



Assessment of morphology and property of graphene oxide-hydroxypropylmethylcellulose nanocomposite films



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ABSTRACT

Graphene oxide (GO) was synthesized by Hummer's method and characterized by using Fourier transform infrared spectroscopy and Raman spectroscopy. The as synthesized GO was used to make GO/hydroxypropylmethylcellulose (HPMC) nanocomposite films by the solution mixing method using different concentrations of GO. The nanocomposite films were characterized by X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM) and thermo-gravimetric analysis. Mechanical properties, water absorption property and water vapor transmission rate were also measured. XRD analysis showed the formation of exfoliated HPMC/GO nanocomposites films. The FESEM results revealed high interfacial adhesion between the GO and HPMC matrix. The tensile strength and Young's modulus of the nanocomposite films containing the highest weight percentage of GO increased sharply. The thermal stability of HPMC/GO nanocomposites was slightly better than pure HPMC. The water absorption and water vapor transmission rate of HPMC film was reduced with the addition of up to 1 wt% GO.

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1. Introduction

In recent years, polymer nanocomposites fascinated the mind of many researchers because of their enhanced properties arising in the presence of nanofillers [1]. A diversity of carbon nanomaterials such as carbon nanotubes and graphite nanoplatelets have been used for preparing polymer nanocomposites [2,3]. Currently, graphene oxide (GO) has also attracted a great deal of attention due to its unique structure and properties. It also can be used as a carbon precursor to produce graphene and its composites in bulk quantity. These materials display extraordinary properties such as mechanical stiffness and chemical stability. GO itself is an insulator, almost a semiconductor, with differential conductivity between 1 and $5 \times 10^{-3} \text{ S cm}^{-1}$ at a bias voltage of 10 V [4]. GO consists of a two-dimensional (2D) sheet of covalently bonded carbon atoms bearing various oxygen containing functional groups (e.g.

hydroxyl, epoxide, and carbonyl groups) on their basal planes and edges [5]. It contains both sp^2 and sp^3 hybridized carbons bearing hydroxyl and epoxide functional groups on its "basal" plane [6] whereas the edges are mostly decorated by carboxyl and carbonyl groups [7]. Therefore, GO is hydrophilic in nature and can be readily dispersed in water as individual sheets to form stable colloidal suspensions [3]. Meanwhile, these oxygen-containing groups present in GO sheets are responsible for strong interactions with polar small molecules or polar polymers to form GO intercalated or exfoliated composites [8]. As most of the synthetic polymers are non-biodegradable, so today in an environment-sensitized age the applications of bio-degradable polymers are gaining interest for different applications. But, bio-degradable polymers have lower mechanical, thermal and water resistance properties compared to synthetic polymers. Hence to boost the above said properties, bio-degradable polymers can be blended with synthetic or natural polymers [9] or by adding nanofillers such as GO [10] and also by cross linking [11]. On the contrary, it has been widely demonstrated that the composites of polymers and nanofillers can combine the ductile property of polymer matrix and high strength of nanofillers [12].

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Preparation of biodegradable polymer/GO nanocomposites is the most crucial part towards enhancement of properties such as mechanical, thermal and water resistance. As GO consists of a layered structure of hydrophilic 'graphene oxide' sheets, intercalation of water molecules between the layers readily occurs. The interlayer distance between the graphene oxide sheets increases reversibly from 6 to 12 Å [13] with increasing relative humidity. Notably, GO can be completely exfoliated to produce aqueous colloidal suspensions of graphene oxide sheets by simple sonication and by stirring the water/GO mixture for a long enough time. So, Xu et al. reported synthesis of strong and ductile nanocomposite of PVA/GO [14]. It was also reported that GO can form a mechanically strong and thermally stable composite with sodium alginate [15].

Hydroxypropylmethylcellulose (HPMC) is a bio-degradable and bio-compatible polymer and widely used for different applications [16]. It is also water soluble. So, we have preferred HPMC for preparing nanocomposites films with GO. GO based composites of bio-degradable polymers and nanofillers can combine the ductile property of the polymer matrix and high strength of nanofillers due to its various functional groups which improve interfacial interactions between graphene oxide and polar polymer matrix [17].

In the present work we prepared mechanically strong GO/HPMC nanocomposite films at different wt% of GO (0.02, 0.1, 0.2, 0.3, 0.4, 0.7, 1 and 1.3%). The formation of GO was confirmed by using the Fourier transform infrared (FTIR) and Raman spectroscopic analysis. GO/HPMC nanocomposite films were prepared by the solution casting process. The nanocomposites of GO/HPMC were characterized by X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM) and thermo gravimetric analysis (TGA). Mechanical properties, water vapor transmission rate (WVTR) and moisture absorption of nanocomposites films were also measured.

