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paper text:

Synthesis and characteristic of novel hybrid Gold/HPC nanocomposite with more complete coverage of gold nano-shell

11

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Transmission electron microscopy (TEM), TGA ABSTRACT In this study, the

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gold nanocomposite supported on hydroxypropyl cellulose (HPC) using NaBH

as a medium has been prepared successfully. A brownish red solution in 4 its UV –vis absorption spectrum showed surface plasmon resonance absorption bands between 520 and 560 nm in solutions.

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The Au/HPC

nano-composite was characterized by X-ray diffraction (XRD) measurement, transmission electron microscopy (TEM),

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and thermo-gravimetric analysis (TGA). XRD showed the fcc crystal structure of the bulk Au

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with particles of less than 22 nm in size similar to that is observed by TEM

and HPC

is crucial to the formation of such gold

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nano-composite.

TGA confirmed enhanced thermal stability of the polymer.

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1. Introduction

Polymer-protected metal colloids are usually prepared from suitable metal precursors in the presence of protective polymers. It is possible to control the growth and distribution of the sizes of nanoparticles by selecting suitable reaction conditions, such as the concentration of the protective polymer [1]. Although it has been demonstrated

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that combining the desired properties of inorganic and polymeric components into an inorganic/organic hybrid material can be achieved [2], there are, however, only a few papers that describe the synthesis, control of the dimensions, and spatial properties of inorganic/organic hybrid materials.

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Gold nanoparticles are widely studied because they exhibit extraordinary properties that might have strong impact in

different science and technology fields [3-5].

Gold is widely thought of as being one of the most commonly inert of all the metallic elements.

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However, when finely divided and supported upon certain substrates, preferably on an oxide of the first transition series, unique heterogeneous properties are apparent [6, 7]. Much of this original research was established by Haruta et al. during the late 1980s. His work demonstrated the catalytic activity of hemispherical gold particles with diameters of less than 5 nm [8, 9]. In the preparation of low temperature oxidation catalysts, it has been reported that catalytic activity is strongly related to the preparative method of the catalyst

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[10-13]. Conventional

deposition-precipitation and co-precipitation seem better than impregnation methods in preparing active gold samples [7, 12]. It is believed that deposition-precipitation and co-precipitation methods provide a higher level of intimacy of contact between the gold nanoparticles and support matrix

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[13-15].

We have recently developed a reduction method of converting Ag nanospheres into nanorods [16], nanoplates

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[17], their antibacterial activity [18,19] and an improved

an easy synthetic route for silver nano-particles in

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poly (diallyldimethylammonium chloride) (PDDA) [20].

The nano-composites were characterized by various techniques and the preparation and characterization of nanocomposite solutions and film are discussed.

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In this note, we synthesized a new nanogold composite constructed by a different

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polymer.

This method offers great ease of control and a high yield of hexagonal

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hydroxypropyl cellulose-protected nanogold particles (Au/HPC), which were formed a nanocomposite and providing a novel constructed composite nanogold materials. . Use of hydroxypropyl cellulose (HPC)

is well known for nano-particles and therefore, the current methodology offers two fold advantages; (i) availability of functional group, i.e. the hydroxy in HPC are considered to be better suited for a chemical bonding with the metal ions and (ii) high solubility of HPC in

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sodium borohydride (NaBH_4)

as well as use of NaBH_4 being more suitable for

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4 hydrogen tetrachloroauric acid (HAuCl_4) reduction [7, 12]. On the other hand, the presence of hydroxyl groups, which are susceptible to covalent modification, in the Au/HPC nanocomposites stable shell, could open interesting possibilities for cell targeting and slow drug delivery [21].

Controlling the surface properties of nanoscale materials is a major technological research area that touches on studies in the fields of pharmaceuticals, mining, semiconductors, biology,

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and medicine. Coating particles with various types of polymers can determine their ultimate surface characteristics. This paper describes a method for generating nanocomposites that represents a powerful and general strategy for preparing highly functional materials.

2. Experimental 2.1. Materials All chemical substance include hydrogen tetra- chlorauric acid (HAuCl_4), Sodium borohydride (NaBH_4), Hydroxypropyl cellulose (HPC) powder 4 (average molecular weight: 10,000 g/mol, 20 mesh particle size) and CH₃CH₂SH were obtained from Merck. Deionized water

was used during all the experimental procedures. 2.2.

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Characterization

The UV-visible spectrum, in absorbance mode was recorded on an UV-visible Hitachi spectrophotometer model U-2101 PC. The solution form of the sample was prepared by suspending a small amount of

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M. Ghorchibeigy et al. / Journal of Nanostructure in Chemistry 2 (2) (2011) 112 powder in ethanol.

The X-ray diffraction (XRD) pattern

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for gold nano-composite

was recorded on a Seisert Argon 3003 PTC using nickel-filtered $\text{CuK}\alpha$ radiations ($\lambda = 1.5418$

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$^\circ\text{A}$).

