

Accessory Publication

Covalently Cross-Linked Ferrocenyl PAMAMOS Dendrimer Networks

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Materials and Instruments

Solvents were purified by distillation from appropriate drying agents under an atmosphere of dry argon. PAMAMOS-TMOS G0, (10 wt% isopropyl alcohol solution) was purchased from Aldrich and used as received. (Triethoxysilylpropyl)amidoferrocene **1** was prepared by the same procedure as that described previously for a similar derivative (C. M. Casado, M. Morán, J. Losada, I. Cuadrado, *Inorg. Chem.* **1995**, *34*, 1668.).

Infrared spectra were recorded on a JASCO FT/IR-410 spectrometer. Solid state ²⁹Si cross polarization magic angle spinning nuclear magnetic resonance (²⁹Si CP MAS NMR) spectra were obtained on a Bruker AV-400 WB spectrometer. The spectrometer frequency was set to 79.49 MHz. The samples were packed into a rotor spinning at 10 kHz and the spectra were recorded using 6 ms of contact time and a pulse wide of 3 μs. Glass temperatures (T_g) of the networks were measured by differential scanning calorimetry (DSC) using a DSC Q-100 unit. Measurements were performed between -90 and 250 °C with about 1 mg samples at a heating rate of 10 °C/min. The TGA thermograms were obtained from a TGA TA INSTRUMENTS Q-500 analyzer. The temperature program was

from 25 to 500 °C, with a temperature rate of 10 °C/min under a nitrogen flow. Scanning electron microscopy (SEM) was carried out with a Hitachi S-3000N instrument. Samples were coated with gold by a Sputter Coater SC502 instrument. Cyclic voltammetry and differential pulse voltammetry experiments were recorded on a BAS CV-50W potentiostat. Voltammetric measurements in non aqueous solution were performed in CH₂Cl₂ (spectrograde) freshly distilled from phosphorus pentoxide, using TBAH (Fluka) as supporting electrolyte. In aqueous solution, LiClO₄ (Aldrich) or LiCl (Fluka) were used as supporting electrolyte and the concentration was typically 0.1 M. All CV experiments were performed using an Indium Tin Oxide (ITO) working electrode. All potentials are referenced to the SCE and a coiled platinum wire was used as a counter electrode. DPV was done with a scan rate of 20 mV s⁻¹ with pulse amplitude of 50 mV during 50 ms at intervals of 2 s.

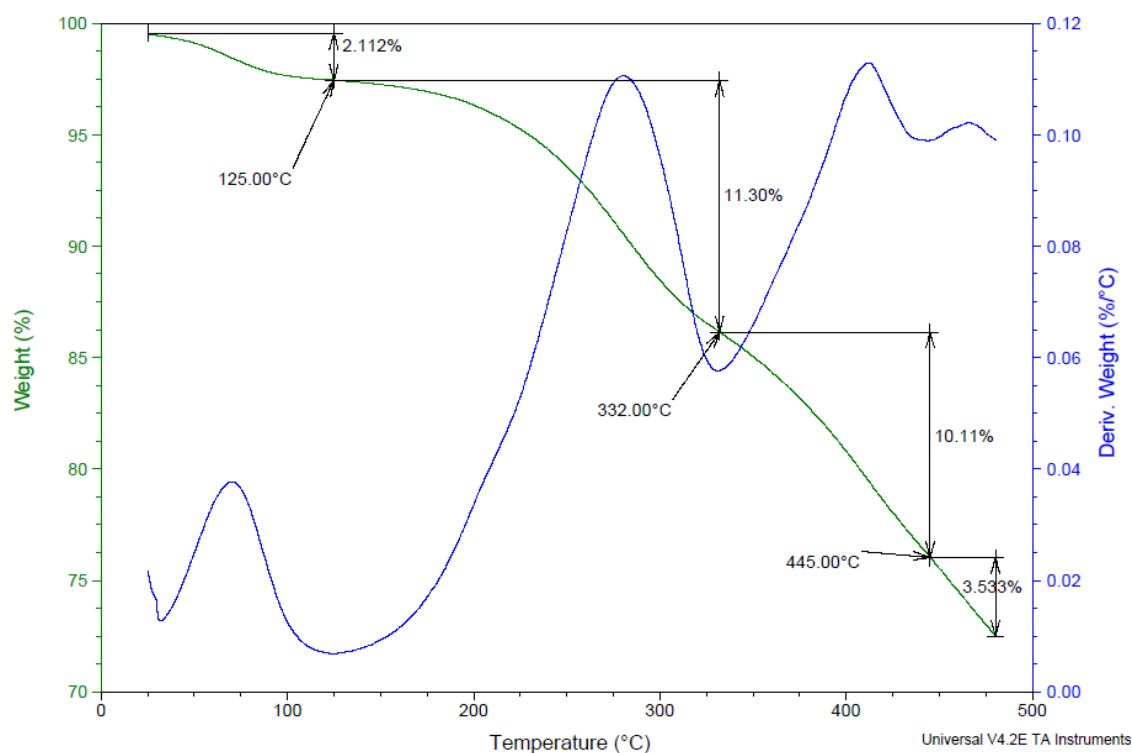


Fig. 1. TGA thermograms of a finely grinded film of Fc-PAMAMOS-G0-dend networks.

