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The effect of graphene loading on mechanical, thermal and biological properties of poly(vinyl alcohol)/graphene nanocomposites

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ABSTRACT

Poly(vinyl alcohol)/graphene (PVA/Gr) nanocomposite was synthesized with the aim of developing a novel improved material. The PVA/Gr nanocomposite was characterized by UV–visible spectroscopy, cyclic voltammetry, Raman spectroscopy, X-ray diffraction, Fourier transform infrared spectroscopy, and X-ray photoelectron spectroscopy analyses, and the interaction of PVA molecules with graphene was examined. Introduction of Gr led to improved mechanical and thermal properties of the PVA/Gr nanocomposite compared to pure PVA, as well as strong antibacterial activity against the bacterial strain Staphylococcus aureus. The PVA/Gr nanocomposite was non-cytotoxic against healthy peripheral blood mononuclear cells by MTT assay, indicating its high potential for biomedical applications.
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Introduction

Poly(vinyl alcohol) (PVA) is a synthetic polymer that is highly hydrophilic, biocompatible, and, most importantly, non-toxic, and can be used as a carrier in drug delivery and a component of biomedical materials [1–4]. Hydrogels are becoming especially attractive in the new field of tissue engineering and regenerative medicine because they can be used as matrices for repairing or regenerating tissues and organs [5]. However, a persisting problem is that hydrogels have poor mechanical properties and/or are too brittle to maintain their shape. In addition, since PVA can be easily dissolved in water stabilizing the PVA film and hydrogel structures against disintegration in water is important for tissue engineering applications; however, most PVA chemical crosslinking agents, including glutaraldehyde, acetalaldehyde, and formaldehyde, are not appropriate for bioengineering because of their cytotoxicity [6–8].

As a result of its extraordinary structure, graphene (Gr), a single layered hexagonal honeycomb lattice consisting solely of sp^2-bonded carbon atoms, has very unique properties, such as high mechanical strength, electrical conductivity, and a large specific surface area, and possesses enhanced transport characteristics [9–11]. Graphene can significantly improve poor mechanical properties or even facilitate drug delivery and targeting of cells [12–14]. Gr can be present in different composite materials in a chemically modified form or in the pristine-like state [15–24]. Even when present at very low concentrations in the final composite, Gr can improve mechanical, thermal, electrical, and gas barrier properties compared to the starting pure polymer [25–29]. Although increased mechanical properties have also been obtained using carbon nanotubes, these reinforcements resulting from the presence of a metallic catalyst usually impair the biological properties of the material or have adverse effects on adjacent tissues. The general aim of using graphene nanosheets as nanofillers is to significantly improve mechanical properties while retaining the original biocompatibility.

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In this study, the nanocomposite of poly(vinyl alcohol) (PVA) and graphene (Gr) nanosheets was synthesized and analyzed to effectively yield a novel improved material. The attractive nature of PVA based composite with graphene as the nanofiller was appealing to many researchers and they have reported improved thermal and mechanical properties of PVA and graphene-oxide nanocomposite [30], reinforcements were documented even at low loading of PVA using partially reduced graphene oxide [26,31]. The others were successful at producing PVA and graphene nanocomposites in the presence of metal catalysts [32] or wrapped in surfactant [10]. However, it has been well established that biocompatibility of graphene-based materials directly depends on the raw materials and production methods used [33]. Use of high-purity, chemically unmodified starting components was the key for obtaining most of the biocompatible composite. Gr is synthesized in relatively pure ways and is therefore expected to show little cytotoxicity, since few metallic catalyst particles are associated with its production. Our aim was to improve on mechanical properties of PVA hydrogels while keeping the filler component (*i.e.*, high-purity graphene) at the minimum. Another important feature is that encapsulation of graphene-based materials into a polymer matrix reduces potential toxicity. The aim of our work was to prepare PVA/Gr nanocomposite colloid dispersion, film, and hydrogel and to investigate the effect of graphene loading on the mechanical, thermal, and biological properties of PVA, as well as to evaluate their potential biocompatibility (*i.e.*, antibacterial activity and cytotoxicity) for use in wound dressings and soft tissue implants and as drug carriers.

Experimental

Materials

The following chemicals were used in this work: fully hydrolyzed PVA powder ("hot soluble", M_w = 70,000–100,000 g/mol, Sigma, St. Louis, MO, USA), and graphene powder (Graphene Supermarket, Calverton, NY, USA). Ultra-pure water from a Milli-Q system (Millipore, Billerica, MA, USA) was used in all experiments.

