Electronic Supplementary Information (ESI)

Highly Ordered Cubic Mesoporous Silica/Graphene Nanocomposite

Chang-Wook Lee,a Kwang Chul Roh,*,b and Kwang-Bum Kim*

*E-mail: kbkim@yonsei.ac.kr

Experimental Section

Preparation of KIT-6/graphene nanocomposite

KIT-6/GO was prepared in GO containing aqueous solution, using a 1:1 (wt%) mixture of Pluronic P123 ($EO_{20}PO_{70}EO_{20}$, MW=5800, Aldrich) and butanol, with 0.5 M HCl at 35 °C. Tetraethoxysilane (TEOS) was used as the silica precursor. In a typical synthesis procedure, 6 g of P123 was dissolved in 217 g of distilled water, 6 g of butanol (Aldrich, 99.4%), and 11.8 g of conc. HCl (35%). To this, 1 g of GO was added under stirring at 35 °C. After stirring the resulting solution for 24 h, 12.9 g of TEOS (ACROS, 98%) was added at 35 °C (TEOS:P123:HCl:H_2O:BuOH = 1:0.017:1.83:195:1.31 in mole ratio). The mixture was left under stirring for 24 h at 35 °C, and subsequently refluxed for 1 h at 100 °C under microwave irradiation (STARTSYNTH Labstation, Milestone, USA). After the microwave treatment, the KIT-6/GO thus obtained was filtered and dried at 100 °C. Finally, KIT-6/GO was heat treated at 400 °C in air, followed by an additional heat treatment at 700 °C in 5 wt% H_2/Ar. For comparison, KIT-6 was synthesized using the same procedure, in the absence of GO. The as-synthesized KIT-6 was heat treated at 400 °C in air.

Characterization

The TEM images were recorded on a Philips CM200 microscope operated at 200 kV. The SEM images were recorded on a Hitachi S-4300SE microscope operated at 15 kV. The low-angle XRD patterns were recorded on a Rigaku D/MAX 2200V/PC X-ray diffractometer using Cu Kα radiation (40 kV, 20 mA) in the 2θ range 0.8–5°, at intervals of 0.2°. The FTIR spectra of the samples in KBr pellets were recorded on a Mattson 3000 FTIR spectrometer in ATR mode. The spectra were collected in the wavelength range 500–4000 cm^{-1} with a 4 cm^{-1} resolution over 120 scans. The Raman spectra were measured using a Jobin-Yvon LabRam HR with an LN_2-cooled charge-coupled device (CCD) multichannel detector at room

temperature, employing conventional backscattering geometry. The nitrogen adsorption-desorption isotherms were measured on a Micromeritics ASAP ZOZO at 77 K, and the surface area was calculated by the Brunauer-Emmett-Teller (BET) method. XPS measurements were performed using an Omicron ESCA Probe (Omicron Nanotechnology, Taunusstein, Germany) with a monochromated Al Kα radiation (hm = 1486.6 eV). Thermogravimetric analysis was carried out using a STA 409 PC differential thermal analysis-thermal gravimetric analyzer (Netzsch, Germany) from 25 to 900 °C with a 10 mL min^{-1} airflow at a heating rate of 10 °C min^{-1}. The AC impedance (VMP2, Biologic) of the as-prepared KIT-6/graphene nanocomposites was measured with a pellet in the form of a disc, using 2-point probe method in a cell. For KG-400-700 and KG-700, the electrical conductivity was calculated from the impedance data measured in the frequency range from 10 mHz to 100 Hz, at an AC voltage of 10 mV. For KG-400, electrical conductivity was measured in the frequency range from 10 mHz to 100 mHz at an AC current of 10 nA, under a constant pressure of 8000 psi.

