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Letter to the Editor

Single step preparation of meso-porous and reduced graphene oxide by gamma-ray irradiation in gaseous phase

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ABSTRACT

A facile and highly efficient route to produce simultaneously porous and reduced graphene oxide by gamma ray irradiation in hydrogen is here demonstrated. Narrowly distributed nano-scale pores (average size of ~3 nm and surface density >44,900 pore μm^{-2}) were generated across 10 μm thick graphene oxide bucky-papers at a total irradiation dose of 500 kGy. The graphene oxide sheet reduction was confirmed to occur homogeneously across the structures by Fourier transform infrared spectroscopy and Raman analysis. This one-step, catalyst-free, high penetration and through-put technique, offers great promises potential for the mass production of reduced graphene oxide from cheap graphene oxide.

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The fabrication of graphene, a material made of two-dimensional stacked sheets of sp2-hybridized carbon atoms, is a highly dynamic field in materials science. Graphene has been previously prepared by chemical vapor deposition, mechanical exfoliation or thermal oxidation of graphite, and thermo-chemical reduction of graphene oxide (GO). Novel processing routes for altering the morphology of graphene sheets have attracted both theoretical and experimental interest as a result of its unique properties and their potential translation into engineering applications. Processes to produce porous graphene, where nano-pores are generated in the plane of graphene sheets, have especially attracted increasing attention for their potential applications in sensing, energy storage, membrane separation, biological sequencing, composite materials and nano-electronics [1–3]. The size of the pores generated across graphene sheets may vary from single atomic openings to nano-scale pores, opening the route to fabrication of extremely porous, thermally and electrically conductive meta-materials of high surface area. Recently, 2D poly(phenylene), a porous graphene with single-atom-wide pores and sub-nano-meter periodicity, was successfully synthesized by self-assembly of alkyl substi-

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tuted dendrimers, resulting in pristine graphene sheets with a theoretical pore width of ~2.48 Å [4]. Electron or ion beam irradiation also introduced high density arrays of pores in the range of 2–40 nm across graphene sheets, despite local oxidation damage around the pores [5]. Steam and chemical etching also led to the facile perforation of graphene or GO, and materials with broad pore size distributions and enhanced specific surface area [6,7]. Despite being highly innovative, these techniques either offer limited opportunity for up-scaling and mass production, or lead to highly oxidized and damaged porous graphene structures requiring further post-treatment to restore the graphene properties.

Here we demonstrate a highly novel and unique approach to simultaneously reduce and perforate a high density network of pre-synthesized graphene oxide to produce high pore density porous graphene sheets with narrow pore size distribution by gamma-ray (γ-ray) irradiation. These modifications were performed solely with γ-ray irradiation, without introducing any chemical or catalyst precursors, rendering a process with no chemical requirements or waste streams. Although radiation induced modifications such as the exposure of carbon nanotubes (CNTs) to soft X-rays in air were demonstrated to produce massive surface oxidation and functionalization a dozen of micrometers deep into CNT bucky-papers (BPs) [10], CNTs were previously shown to be highly resistant to γ-rays (up to 11.7 MGy in air) [8]. Functional groups, such as carbonyl or carboxylic moieties present on graphene surfaces facilitate functionalization and decoration reactions, and routes to produce high surface densities of chemical groups were consequently investigated [9].Here, graphene oxide BPs formed by vacuum filtration of GO [10] were irradiated for the first time in pure gaseous phase hydrogen with γ-rays generated from a cobalt-60 source for a range of doses generating nano-scale pores.

Although the morphology of the irradiated GO BPs were shown to be largely un-affected by the irradiation at low total dose, up to 100 kGy, high density pores were formed at 500 kGy across the GO sheets (Transmission Electron Micrographs (TEM) – Fig. 1). The pore distribution was found to be narrow (average slightly smaller than 3 nm, standard deviation of ±0.72 nm) while the pore coverage density was up to 42,924 pores μm^{-2}, corresponding to an approximate surface porosity of 33.5% which is the largest GO surface porosity reported to date [2–5] (Figs. 2A and B, 3A and S.I.). The pore distribution was confirmed by Small Angle X-ray Scattering (SAXS) analysis of the 500 kGy sample where a clear a broad Guinier knee was found at 0.05 $Å^{-1}$, corresponding to an average pore size of ~4.25 nm (Fig. 3B and C – see S.I.). In addition,

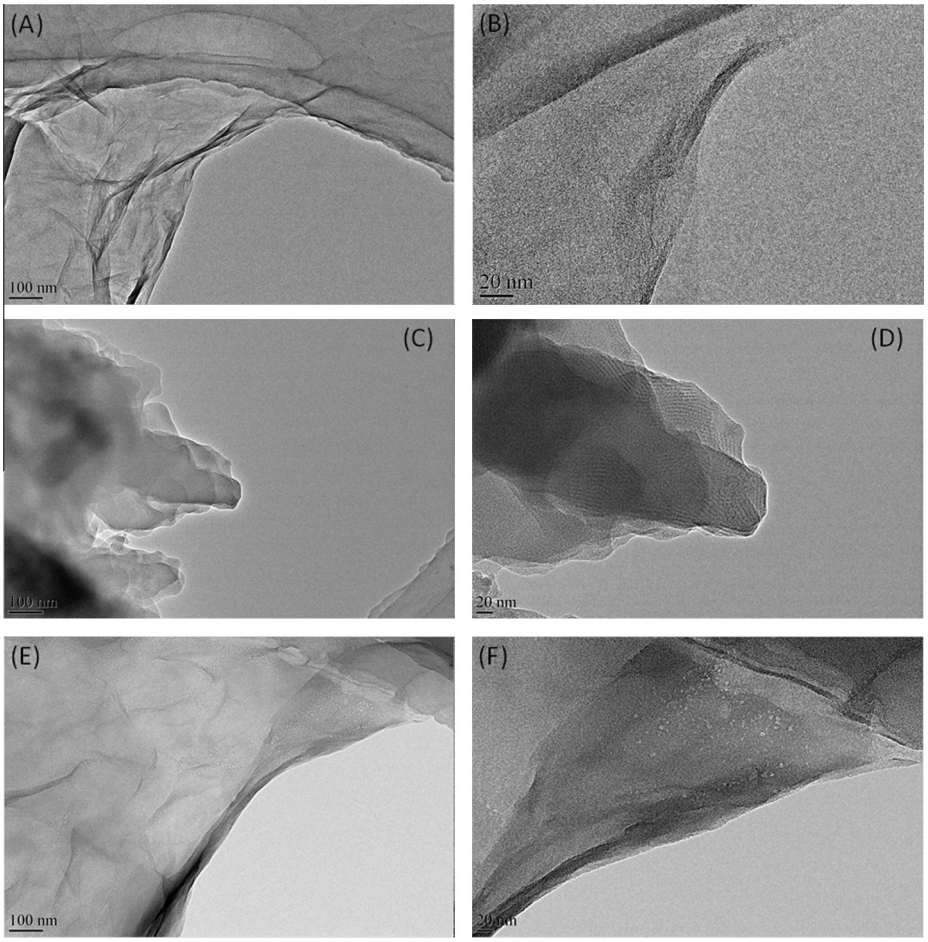

Fig. 1 – Transmission Electron Micrographs (TEMs) of (A, B) pristine GO sheets (C, D) 100 and (E, F) 500 kGy GO sheets; the samples were dispersed in water prior to be filtered onto a carbon lacey TEM grid; tests were performed at 200 kV on a JEOL JEM-2100 (Japan) under high vacuum. Pores were found on all the examined 500 kGy irradiated GO sheets.

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