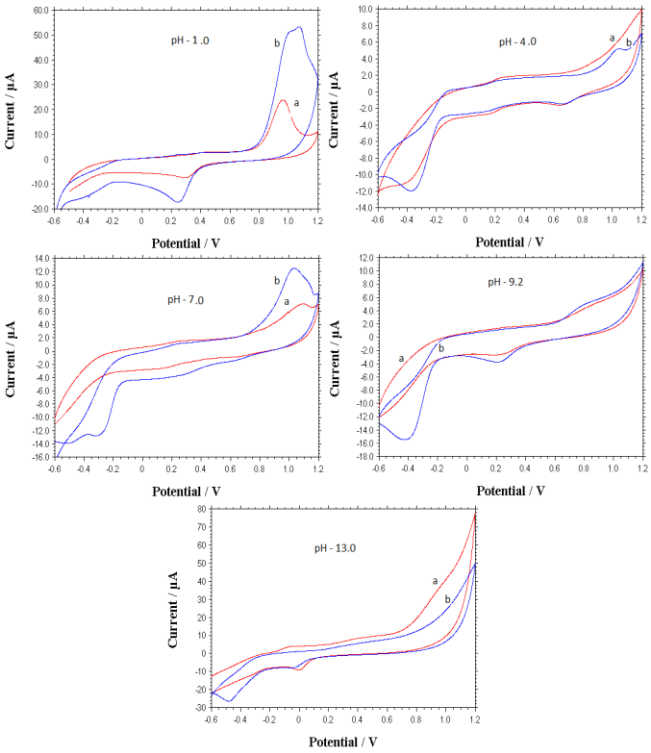

Fig. 4 Cyclic voltammetric behaviour of colloidal gold nanoparticles coated GCE, at different pH 1.0, 4.0, 7.0 9.2 and 13.0: scan rate 100 mV/s. (a) cold water and (b) hot water extract of *pedalium murex* L

Table 1 Electrochemical behaviour data of gold nanoparticles

Medium	Cold water extract				Hot water extract			
	Oxidation Peak		*Reduction Peak*		*Oxidation Peak*		*Reduction Peak*	
	E(V)	i(µA)	E(V)	i(µA)	E(V)	i(µA)	E(V)	i(µA)
pH 1.0	0.9708	23.7	0.3072	7.59	1.0831	25.7	0.2873	6.61
pH 4.0	0.8921	3.89	0.1692	2.77	1.0445	5.21	0.1397	2.38
pH 7.0	1.0889	7.03	0.1046	2.72	1.0341	12.3	0.2738	3.79
pH 9.2	0.9071	5.29	0.2534	2.72	0.8124	4.87	0.2124	3.85
pH 13.0	0.9614	37.8	0.0022	9.45	0.9252	18.5	0.0389	8.28

3.3 XRD Studies of Nanoparticles

X- ray diffraction to confirm the crystalline nature of the particle. Fig. 5 shows the XRD behaviour of colloidal gold nanoparticle coated plates. It is important to know the exact nature of the gold nanoparticles formed and this can be achieved by measuring the XRD spectrum. The peak position at 37.6 represents the presence of gold and the value is consistent. Although the Fig. 5 is in agreement with Bragg's reflection values at two theta, the produced gold nanoparticles exhibit irregular morphology. The XRD patterns clearly show that both the nanoparticles are crystalline in nature. In earlier studies, the biological methods of synthesis gold nanoparticles using plant extract [31], yeast [34], bacteria [35] and fungi have been successfully carried out, and substantiates our present finding of biosynthesis of gold nanoparticles. We believe to the best of our knowledge this is the first report in which a *pedalium murex* L. has been used to synthesize highly stable gold nanoparticles within short period compared to other biological methods.

3.4 SEM Studies of Nanoparticles

SEM image shown in Fig. 6 under the 10,000 magnification, prepared gold nanoparticle are in semi-spherical shape and their sizes are controlled within the range of 120 to 200 nm. It was noted that the particles aggregated with the XRD results. The particle aggregation occurred due to the high surface energy of the nanostructured Au polycrystals. The elemental mapping indicates that the Au nanoparticles are distributed on the surface of the substrate evenly.