TG/DTG analysis was done on

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at a temperature rate of 10° C /min, with nitrogen as carrier gas.

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The samples were rinsed with distilled water, dried and coated with a thin layer of gold by evaporation at vacuum to form conducting film.

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The images of nanocomposites were obtained using a Hitachi H-7500 transmission electron microscope (TEM). Samples for TEM were prepared by dip-coating a solution of a sample on formvar/carbon-film Cu grids (200 mesh, 3 mm).

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2.3. Synthesis of the Gold/

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HPC nanocomposites The typical preparation procedure of the Au/HPC nanocomposites is as follows: the

desired amount of HPC (concentrations in the range 2.13–42 μ M: ca. 0.32–6.

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3 mM) was dissolved in a CH₃CH₂SH/EtOH 3:2 (5 ml, 0.01 mol/L) solution containing HAuCl₄

(1 mM). The solution was stirred for 10 min at room temperature (ca. 25 $^{\circ}$ C). An aqueous solution

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of NaBH₄ (3 mM, 30 ml)

was freshly prepared and rapidly added to the stirring HAuCl₄/polymer solution at a ratio of 1:5 (v/v). The color of the reaction mixtures changes rapidly from yellow to brownish

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red, indicating the formation of gold nanoparticles. UV-vis spectra were obtained before and after the synthesis of the gold nanocomposites (Fig. 3).

The formation process of the sample is illustrated in Scheme.1. 3. Results and discussion Comparisons of hydroxypropyl cellulose (HPC)

during and after the addition of gold precursor revealed significant changes in color. Post processing, all samples took on a uniform brownish red coloring indicating the formation of nano-sized particles of gold. The metallic yellow color commonly associated with bulk gold was not observed.

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Powder XRD patterns obtained for the Au/

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HPC nanocomposite exhibit

four reflections are representative of elemental gold, showing the face-centered cubic structure of metallic gold

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[22].

Inspection of the XRD patterns of Au/HPC (Fig. 1)

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reveals the characteristic gold peaks at a scattering angles (2θ) of 37.96° , 44.32° , 65.16° , 77.48°

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originating from 111 200, 220, 311

crystal planes of tetragonal phase of

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gold.

No other reflections were observed. Average particle size obtained by applying the Scherrer equation to the XRD patterns was 26.1 nm.

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Scheme 1. A schematic diagram of the Au/HPC nano-composites. Fig.1. Powder X-ray diffraction patterns of Au/HPC. 113 M. Ghorchibeigy et al. / Journal of Nanostructure in Chemistry 2 (2) (2011)

Representative TEM images demonstrate the homogeneous dispersion of gold particles throughout the HPC samples.

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But, from the TEM image Fig. 2, no aggregates are obvious among the particles and only some slight change occurred.

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Micrographs shown in Figure 2 confirms the formation of nanoparticles all less than 30 nm in diameter.

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To prove these particles were in fact metallic gold in nature, the UV-vis spectra were obtained. The surface plasmon absorption of gold occurs at a wavelength range of

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approximately 520–560 nm. No absorption peak was observed for the hydro-xypropyl cellulose (HPC) backbone [23].

All UV- vis spectra were recorded at room temperature against the same reference sample.

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The spectral shift is similar to that which occurs in some colloid solutions due to the aggregates of gold particles [24]. The

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UV/visible spectrum of the product (Figure 3) exhibits a small plasmon peak at 520 nm, with a characteristic intensity of gold nanoparticle samples which contain cores of approximately 22 nm [25,26]. It is interesting to note that the post-reduction process produces

an increase in the peak intensity, which indicates a

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larger size

of gold cores. On the other hand,

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the plasmon peak remains in the same position, which rules out nanoparticle core aggregation.

The UV-vis spectra of the different reagents and reference materials used in the

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particle preparation are included in the Supporting Information. Fig.3

shows representative UV-vis absorption spectra characteristic of gold nanoparticles. By the fine control of particle sizes,

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the

color of the solution changes from yellow to brownish red. The peak assigned to HAuCl_4 in the UV-vis spectrums at 230, 430 nm disappears after this reaction, which indicates that the Au^{3+} ions are reduced.

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large gold particle sizes will cause a red-shift to longer wavelengths, lower frequency and lower energies [27–29]. Conversely smaller particle sizes will be blue-shifted to shorter wavelengths, higher frequency and higher energies.

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Fig.2. TEM image of Au/HPC nanocomposite.

Fig.3. UV-Vis absorption spectra of Au/

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HPC

On the other hand, when the progress of the reaction with time for

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gold- hydroxypropyl cellulose (Au/HPC)

was monitored, it was observed (Fig. 4) that the intensity of this band (540 nm) decreases with increase in reaction time thus demonstrating

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the effect of reaction time on the particle growth.

