

Effect of gamma-irradiation on (PEO/PVP)/Au nanocomposite: Materials for electrochemical and optical applications



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ARTICLE INFO

Article history:

Received 1 October 2015

Received in revised form 18 February 2016

Accepted 19 February 2016

Available online 26 February 2016

Keywords:

Gamma-irradiation

Nanocomposite

Structural properties

UV/vis

TEM

Thermal stability

ABSTRACT

Gold nanoparticles (Au NPs) have been successfully biosynthesized by *Chenopodium murale* (*C. murale*) leaf extract. Au NPs were incorporated within polyethylene oxide (PEO)/polyvinyl pyrrolidone (PVP) blend by casting method. Prepared samples were characterized before and after subjecting to successive gamma-irradiation doses (1, 2, 3, 4, 5 Mrad). XRD confirmed the semicrystalline nature of blend which was largely decreased after embedding Au NPs and subsequently through high irradiation doses. Before irradiation, FT-IR spectra revealed presence of main vibrational bands due to both PEO and PVP. After irradiation, FT-IR spectra indicated the existence of structural rearrangements in the polymeric matrix due to irradiation process. UV/vis spectra indicated presence of $n-\pi^*$ transition results from single-bond in the backbone of blend and showed a visible band at 531 nm attributed to the surface plasmon resonance of Au NPs that is largely affected by irradiation dose. The values of optical parameters were enhanced due to embedding Au NPs and varied according to the irradiation dose. TEM images showed that particle size range was reduced from 1 to 34 nm for nanocomposite to 4–22 nm after exposure to γ -radiations of 5 Mrad. TGA analysis showed an improvement for the thermal stability of nanocomposite sample after gamma-rays treatment.

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1. Introduction

Nanocomposite materials have been of great interest for a number of reasons, which include the materials development with innovative thermal and mechanical properties, optimization of processes and new applications, and the understanding of fundamental phenomena occurring at the nano-scale level [1]. These materials exhibit unique electrical mechanical and optical properties making them valuable for applications in areas like photo-imaging, optics and sensor design, information storage, patterning, and as antimicrobial coatings [2,3].

Polymer blending is a simple and practical method to form new materials. The samples produced by blending of two or more polymers usually result in modified physical and mechanical properties compared to samples made of the initial components. In addition, the blending of synthetic polymers may enhance the cost-performance ratio of the resulting films [4].

Polyethylene oxide (PEO) has a high thermal stability and is a semi-crystalline polymer which has both crystalline and amorphous phases at room temperature due to its high regularity of its structural unit. PEO is a linear polymer with ether linkages (i.e. ether group C—O—C) and can solvate different types of inorganic salts. But, a high ordering of

PEO structure restricts its conductivity so it must be coupled with amorphous polymer. Polyvinyl pyrrolidone (PVP) has been taken as a partner for PEO in preparing polymer blend, due to its individual characteristics. PVP is an amorphous polymer, which can allow faster ionic mobility compared with other semi-crystalline polymers having low scattering losses for different applications. Because of the existence of carbonyl group (C=O) in the PVP side chains, it produces a variety of complexes with different inorganic salts. Another utility of choosing PVP is that it can be thermally cross-linked, producing a good thermal stability and mechanical strength of the blended material and also due to the PVP solubility in water which is a common solvent of PEO, so it is favorable to eliminate phase separation in the polymers of blend [5–7]. Also, PVP is a suitable capping agent for nanoparticles (NPs) due to the presence of both carbonyl group (C=O) and $>N-$ that make anchoring for NPs and having long chain. The functional groups having lone pairs of electrons help in metal NPs stabilization at their surface by covalent interaction, while the polymer chain binds metal NPs aggregation through steric hindrance [8].

Gold nanoparticles (Au NPs) have been widely studied in the past 10 years due to their unique properties, such as quantum size effect, catalytic, and optical properties [9–11] which make them suitable materials for potential applications in various fields. Au NPs have been found to play an important role in many gas sensing processes, catalytic and nano-medicine applications [11]. Also, Au NPs have high performance

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catalysts for the oxygen reduction reaction (ORR) in electrochemical applications, such as fuel cell, which gives decrease to the larger overpotential and higher current density [12].

Many physical properties of polymeric materials such as optical, structural, electrical, thermal, and mechanical properties are modified, after subjecting to γ -irradiation. These modifications are because of the chemical bond scissions and/or cross-linking induced by high-energetic radiation [13]. When an ionization radiation passes through a polymeric material, excitation and ionization for the molecules of material are existed. These lead to breaking of original bonds, chain scission, radical formation and cross-linking in polymeric material. Scission and cross-linking not only depend upon polymer structure but also upon the energy deposited per unit track length (LET). This, in turn, tends to modify the structure and optical properties of polymers [14].

The aim of present study is to analyze structural, morphological and physical properties of (PEO/PVP) blend/Au nanocomposite at different irradiation doses after embedding biosynthesized Au NPs by *Chenopodium murale* extract (*C. murale*) instead of using common hazardous chemicals. The obtained samples are suitable in electrochemical application, as a solid polymer electrolyte, and optical applications.