Supporting Information

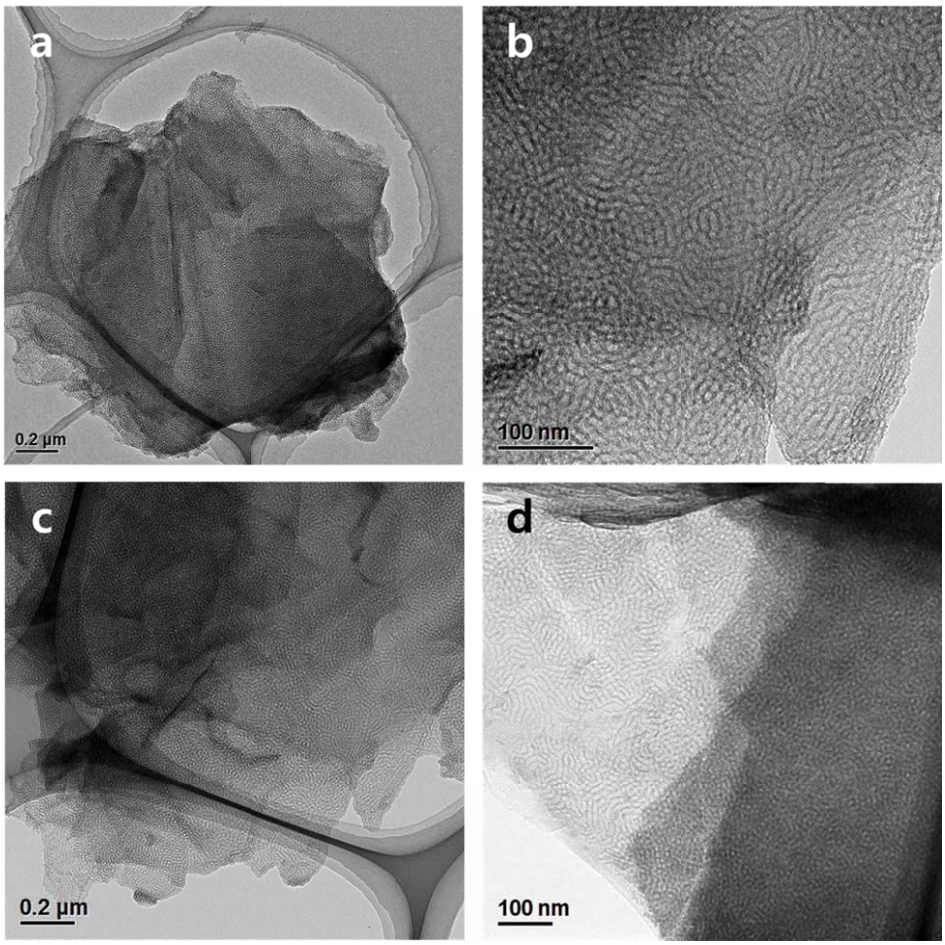

Figure S1. Typical TEM images of disordered mesoporous silica/graphene nanocomposite with different loading amount of (a-b) 66 wt.% and (c-d) 50 wt.%.

Figure S1 shows the typical TEM image of disordered mesoporous silica/graphene nanocomposites prepared at the bulk concentration of P123 of 1.38 and 0.69 wt.%. Under these conditions, the loading amount of mesoporous silica in nanocomposite is 66 and 50 wt.%, respectively. While the KIT-6/graphene nanocomposite shows the extensive domains of cubic Ia3d structure (see Figure 1b of the revised manuscript), no structural ordering could be found in Figure S1.

