



# Polyacrylamide grafting of modified graphene oxides by in situ free radical polymerization



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## ABSTRACT

Graphene oxide (GO) was modified using chemical reactions to obtain three types of functionalized GO sheets (FGO). The FGO sheets and the GO were then subjected to in situ free radical polymerization in order to study the grafting polymerization. The FGO and grafted-FGO were analyzed with Fourier transform infrared spectroscopy, scanning electronic microscopy, thermo-gravimetric analysis (TGA) and X-ray photoelectron spectroscopy (XPS). The grafting percentages in the materials were calculated using the TGA and XPS results. The FGO sheets with different functional groups exhibited different grafting abilities, and hydroxyl groups were proven to be the most reactive grafting sites for the in situ free radical grafting polymerization of polyacrylamide.

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## 1. Introduction

Graphene has attracted a great deal of attention owing to its excellent electronic [1–7], thermodynamic [8] and mechanical properties [9–13], which arise from its flat monolayer structure of sp<sup>2</sup>-bonded carbon atoms that tightly packed into two-dimensional honeycomb lattices. However, it is chemically inert and hydrophobic, so graphene does not dissolve in polar solvents like water. This narrows its applications. One possible route to solve this problem is to modify the graphene with functional molecules. For example, surfactants have been used to disperse graphene in solvents through physical adsorption [14,15]. Graphene can also be chemically modified by transforming it to graphene oxide (GO) which contains numerous oxygen-contained functional groups [16]. These functional groups allow the graphene to then be further modified. One approach is grafting modification with macromolecules. Here, the functional groups on the GO sheets, including the hydroxyl, epoxy, and carboxyl groups are possible grafting sites for the macromolecules [17].

Several methods have been attempted to prepare macromolecule grafted graphene, including atom transfer radical

polymerization (ATRP) [18–20], reversible addition fragmentation chain transfer processes (RAFT) [21,22], nitroxidemediated polymerization (NMP) [23,24] and in situ free radical polymerization (FRP) [25,26]. Among these methods, FRP is the simplest and most universal because the other methods all require monomers and special reaction conditions. Water-soluble PAM-g-GO (polyacrylamide grafted GO) has been prepared by FRP [27–29].

An adjustable composition (or grafting percentage) is desirable in grafted graphene and a high grafting percentage is especially good. The grafting percentage is dependent on the number of grafting sites on the graphene. Therefore, the grafting sites play an important role in grafting polymerization. For example, alkyl halides, thioesters, and alkoxyamines have been incorporated onto the surface of GO nanosheets to act as grafting sites in ATRP, RAFT and NMP, respectively [19]. However, the nature of the grafting sites in FRP has not been determined and needs to be studied in detail [30,31].

In this paper, GO was modified using chemical reactions to obtain three types of functionalized GO (FGO) nanosheets. Then the GO and the FGO were grafted with polyacrylamide (PAM) to prepare PAM-g-GO and three kinds of PAM-g-FGOs (PAM grafted FGOs) by FRP. The grafting was verified by Fourier transform infrared spectroscopy (FTIR), scanning electronic microscopy (SEM), thermo-gravimetric analysis (TGA) and X-ray photoelectron spectroscopy (XPS). The grafting percentages of these grafted

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graphene materials were determined by TGA and XPS and the reactive grafting sites for FRP were determined by comparing the grafting percentages of the PAM-g-GO and PAM-g-FGOs.

## 2. Experimental

### 2.1. Materials

Graphite was obtained from Qingdao Graphite Factory. *N,N*-dimethylformamide (DMF), thionyl chloride ( $\text{SOCl}_2$ ), tetrahydrofuran (THF), ethylene glycol ( $\text{HOCH}_2\text{CH}_2\text{OH}$ ), hydrogen bromide (HBr), oxalic acid ( $\text{HOOC}-\text{COOH}$ ), phenyl isocyanate, methylene chloride, acrylamide (AM), azodiisobutyronitrile (AIBN) and other reagents were all purchased from Tianjin Chemical Reagent Co. All

the chemicals (except DMF) were analytical grade and used as received.