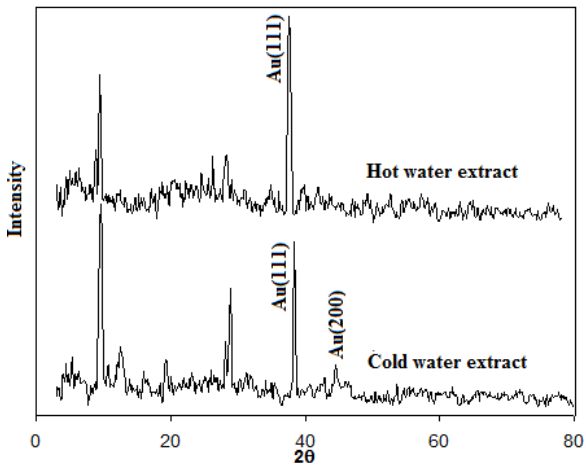

Fig.5 XRD behaviour of colloidal gold nanoparticle coated plate synthesized using hot water and cold water extract of *pedalium murex* L

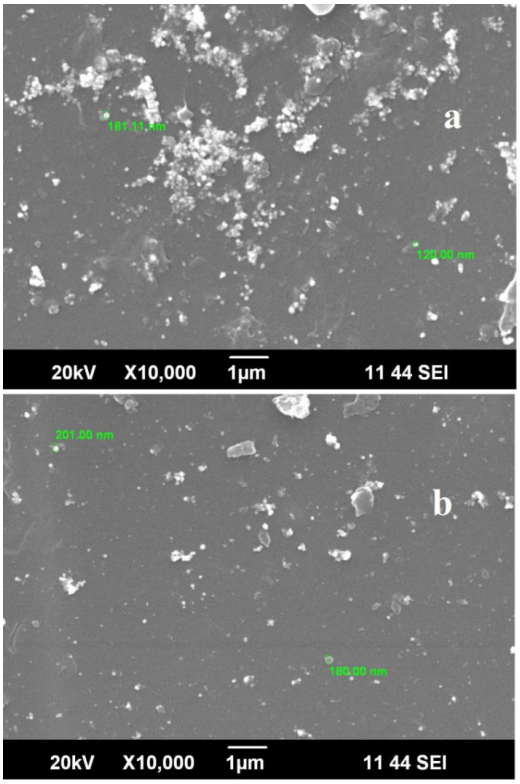

Fig.6 SEM behaviour of colloidal gold nanoparticle coated plate synthesized using hot water (a) and cold water (b) extract of *pedalium murex* L.

3.5 Transmission Electron Microscopy

Fig. 7 represents TEM image and Fig. 8 represents SAED pattern of gold nanoparticles synthesized by the reaction of aqueous chloroaurate ions. This is in concordance with the shift in the ultraviolet spectra of the gold nanoparticles. Hexagonal, triangular, and spherical nanoparticles could be seen in the transmission electron micrographs. It was observed that with hot water extract of *pedalium murex* L. the average size and number of nonspherical gold particles was larger. The lattice fringes with d = 0.235 nm were clearly visible, which could be attributed to the (111) planes of Au. With cold water leaf extract gold nanoparticles were formed in several different shapes, ranging from polydisperse small spheres to large polygons (triangles and hexagons). The lateral sizes of the triangles were in the range of 30 – 40 nm and the thickness was approximately 100 – 120 nm. It is clear that the *pedalium murex* L. releases reducing agents into the solution which are responsible for the formation of gold nanoparticles. The appearance of some darker particles results from an enhanced diffraction contrast due to their orientation with respect to the electron beam. The selected-area electron diffraction patterns reveal that the sample is semi crystalline (002) and (111) phase.