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This figure reveal that the time play

an important role in controlling the gold particle size and size distribution. Shift of

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about 30 nm for this absorption for Au/HPC (Fig.4)

probably indicates the formation of larger sized Au/HPC nanocomposites thus corroborating with the earlier report 3

[20]. Fig.4. TGA/DTG curve for Au/HPC.

The thermal analysis of Au/HPC nanocomposites was conducted to understand the effect of temperature on the stability of the particles as well as the weight loss due to presence of organics around the particles. Thermogravimetry/Derivative thermogravimetry (TGA / DTG) profile of 3

Au / HPC

revealed that the weight loss mainly takes place below 300 °C and there is total loss of about 2.5 % in two steps. The first step loss (~1%) may be due to the adsorbed solvent where as the second step loss (~1.5%) may be due to removal of organic matter associated with the metal particles. After removal of the 3

capped organic materials, slow weight gain is seen in TGA curve after 350 °C possibly due to the oxidation information of 3

gold nanocomposite [30].

This provides a new route for construction of complex nanogold structures. In a 7

plausible reaction scheme (Scheme 2), the Au precursor (HAuCl_4) is reduced by $\text{CH}_3\text{CH}_2\text{SH}$, producing Au_2SR ($\text{R}=\text{CH}_2\text{-CH}_2$) oligomers (steps 1 and 2) [31, 32]. As HSR is added in defect with respect to HAuCl_4 (considering the 3:1 stoichiometric relation), some HAuCl_4 remain in Excess [33]. Then, this reactant and the Au_2SR produced in steps 1 and 2 react with NaBH_4 to form the Au-HPC nanocomposites already described. Shon et al. reported that the reducing agent causes fragmentation of the Au intermediate en route to gold nanoparticles [34]. Furthermore, it was stated that there is a complete breakup of the Au-thiolate species upon addition of NaBH_4 to the reaction mixture. Then, it

is not yet clear whether the observed kinetic hindrance is simply related to the nanocomposite passivation by the polymer layer or if it is also connected to a low rate of the polymer decomposition upon NaBH_4 addition. The final products obtained in both systems (the as prepared and the post-reduced Au-HPC nanocomposites) could be seen as intermediates in the synthesis of thiolate monolayer-protected metallic nanocomposites that, somehow, were not able to be fully reduced. In this regard, the possibility of partially reducing the Au (I) by carrying out the post-reduction indicates that the Au-HPC nanocomposites are not a thermodynamically stable product when NaBH_4 is used. Therefore, a complete reduction of the Au-HPC nanocomposites would lead to thiolate monolayer protected gold nanocomposites. In the current case, as an incomplete reduction of Au (I) was achieved; the metallic gold core is surrounded by a multilayer Au-HPC nanocomposites [35-37]. From the above proposed reaction scheme, it could be claimed that the Au(I)_{RS} oligomers are the precursors of the motifs observed at the surface of thiolate covered gold nanoparticles. 115 M. Ghorchibeigy et al. / Journal of Nanostructure in Chemistry 2 (2) (2011) However, as a thick Au-HPC layer covers the Au cores, it is not possible from our data to propose a structure for the interface between metallic Au and Au-HPC.

Scheme 2. Possible reactions involved in the Au/HPC nanocomposite synthesis. HPC

is a kind of strong anionic polyelectrolyte. Therefore, it is reasonable to consider that the negatively charged HPC chains can further absorb at such positively charged particle surface. However, it may be expected that the adsorption ability of HPC to various planes of gold particle is different.

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This polymer can gradually cover the growing Au clusters, finally blocking completely their growth. Therefore,

different growth kinetics of such planes of gold particles occurs due to the selective adsorption of these chains of HPC to such crystallographic planes of gold particles, which results in the formation of gold nanocomposite with preferential growth direction along the gold {111} plane. However, the detailed formation mechanism is presently not clear and needs further investigation.

4

In our present study, NaBH_4 serves as a reducing agent to reduce AuCl_3 into metallic gold, because there are no other reducing reagents in this system. When gold particles are formed by chemical reduction of AuCl_3 , they can absorb Au^{3+} ions giving positively charged particle surface

4

The present work presents an easy strategy for the controlled synthesis of highly pure gold

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nanosstructures that have many potential applications, such as in sensors, microelectronic systems, and optical coatings.

Analysis of the composite materials by TEM and XRD has allowed the measurement of average crystallite sizes, whilst UV-vis

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has

confirmed these nano-crystallites to be gold in nature.

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The obtained Au particles exhibited little aggregation. The Au/HPC nanocomposites

by this chemical method not only have better crystalline nature but also uniform particle size distribution.

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We also reveal that the glass-transition temperature (T_g) of the Au/HPC nanocomposites is lower than that of the bulk

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HPC.

The current method would provide a simple and cheap way to produce the nanocomposites of gold metal, which has great commercial potential. Acknowledgments The financial and encouragement support

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