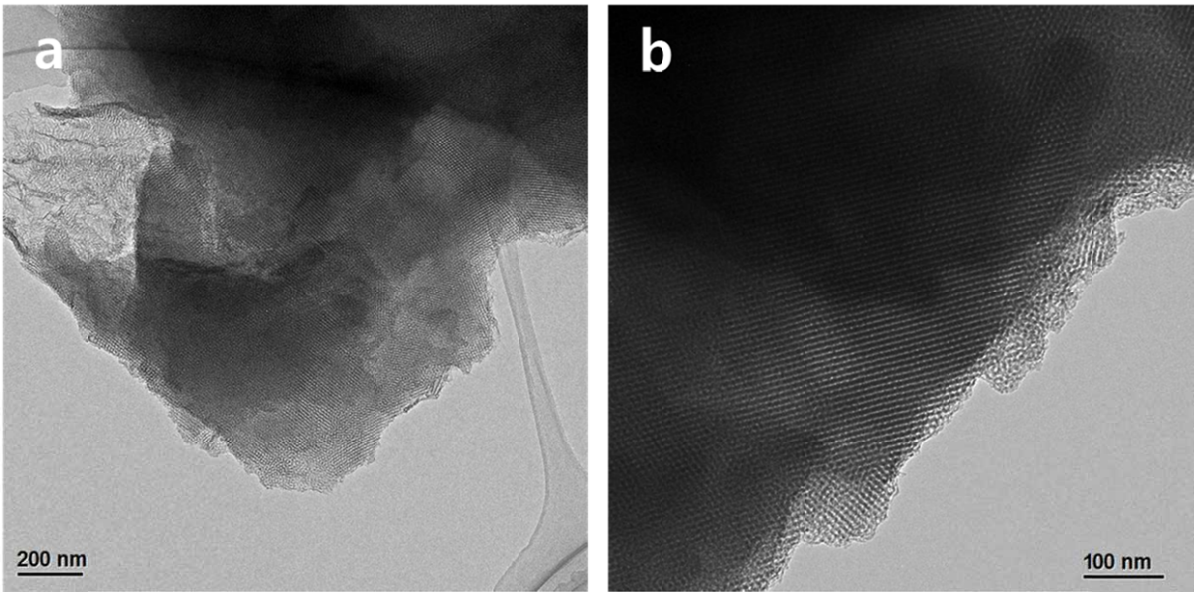

Figure S2. Typical TEM image of (a) KIT-6/GO nanocomposite and (b) HRTEM image of KIT-6/GO nanocomposite.

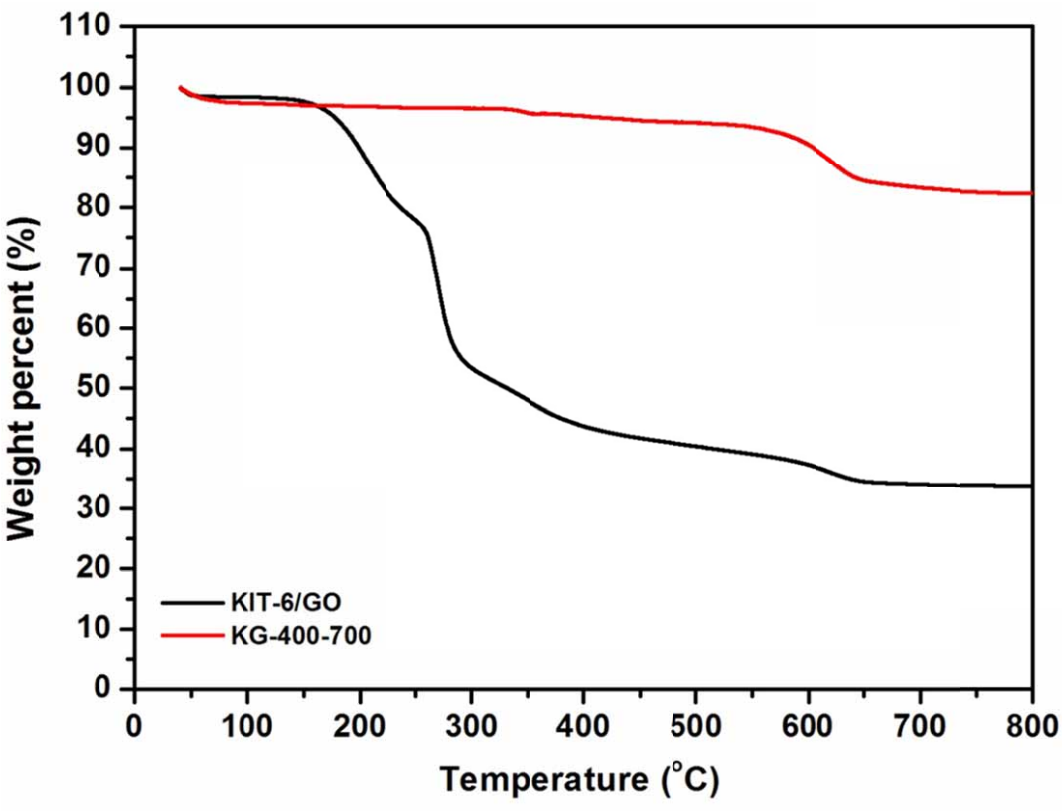

Figure S3. TGA plots of KIT-6/GO and KG-400-700 obtained at 10 $mLmin^{-1}$ air flow and a heating rate of 10 $°Cmin^{-1}$.