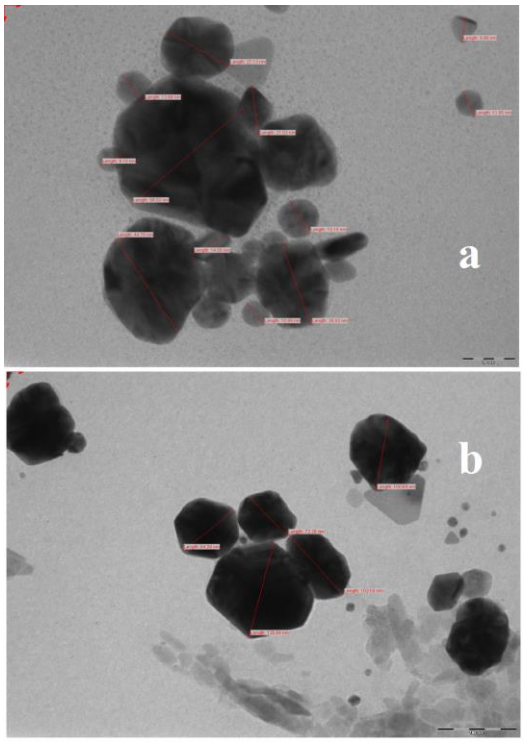

Fig. 7 TEM behaviour of colloidal gold nanoparticle coated plate synthesized using hot water (a) and cold water (b) extract of *pedalium murex* L.

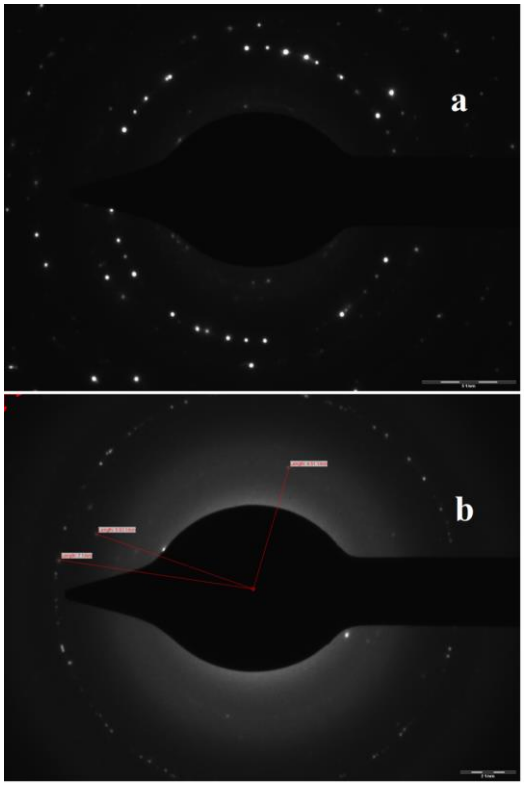

Fig.8 The selected area electron diffraction (SAED) pattern of colloidal gold nanoparticle prepared using hot water (a) and cold water (b) extract of *pedalium murex* L.

4. Conclusion

Cold water and hot water extract of *pedalium murex* Linn were prepared and used as a bio-reducing agent. $1x10^{-3}$ M aqueous $HAuCl_4$ solution was reduced to produce gold nanoparticles. Optical absorbance spectra of gold nanoparticles was observed, strong surface plasmon resonance band absorption peak centred maximum at 540 nm and 549 nm, transmittance value is 92% and 94 % for cold and hot water extract with respectively, which indicated that these particles are isotropic in shape, uniform in size and shows good optical properties.The cyclic voltammetric behaviour of both types of nanoparticles coated on GCE was studied at pH 1.0, 4.0, 7.0 9.2 and 13.0. One cathodic peak and one anodic peak are observed in all pH. As pH increases peak response of particles was decreased, hence gold nanoparticles are more active in acidic medium. The XRD spectra for deposited thin film samples confirm the crystalline nature and highly stable gold nanoparticles. SEM image for gold nanoparticle coated plate shows semi-spherical and their sizes are controlled within the range of 120 to 200 nm. Hexagonal, triangular, and spherical nanoparticles could be seen in the transmission electron micrographs for both types of particles. The appearance of some darker particles results from an enhanced diffraction contrast due to their orientation with respect to the electron beam. The selected-area electron diffraction patterns reveal that the sample is semi crystalline (002) and (111) phase.

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