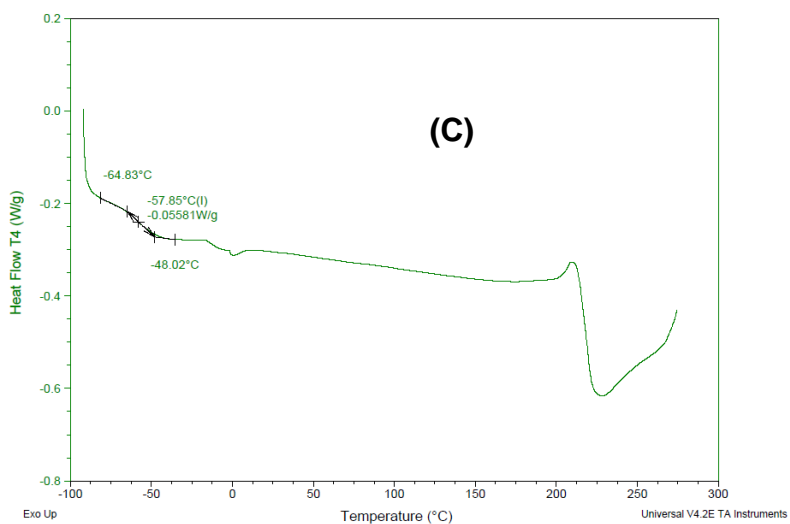
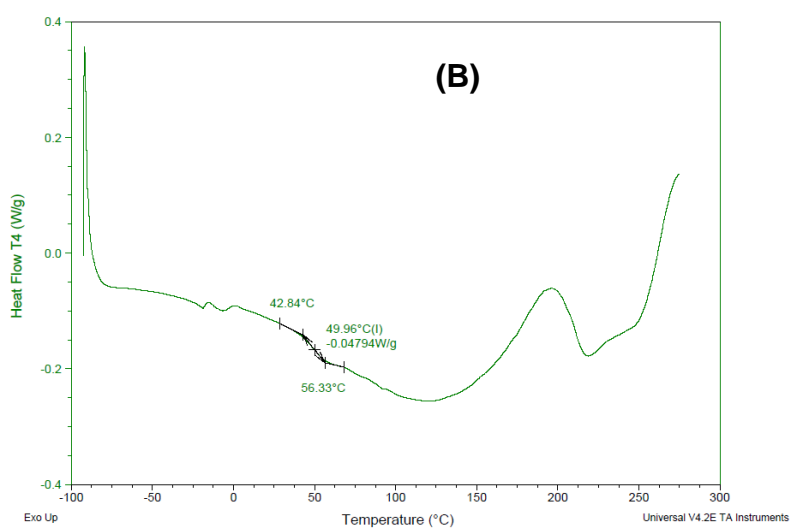
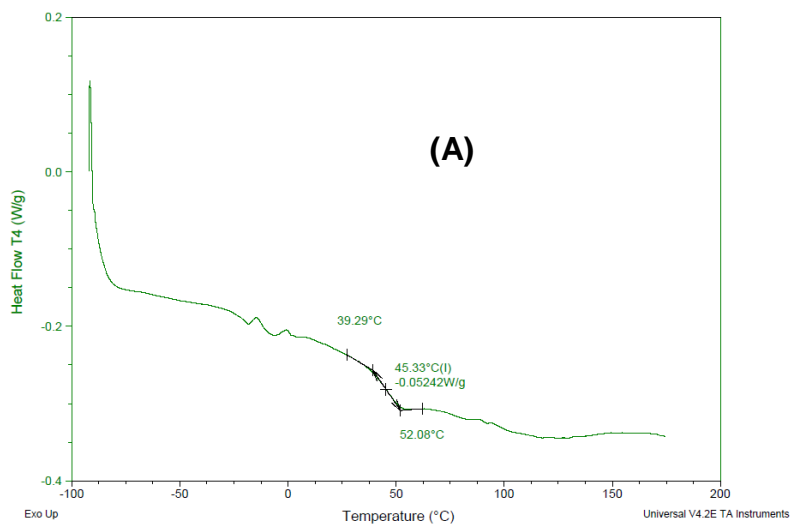


Fig. II. DSC of (A) Fc-PAMAMOS-G0-dend network cured at 100 °C; (B) Fc-PAMAMOS-G0-dend network cured at 120 °C; (C) PAMAMOS-G0-dend.

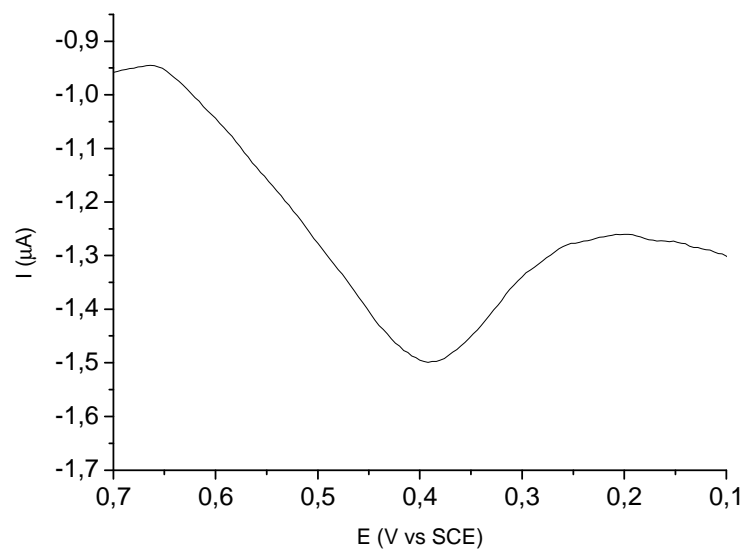


Fig. III. Differential pulse voltammogram in H₂O/0.1 M LiClO₄, for an ITO electrode modified with a film of Fc-PAMAMOS-G0-dend networks.