### 2.2. Preparation of GO-g-PAM and FGO-g-PAMs by FRP

#### 2.2.1. Preparation of hydroxyl-functionalized GO (HGO)

GO was prepared from purified natural graphite by a modified Hummer's method [32–34]. GO (50 mg) was suspended in  $\text{SOCl}_2$  (20 mL) and stirred for 24 h at  $65^\circ\text{C}$  in a four-neck round-bottom flask equipped with a condenser,  $\text{N}_2$  inlet and magnetic stir bar. After the reaction, the excess  $\text{SOCl}_2$  was removed by alternating centrifugation and re-dispersion steps using THF. The resulting solid was then suspended in  $\text{HOCH}_2\text{CH}_2\text{OH}$  (20 mL) in a round-bottom flask equipped with a condenser and the suspension was

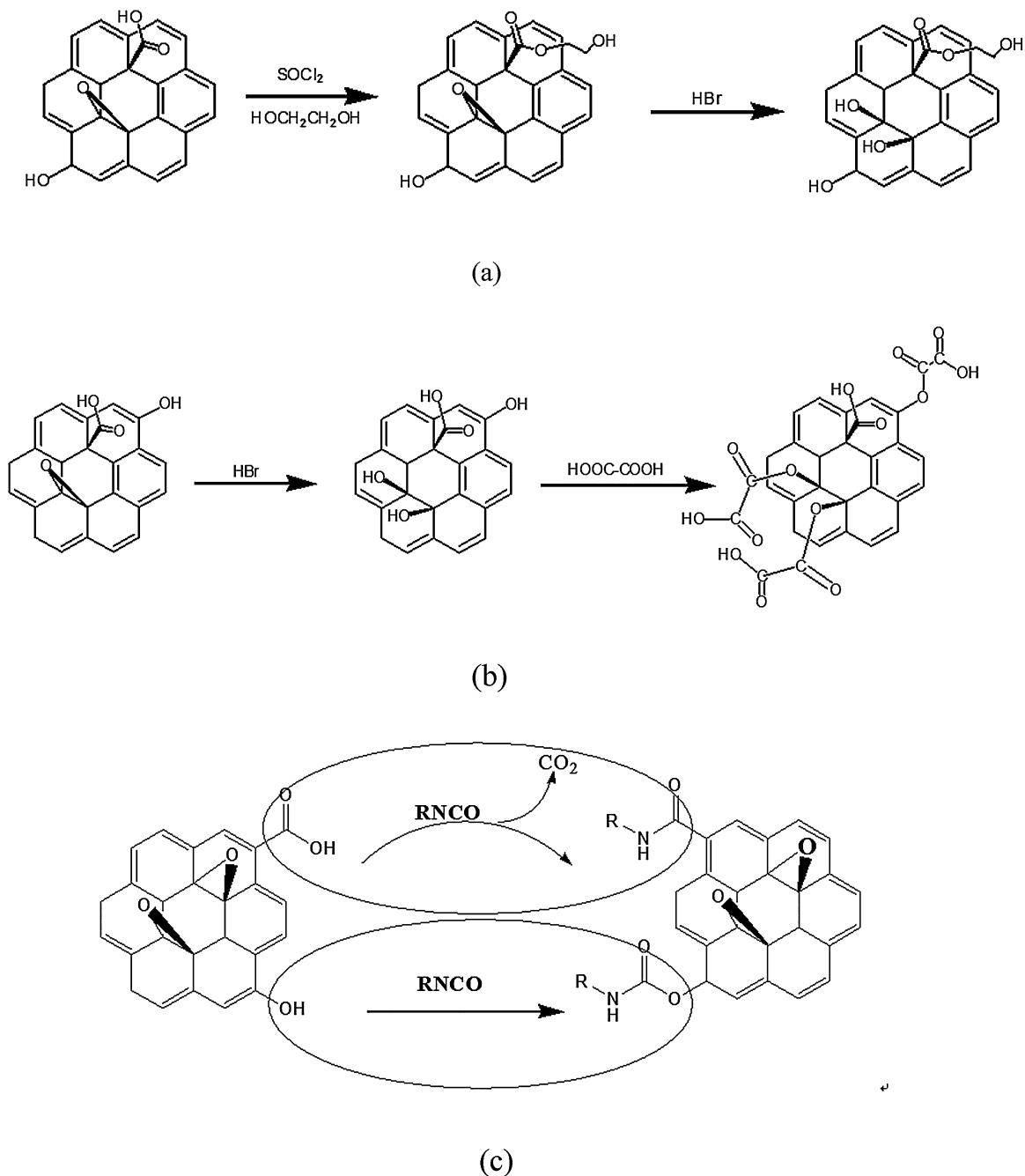


Fig. 1. Schematic preparation process of HGO (a), CGO (b), and EGO (c).

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