



SYNTHESIS AND CHARACTERIZATION OF GRAPHENE OXIDE/POLY (GLYCIDYL METHACRYLATE) NANOCOMPOSITES

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ABSTRACT:

Graphene/graphene oxide (GO)-based polymer article is of great importance owing to its numerous functionalities. In this study, we successfully synthesized, a novel graphene oxide polymer nanocomposite (GO/PGMA). GO/PGMA had been synthesized by surface-initiated Atom transfer radicle polymerization technique and characterized by XRD, TEM and Raman spectroscopy. GO/PGMA dosage and temperature are also performed.

Keywords: Graphene oxide; Polymer; Nanocomposite; SI-ATRP

INTRODUCTION:

The single atom layer carbon material graphene oxide (GO) attracts a large number of attention due to its unique structural and different physical/chemical properties [1-3]. Due to the large specific surface area and high biocompatibility makes GO is a helping in supporting material for proteases conjugation via the surface hydroxyl or carbonyl groups of GO [4-6]. However, the potential of GO has not been fully exploited because of the relatively limited amount of functional groups on GO and possible unfavourable orientations of the surface conjugation proteases [7,8]. Graphene oxide (GO) is the perfect candidate for the fabrication of artificial nacre with several functional groups on its surface [9]. The GO-based composite has shown different application for biological support such as energy storage, high power anodes [10], drug delivery, super capacitors [11] etc. Graphene oxide (GO) and graphene-based composites have been the interesting topic for many research groups around the world [12]. The strong interfacial interaction and the different layered structure of the graphene or GO-based composite are responsible for their unique mechanical performance. Two-dimensional graphene or GO sheets that served as the layered structure at nanoscale have ultrahigh tensile

strength and modulus. During the construction of graphene or GO-based composite the interaction of the graphene or GO sheets has become the strategic point [13]. Graphene or GO-based composite are mainly constructed by three approaches, such as hydrogen bonding, ionic bonding and covalent bonding. Furthermore, different polymers have been attached to graphene sheets to give electrically conductive, fluorescent, transparent and flexible materials [14, 15]. Subsequent incorporation of polymer or polymeric material [16], helps in reducing graphene sheet agglomeration [17] giving rise to interesting and different graphene - polymer structures in the form of brushes [18], micelle, sponge etc. have also been realized.

MATERIAL AND METHODS

Graphite powder, glycidyl methacrylate (GMA), N,N,N',N',N''-pentamethyldiethylenetriamine, (PMDETA) were purchased from Alfa Aesar, India. α -Bromoisobutyryl bromide (BIB) was obtained from Spectrochem Pvt Ltd., India. All other materials used were of AR grade. De-ionized (DI) water has been used throughout present study.

Characterization

Morphological characterization was carried out using TECNAI 200 kV TEM (Fei, Electron Optics) equipped with digital imaging and 35 mm

photography system. Raman spectrums were recorded by RenishawInvia Reflex Micro-Raman spectrometer in which the sample was excited by 514 nm wavelength Ar⁺ laser. X-ray diffraction patterns were recorded using X-ray diffractometer (Model No. D8 DISCOVER) [19].

Synthesis of GO and GO-Br

GO was synthesized by improved hummer's method as reported earlier [32]. GO-Br was synthesised by sonication 200 mg of GO was 10 ml DMF for 1 h. 8 ml TEA and 12 mL BIB were added to the resulted suspension at 0 °C. The product was collected via filtration after 24 h and subsequently washed by chloroform (3x20ml) with deionized water (3x20ml) followed by dried under high vacuum for 18 h.

Synthesis of Graphene oxide/Poly (glycidyl) methacrylate (GO/PGMA)nanocomposite

100mg of GO-Br was sonicated in 5ml of DMF for 1 h followed by addition of 6 ml GMA, 50mg CuBr and 75 µl PMDETA. Reaction mixture was degassed using three freeze pump thaw cycles and heated for 24 h at 100 °C. Resulting viscous solution was poured into 30ml ethanol to precipitate the product and remove unreacted monomer and catalyst followed by some more washing of the product by ethanol (3x50ml), product was dried overnight in vacuum at 80 °C.

RESULTS AND DISCUSSION

X-Ray diffraction and Raman Spectroscopic studies of GO/PGMA

X-ray diffraction pattern and Raman spectra of GO and GO/PGMA are illustrated in Figure1. XRD patterns of GO in Figure 1(a) shows its characteristic peak at $2\theta = 9.74$ with an interlayer spacing of 9.066 Å represents plane <001> and at $2\theta = 42.64^\circ$ plane <100> with interlayer spacing of 2.12 Å. Broad merged peaks encircled in Figure 1(a) depicts XRD pattern of GO/PGMA representing the fused peaks of PGMA and RGO, formed due to the co-reduction of GO to RGO. Raman spectra of Graphene oxide characterised by the presence D

and G peaks. The characteristic D peak in graphene is due to breathing mode j-point phonons of A_{1g} symmetry associated with sp³ bonded carbon atoms in disordered graphene whereas, peak G is assigned due to first order scattering of E_{2g} phonons in sp² bonded carbon atoms. Raman spectra of GO and GO/PGMA are shown in Fig. 1 (b). Raman spectra of GO shows G peak at 1600 cm⁻¹ and D peak at 1350 cm⁻¹. Same bands for GO/PGMA are observed to be shifted due to polymer functionalization on GO surface. Intensity ratio of D and G band (I_D/I_G ratio) indicates disorderness in the material are found to be 0.86, 1.34 for GO, and GO/PGMA respectively. Increase in the I_D/I_G ratio from GO to GO/PGMA is due to increased sp³ character attributable to growth of polymer brushes over GO surface.

