

Supplementary information

Integration of Polymerized Ionic Liquid with Graphene for Enhanced CO₂ Adsorption

P. Tamilarasan and S. Ramaprabhu*

*Alternative Energy and Nanotechnology Laboratory (AENL), Nano Functional Materials
Technology Centre (NFMTC), Department of Physics, Indian Institute of Technology Madras
(IITM), Chennai, India*

ESI 1. Structural and elemental compositional analysis

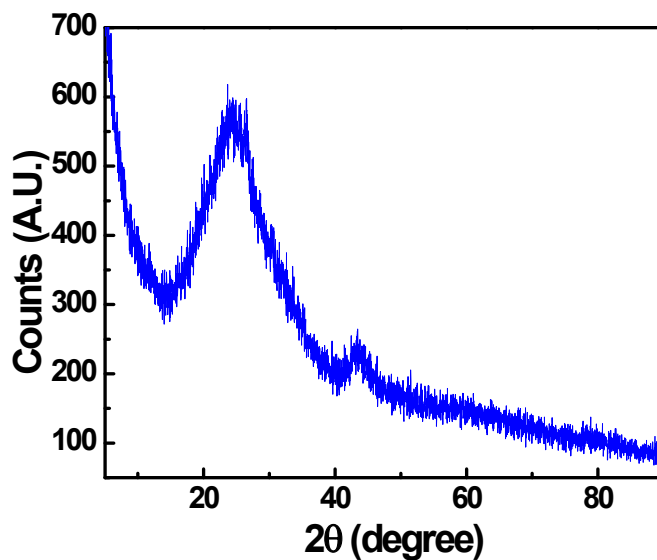


Figure S1. X-ray diffraction pattern of HEG.

Figure S1 shows X-ray diffractogram of HEG, which reveals a broad (002) diffraction peak, centered at 24.2° , corresponding to the inter-layer distance of ~ 0.37 nm. The rapid removal of functional groups results in exfoliation of graphene layers, which results in disordered graphitic structure. The broadening of (002) peak suggests the distribution of interlayer distance in HEG due to the presence of wrinkles and residual functional groups. The weak intensity of (002) peak evidences high degree of disorder in graphitic structure and formation of few layer graphene.

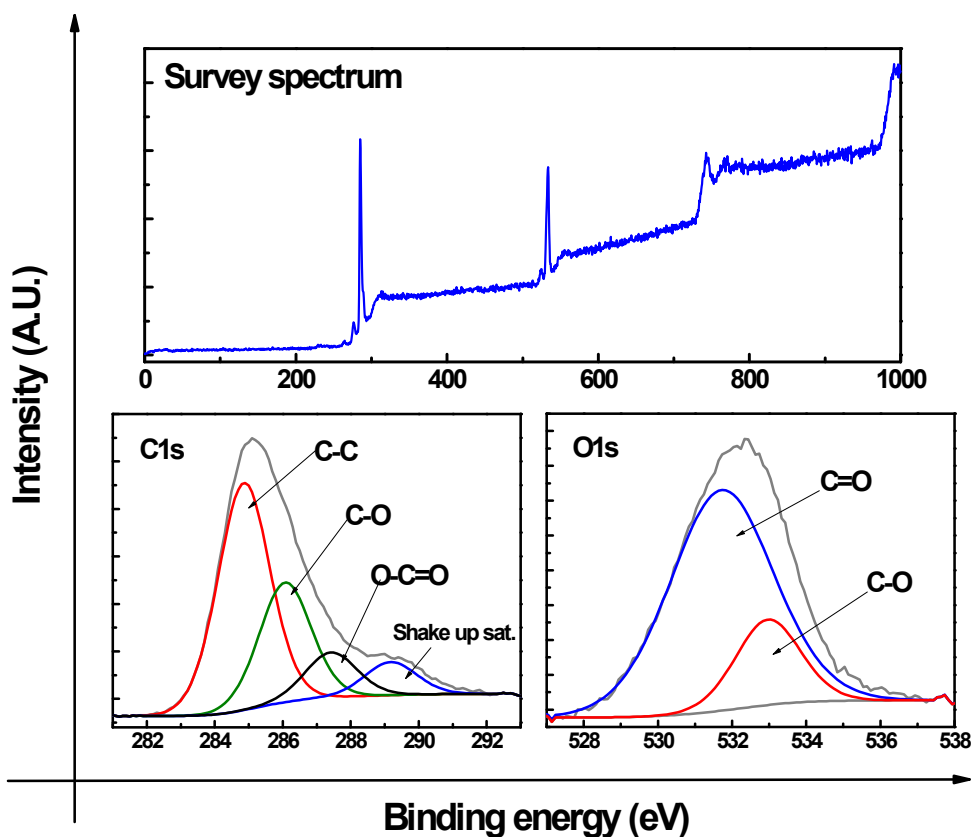


Figure S2. X-ray photoelectron spectrum of HEG.

The X-ray photoelectron spectrum of nitrogen doped graphene (N-HEG) is recorded by SPECS X-ray photoelectron spectrometer equipped with Mg K α (1253 eV) X-ray source and PHOIBOS 100MCD energy analyzer at ultrahigh vacuum (10^{-8} Pa). The survey spectrum (Fig. 2.15(a)) shows the presence of nitrogen along with carbon and oxygen with 6.1 carbon to oxygen ratio (C/O ratio). This can be imputed to the presence of residual oxygen containing functional groups. The deconvolution of C1s peak show the features corresponding to C-C, C-O, O-C=O and shake up satellite peaks. Similarly, C-O and C=O signals are identified by the deconvolution of O1s peak. These oxygen containing functional groups facilitates better CO₂ adsorption as well as function as anchoring sites to IL or PIL moieties.

ESI 2. Procedure for adsorption analysis

Low pressure carbon dioxide adsorption-desorption studies were carried out by pressure reduction technique, where a surface area analyzer (Micromeritics ASAP 2020) was employed. The adsorption analyses were carried out with 10 s equilibration intervals at 2 % relative tolerance and 200 Pa absolute tolerance (whichever less is considered at each equilibrium pressure). The surface area analyzer is capable of operating up to 100 kPa pressure. Hence, the adsorption isotherm was recorded at various equilibrium pressures up to 100 kPa.

In a typical experiment, a dried sample tube equipped with a seal frit (20 μ m pore size) and filling rod was weighed on a microbalance. Around 200 mg of sample was loaded to the sample tube along with filling rod, which was then capped by seal frit and connected to the activation port. The sample temperature was raised to 373 K at 10 K min⁻¹. At this temperature, sample was degassed and activated for 3 h. The degassed sample tube was weighed again and the sample mass was determined by subtracting the total mass of empty tube, seal frit and filling rod. This

method gives high accuracy (± 0.001 mg) in sample mass. The activated sample was loaded into the analysis port, where high purity CO₂ gas was used for analysis throughout the adsorption experiment. The volume inside the sample cell (free space) was calculated using high-pure helium gas, at corresponding temperature of analysis. Here, the sample temperature was maintained using thermally insulated water bath. The adsorption-desorption measurement was carried out by pressure-swing adsorption method, where the sample was regenerated at reduced pressures. The sample cell was dosed with CO₂ to a known pressure and the drop in pressure was monitored. The number of moles of adsorbed gas on the adsorbent has been calculated using van der Waals equation.

In desorption, the pressure inside the sample cell was reduced step by step and the equilibrium pressure was determined. The amount of adsorbate retained on the surface of adsorbent was also calculated using van der Waals equation, from the equilibrium desorption pressure.