2. Experimental

2.1. Materials

HPMC (50 cps) was purchased from Central Drug House (P) Ltd., New Delhi, India. Graphite powder was received from Sigma-Aldrich. Concentrated sulfuric acid (98% H₂SO₄, GR grade), potassium permanganate (KMnO₄ purified) and hydrogen peroxide solution (30% H₂O₂), sodium nitrite (NaNO₂, extra pure), sodium carbonate (Na₂CO₃) were received from Merck Specialties Private Ltd., India.

2.2. Graphene oxide synthesis by Hummer's method

The synthesis of GO from graphite powder was carried out by following the Hummers and Offeman method [18,19]. At first, 23 mL of 98% concentrated H₂SO₄ was cooled down below 0 °C in an ice bath. Separately, 1 g of graphite powder and 0.5 g of NaNO₂ were mixed together. This mixture was then added to cooled (0 °C) concentrated H₂SO₄ and kept on constant stirring in an ice bath for 45 min, so that the chemicals got sufficient time to mix with each other. Then, 3 g of KMnO₄ was added slowly and gradually to the solution. The solution color immediately turned greenish black in color from black. The solution was removed from the ice bath and allowed to attain the room temperature. After the attainment of room temperature, the solution was kept in oil-bath with constant stirring and the color of the solution changed to brown. MilliQ water (140 mL) was added followed by the addition of 7 mL of 30% H₂O₂ solution to terminate the reaction and kept on stirring for 15 min. The dark brown colored solution changed to yellow in color. The solution was left to settle overnight. Then, the product was filtered. At this time the pH of the solution was very close to 2 which indicated its

high acidic character. Then, the product was centrifuged followed by repeated washing to remove acidity. Finally, the product was vacuum dried at 50 °C to obtain graphene oxide (GO) powder.

2.3. Preparation of HPMC/GO nanocomposite films by solution mixing method

HPMC/GO nanocomposite films were prepared by the solution mixing process. A homogeneous solution of 1% aqueous HPMC (w/v) was prepared by mixing a definite amount of HPMC in MilliQ water with continuous stirring. A series of GO aqueous suspensions (0.02, 0.1, 0.2, 0.3, 0.4, 0.7, 1 and 1.3 wt%) were prepared using ultrasonication for ~2 h. Vigorous stirring for ~4 h of the mixture of 1% HPMC and prepared GO dispersions produced HPMC/GO nanocomposites. In order to fabricate the nanocomposite films, the solutions were then transferred into a glass petri plate at ambient temperature for the complete evaporation of water. After 72 h (complete evaporation of water), the films were peeled off from the petri plate and kept in vacuum pressure of 60 kPa for 30 min. Finally, the nanocomposite films of appropriate thickness were obtained.

2.4. Characterization

2.4.1. FTIR spectroscopy

FTIR spectroscopy was done to confirm the formation of GO and also to define the characteristic peaks of oxygen containing groups of GO such as epoxy, carbonyl, hydroxides etc. FTIR spectrum was performed in PerkinElmer spectrum Express Version 1.03.00 instrument in the range of 250–4500 cm⁻¹.

2.4.2. Raman spectroscopy

Raman spectroscopy is highly sensitive to the electronic structure and has proven to be an essential tool for the characterization of carbon-based materials, especially C=C double bonds that lead to high Raman intensities. We have done this analysis to confirm the formation of GO [20]. The Raman spectra was monitored using 1.96 eV (633 nm) line of a He-Ne laser in HORIBA-JOBIN-YVON Lab RAM HR 800 instrument by placing the sample solution into a semi-micro stopper cuvette with an exposure time of 1 s.

2.4.3. X-ray diffraction

X-ray diffraction analysis of GO and its nanocomposite films with HPMC were performed at room temperature by X-PERT-PRO Pan analytical diffractometer using Cu Kα (λ = 1.5406) as X-ray source at a generator voltage of 40 kV and current of 30 mA. The scanning rate was 1° min⁻¹. From XRD data, the interlayer spacing of GO and nanocomposites were calculated using Bragg's law as follows:

$$d = \frac{\lambda}{2 \sin \theta} \quad (1)$$

where d is spacing (nm) between two lattice plane, λ the wavelength of X-ray beam (nm), and θ is the angle of incidence.

2.4.4. FESEM analysis

Field emission scanning electron microscopy was used at different magnifications to observe the surface morphology of the GO/HPMC nanocomposites films. Before testing, the samples were coated with a thin layer of gold to avoid electrical charging during examination. FESEM study was performed in a Zeiss Auriga instrument.

2.4.5. Mechanical properties

The mechanical properties of the solution casted HPMC and its nanocomposite films with GO were examined using a Zwick Roell

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