Morphological Characterization

TEM has been used for morphological characterization of GO and GO/PGMA as depicted in figure 2. Fig 2(a), TEM micrograph shows characteristic sheet like morphology with wrinkles of GO. Whereas, figure 2(b) shows the GO/PGMA micrograph where small spherical structures of Poly(glycidyl) methacrylate over surface of GO can be observed. These polymers are giving a porous morphology to GO/PGMA, which is contributing to its adsorption properties.

CONCLUSION :

GO/PGMA had been synthesized by surface initiated Atom transfer radicle polymerization technique. GO/PGMA was characterized by X-ray diffraction, Raman spectroscopy and TEM for morphological characterization. PGMA was used as an active adhesive which is produce a layered sheet of graphene oxide/polymer composite. Now compared with other composites the GO/PGMA composites exhibited enhanced mechanical and electrical properties, such as toughness, higher tensile stress, and dielectric constant. Furthermore, the GO/PGMA composites shown better mechanical properties than other GO-based films. Thus, the

GO/PGMA composites show extensive potential applications such as electronic and engineering materials.

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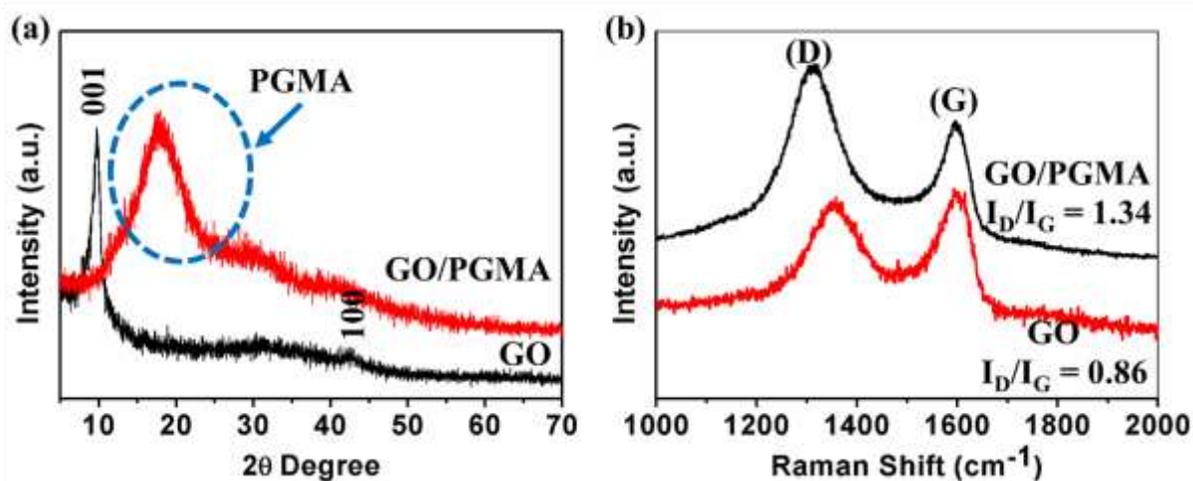


Figure 1, (a) XRD and (b) Raman Spectra of GO and GO/PGMA

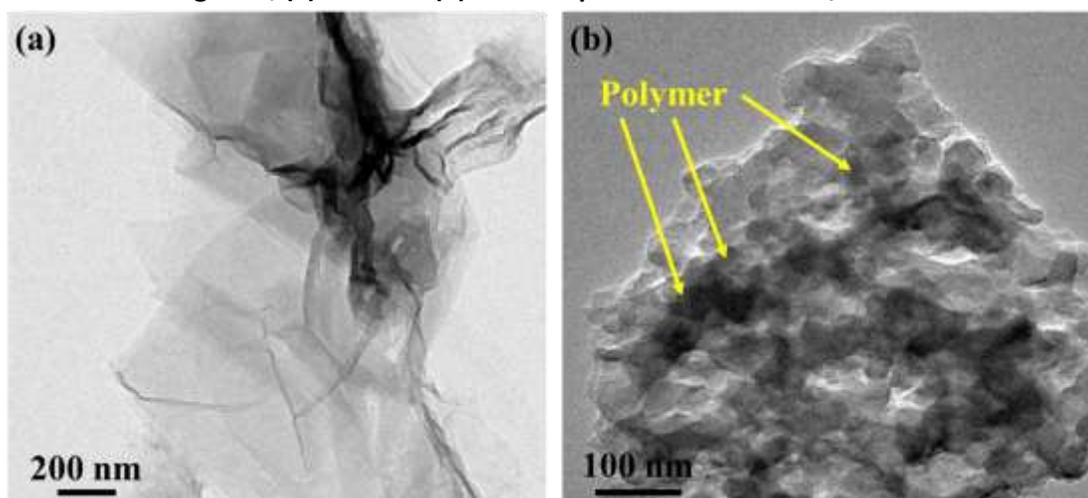


Figure 2, (a) TEM micrograph of GO and (b) GO/PGMA