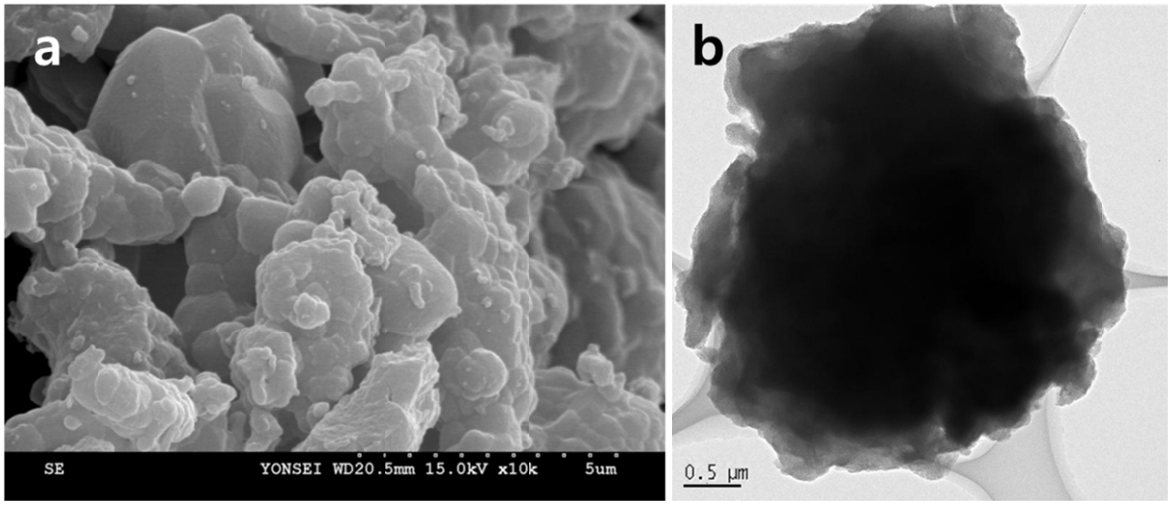

Figure S4. (a) SEM and (b) TEM image of KIT-6.