2. Experimental

2.1. Materials

The leaf of *C. murale* was collected from Mansoura city, Dakahlia governorate, Egypt during December 2014. Tetrachloroauric (III) acid trihydrate ($\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$, 99.5% GR for analysis) was obtained from Merck, Germany. All the chemicals and solvent used in this study were of analytical grade. Both PEO supplied from ACROS, New Jersey, USA with molecular weight (M.W.) $\approx 40,000$ g/mol and PVP from SICO Research Laboratories Pvt. Ltd, Mumbai, India with M.W. $\approx 72,000$ g/mol were used as a basic polymeric materials. The used solvent was double distilled water.

2.2. Preparation of *C. murale* extract and biosynthesis of Au NPs

The leaves of *C. murale* were washed fully thrice with double distilled water. In a 500 ml Erlenmeyer flask, 20 g of washed leaves boiled in 50 ml double distilled water for 30 min at 100 °C. Then the leaf extract was collected in a separate conical flask by a standard filtration method i.e. Whatman No. 40 filter paper. 100 ml of 5×10^{-3} M aqueous solution of gold chloride was prepared in a Stoppard Erlenmeyer flask and 2 ml of leaf extract (0.2 g/ml) was added at room temperature and pH value of the solution was adjusted to 10 with an aqueous solution of 0.1 M NaOH. After one hour in dark, the solution changes from yellow to a pinkish-red solution as shown in Fig. 1, which indicated the formation of Au NPs [2,15,16]. The

resulting solution was centrifuged at 13,000 rpm for 5 min and stored at 4 °C for further use. Finally, powdered pinkish-red colored solid appeared at the bottom of centrifuged tube. This was followed by separation and washing of the powder with double distilled water five times. The powder was then air dried and kept for further use.

2.3. Preparation of PEO/PVP/Au nanocomposite films

Polymer blend electrolyte films were prepared by solution cast technique. A quantity of PEO and PVP (70/30 wt%) was dissolved in double distilled water separately and then the polymer blend solution was stirred continuously at room temperature until a homogenous viscous liquid was formed. Further, 3.6 mg of prepared Au NPs were added to the PEO/PVP polymer solution under continuous stirring for 24 h. Finally, viscous solutions were poured into polypropylene dishes and the common solvent was allowed to evaporate slowly at room temperature for 2 days to obtain free-standing polymer electrolyte films at the bottom of dishes. The obtained films were stored in highly evacuated desiccators to avoid any moisture absorption. Film thickness in the range of 0.07–0.09 mm was obtained.

2.4. γ -Irradiation process

Gamma 1 (type J-3600, Canada, Ltd.) for atomic energy, located at the National Center for Radiation Research and Technology (Cairo, Egypt), was used. A ^{60}Co source, with an activity of 137,000 Ci, was used for the γ -irradiation of the PEO/PVP/Au nanocomposite films. The irradiation doses (1, 2, 3, 4 and 5 Mrad) were measured with radiochromic dye film on Perspex. The dose rate was 2.76 kGy/h. The overall error in the dose measurements did not exceed $\pm 4\%$.

2.5. Measurements

X-ray diffraction scans were obtained using DIANO corporation-USA equipped using Cu K α radiation ($\lambda = 1.5406$ Å, the tube operated at 30 kV, the Bragg's angle (2θ) in the range of 5–80°. FT-IR absorption spectra were carried out for different films using the single beam Fourier transform-infrared spectrometer (Nicolet iS10, USA) at room temperature in the spectral range of 4000–400 cm^{-1} . Ultraviolet/visible (UV/vis) absorption spectra were measured in the wavelength region of 200–1000 nm using spectrophotometer (V-570 UV/VIS/NIR, JASCO, Japan). Transmission electron microscope (TEM), (JEOL-JEM-1011, Japan) was used to study the size, shape and distribution of the biosynthesized Au NPs. TGA curves for the studied samples were recorded using (NETZSCH, STA 409CD, Germany) in temperature range from room temperature to 700 °C and the heating rate was 10 °C/min in the existence of nitrogen as inert atmosphere.

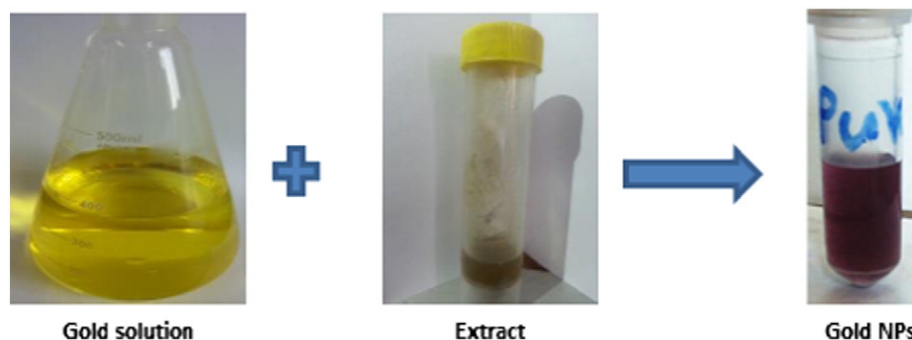


Fig. 1. Biosynthesis of Au NPs by *C. murale* leaf extract.

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