Sample preparation

PVA/Gr colloid dispersion

PVA powder was first dissolved in hot water (90 °C) to obtain PVA solution with a concentration of 10 wt. % PVA. To prepare a PVA/Gr colloid dispersion, graphene was added to dissolved PVA under vigorous stirring to obtain a final concentration of 10 wt% PVA and 0.01 wt% Gr, and then the dispersion was cooled to room temperature and sonicated.

Preparation of PVA/Gr and PVA films

The prepared PVA/Gr colloid dispersion and PVA solution were poured slowly into a Teflon dish. The solvent was allowed to evaporate at room temperature for 3 days, and then the samples were further dried at 60 °C for 2 days to completely remove the remaining water and yield PVA/Gr and PVA films by casting. Films with an average thickness of approximately 70 μm were peeled off the substrate for testing.

Preparation of PVA/Gr and PVA hydrogels

The prepared PVA/Gr colloid dispersion and PVA solution were poured slowly into a Petri dish and subjected to five cycles of successive freezing and thawing (freezing for 16 h at −18 °C and thawing for 8 h at 4 °C). The obtained hydrogels were cut into small discs (d = 10 mm).

Characterization

UV–visible spectroscopy (UV–vis)

UV–vis spectra of PVA/Gr colloid dispersion and PVA solution were recorded using a UV-3100 spectrophotometer (Mapada, Shanghai, China) at a scanning wavelength between 200 and 1000 nm.

Particle size distribution (PDS)

Particle size distribution (PDS) of PVA/Gr colloidal dispersion, as well as polydispersity index (PDI) were measured by dynamic light scattering (DLS) technique using a Zetasizer Nano ZS (Malvern, Worcestershire, UK) with 633 nm He–Ne laser and 173° detection optics (backscatter detection). The experimental data are the average of 5 separate measurements, each based on 14 runs.

Cyclic voltammetry (CV)

CV measurements of the Pt electrode in PVA/Gr colloid dispersion and PVA solution were performed with two platinum electrodes as working and counter electrodes and a saturated calomel electrode (SCE) as a reference, using a Reference 600 potentiostat/galvanostat/ZRA (Gamry Instruments, Warminster, PA, USA) at a scan rate of 50 mV s^{-1}, in the potential region from −1 to 1 V *vs.* SCE, and starting from the open circuit potential, E_{ocp}. All potentials are given relative to the SCE, and voltammograms plotted are the stationary ones.

Raman spectroscopy

HR-Raman analysis of PVA/Gr and PVA films was carried out using a Renishaw Invia Raman spectrophotometer equipped with a 514 nm argon laser. The intensity used was 10% of total power (50 mW). The spectral range of the analysis was between 3500 and 100 cm^{-1}.

Fourier transform infrared spectroscopy (FT-IR)

FT-IR analysis of PVA/Gr and PVA films was carried out using KBr pellets in a Spectrum One spectrophotometer (Perkin Elmer, Shelton, CT, USA). The scan was performed in the range of 450–4000 cm^{-1} with a spectral resolution of 0.5 cm^{-1}.

X-ray diffraction (XRD)

A Philips PW 1051 Powder Diffractometer with Ni filtered Cu K_α radiation (λ = 1.5418 Å) was used for PVA/Gr and PVA films using the scan-step technique in the 2θ range of 8–80° with a scanning step width of 0.05° and exposition time of 50 s per step. The phase analysis was realized using the PDF-2 database with a commercially available computer program, EVA V.9.0. The average crystallite domain size (D_p) was calculated from the half height width ($\beta_{1/2}$) of the XRD reflection of the (0 0 2) plane, using the Scherer Eq. (1):

$$D_p = \frac{K\lambda}{\beta_{1/2}\cos\theta} \quad (1)$$

where, λ is the wavelength of the X-ray radiation, K is the shape coefficient equal to 0.9, and θ is the diffraction angle.

X-ray photoelectron spectroscopy (XPS)

XPS measurements of the PVA/Gr and PVA films were performed using a K-Alpha System (Thermo Fisher Scientific, Waltham, MA, USA) equipped with Al Kα X-ray radiation (1486.6 eV) and a micro-focused monochromator. The elemental profiling was carried out by Ar-ion sputtering.

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