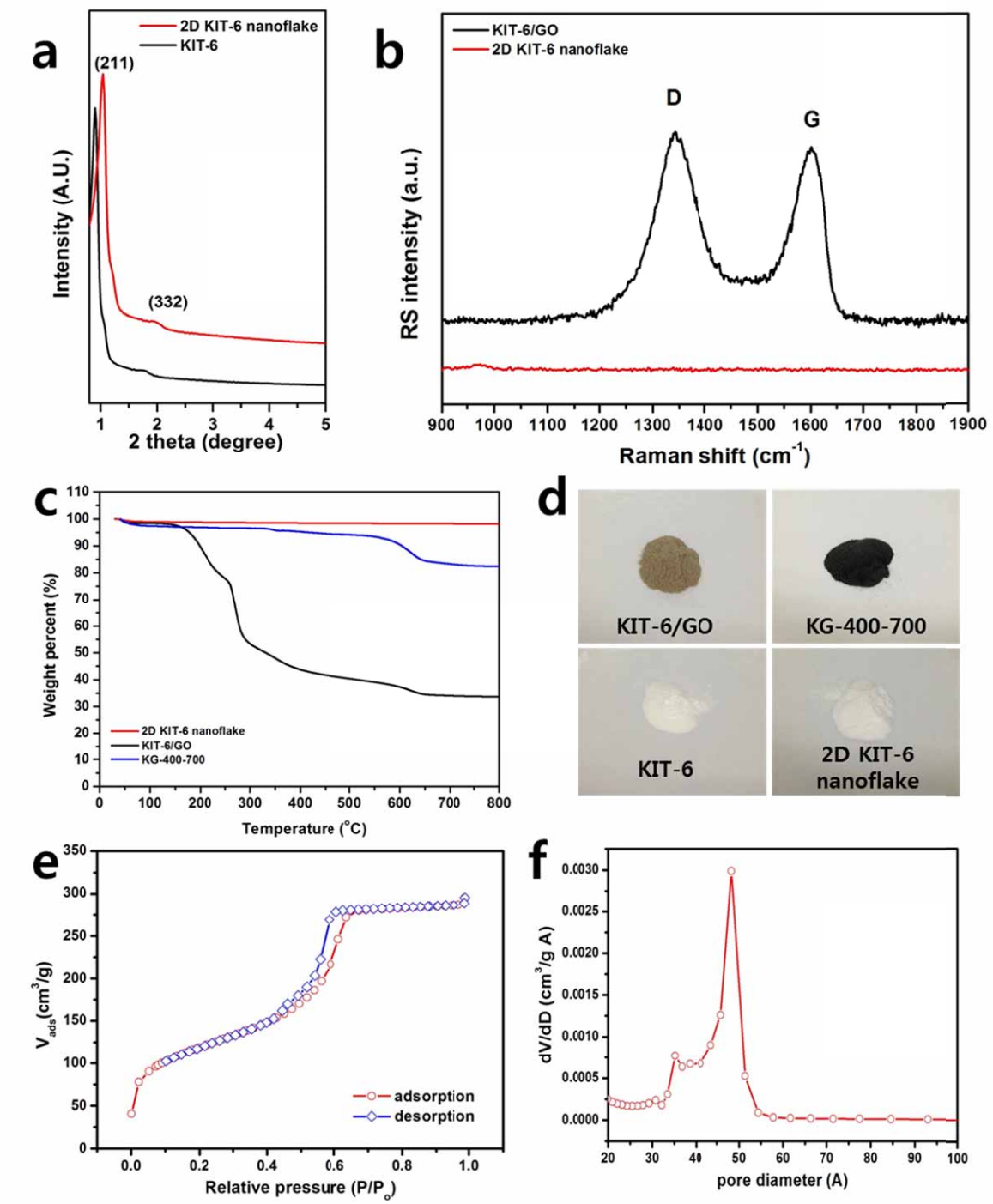

Figure S5. (a) Low angle XRD pattern of KIT-6 and 2D KIT-6 nanoflake (b) Raman spectra of KIT-6/GO and 2D KIT-6 (c) TGA plots of KIT-6/GO, KG-400700 and 2D KIT-6 nanoflake (d) photographic image of KIT-6/GO, KG-400-700, KIT-6, and 2D KIT-6 nanoflake powder (e) N_2 adsorption isotherms and (f) pore size distribution of 2D KIT-6 nanoflake.

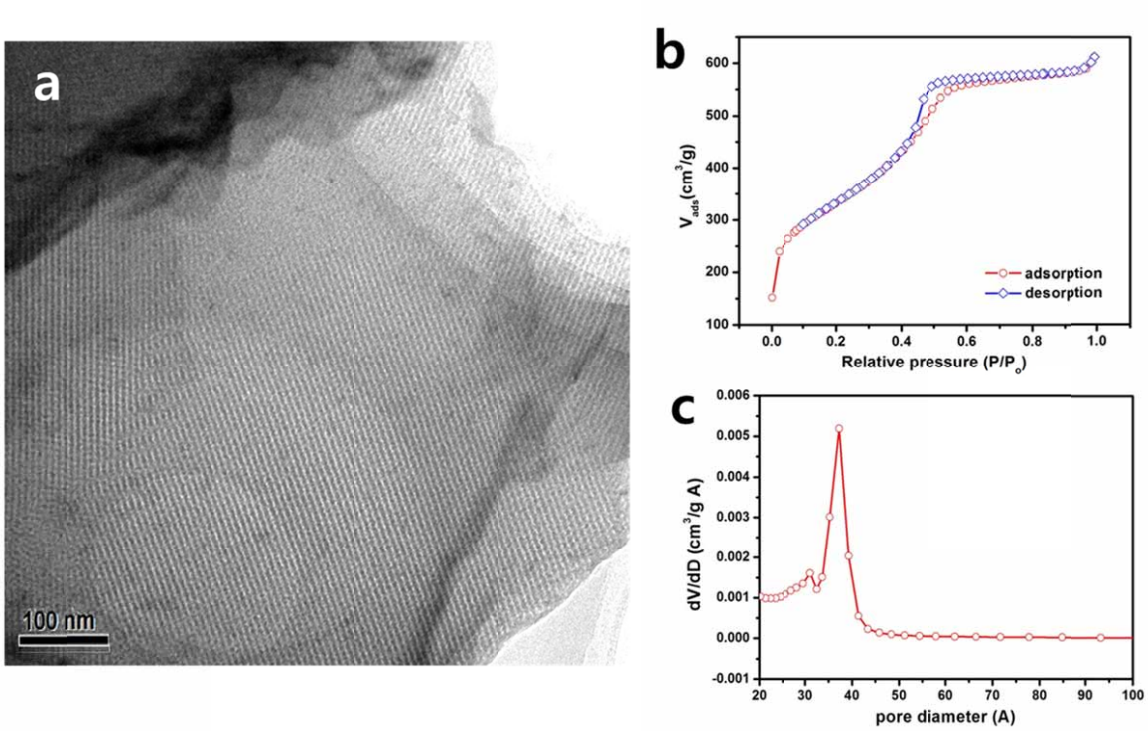

Figure S6. (a) Typical TEM image of mesoporous carbon/graphene composite material (b) N_2 adsorption isotherms and (c) pore size distribution of mesoporous carbon/graphene composite material.

KG-400-700 was employed as a template to replicate a highly ordered mesoporous carbon/graphene nanocomposite using sucrose as a carbon source. The composite thus obtained displayed 2D nanoflake morphology and highly ordered mesoporous structure similar to that of KG-400-700. The N_2 adsorption isotherm shows a BET surface area of 1178.5 m^2g^{-1} and pore volume of 0.94 cm^3g^{-1} with a narrow pore-size distribution centered at 3.1 and 3.7 nm.

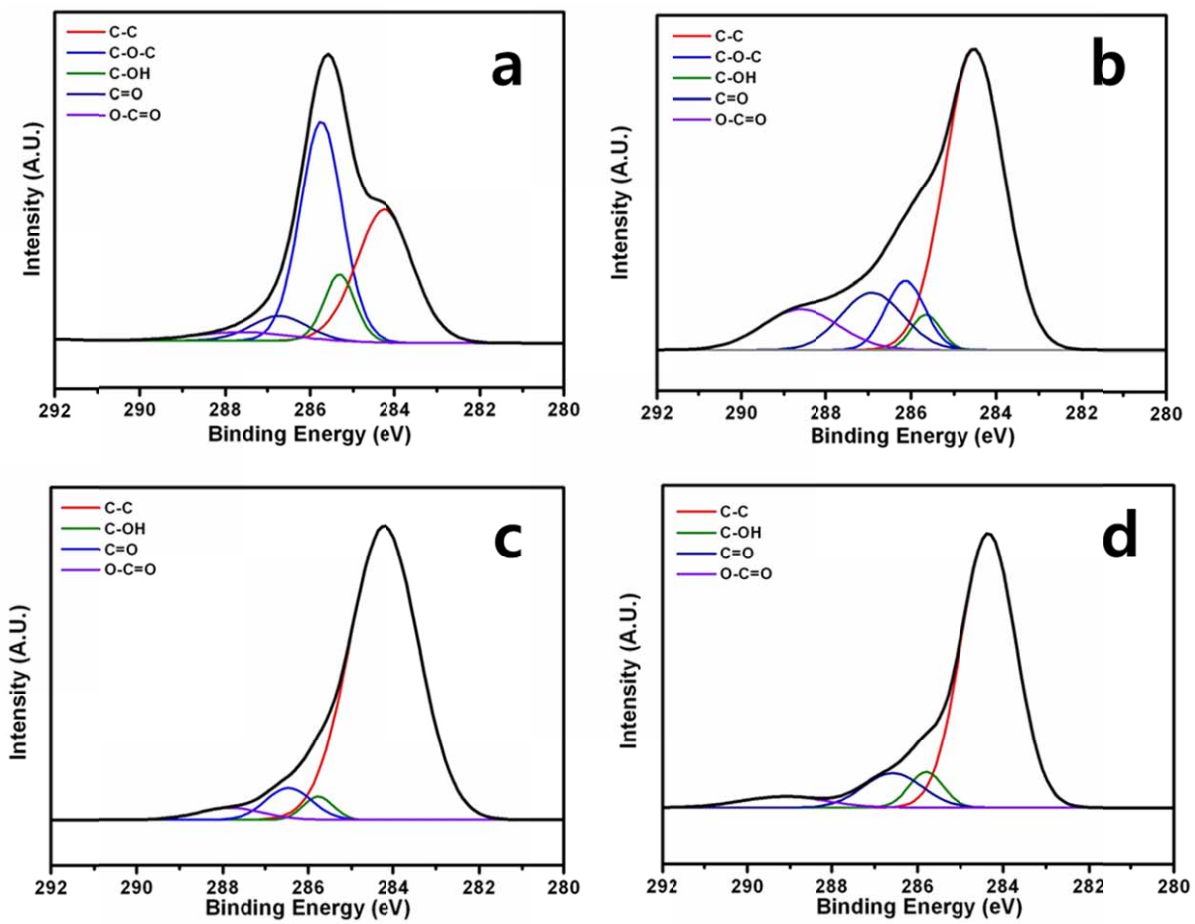

Figure S7. XPS C1s spectra of (a) KIT-6/GO, (b) KG-400, (c) KG-400-700 and (d) KG-700.