

Electronic Supplementary Information for

**Polymer/Clay Nanocomposite Gel via Chlorinated Paraffin
Solvent Initiated Photopolymerization with
Electrorheological Performance**

Yuemei Ye, and Qigang Wang*

Department of Chemistry, and Advanced Research Institute, Tongji University, Shanghai 200092,
P. R. China

1. Materials and reagents

Chlorinated paraffins52 (CP-52) was purchased from Shanghai Wenhua chemical paint Co., Ltd. Clay-NS (Laponite RD) was purchased from Rockwood Ltd.. N, N-dimethylacrylamide (DMAA) was purchased from Sinopharm Chemical Reagent Co., Ltd (Shanghai, China) in analytical grade. All materials and reagents were used with no further purification.

2. Instruments and characterizations

2.1 Electron Paramagnetic Resonance (EPR) measurements: The EPR results were obtained on an EPR Spectrometer (A300, Bruker) at 9.873 GHz. As for the Cl radical signals, α -phenyl-tert-butyl-nitron (PBN) is a kind of catcher of chlorine radical for the EPR measurement, which was added to CP-52, then the mixture was put under UV irradiation (average 20.0 mw/cm² intensity at 365nm), and the spectrum was recorded after 20s. For the carbon radical, 80% CP-52 and 20% DMAA were mixed together, then the mixture was placed in the EPR Spectrometer and irradiated under in-situ UV light, the spectrum was recorded after 2 min.

2.2 NMR measurement: All the proton NMR spectra were obtained using a Bruker ARX-400 (400 MHz) spectrometer. Precursor solution contains CP-52 (780 mg), DMAA (200 mg) and Clay-NS (20mg), dioxane was added as internal standard. The mole concentration ratio of DMAA and dioxane was 32 to 1. The mixture was irradiated under UV lamp (fixed 22.4 mw cm⁻² intensity at 365 nm) to gelation for 0 min, 5 min, 10 min, 15 min, 30 min, 60 min, 120 min, 180 min and then fully dissolved in D₂O and stand for more than 48h, then the suspension solutions were centrifuged and the supernatants were used for NMR measurements.

2.3 Mechanical and morphological measurement: The compressive measurement was finished on a FR-108B tensile-compressive tester which produced by Farui Co.. The thickness of the sample was 13mm and the diameter of the sample was 18mm. The measurements were operated at the cross-head speed of 1mm/min and compressed for 95%.

The rheological measurements were tested on a Haake Rheotest 6000 (Thermo Scientific, Karlsruhe, Germany) rheometer with a parallel plate geometry, all the gap size in parallel plate geometry used for electrorheological measurements were 0.4mm. The rheological measurements of gel under electrical field were measured with a special 35 inert rotor. The clay/chlorinated paraffin nanocomposite gel were tested at a strain of 0.1% and a frequency of 1 rad·s⁻¹, and applied with 2kV/mm electrical field from 78s to 600s. The rheological measurements were all operated with a parallel plate geometry 0.4 mm gap. The recovery in response to applied shear forces was investigated by continuous step strain sweep test with alternate small oscillation force ($\gamma = 1.35\%$) and large one ($\gamma = 267\%$) expressed in terms (duration in parentheses): 1.35% (56 s) → 267% (56 s) → 1.35% (56 s) → 267% (56 s) → 1.35% (56 s) → 267% (56s) → 1.35% (56s) → 267% (56s).

2.4 Scanning electron microscope (SEM) measurement: Scanning electron microscope (SEM) images were obtained on Hitachi S-4800 with 1 kV accelerating voltages. The samples for analysis were cut into cubes of about 2×2×2 mm³ from the inner parts of material. Then the samples were rapidly quenched into liquid nitrogen, and dried by critical point drying (Critical Point Dryer CPD, K850, Quorum), sputter-coated with a thin layer of gold (about 5 nm) before scanning.

3. Figures

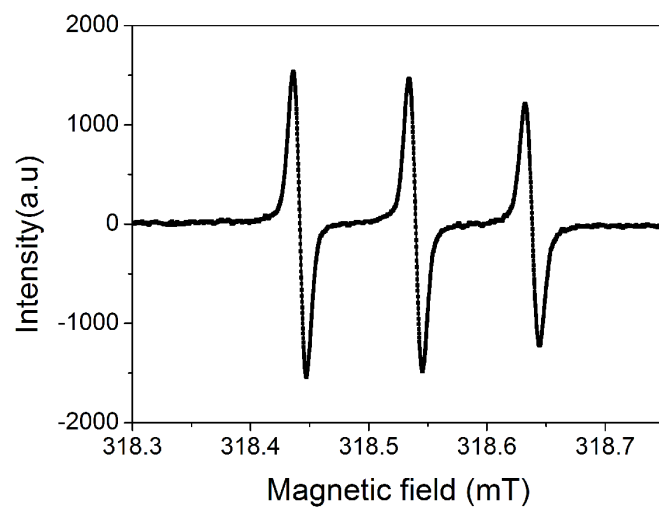


Fig. S1 EPR spectra of DMAA with CP-52 under 2 min UV irradiation.

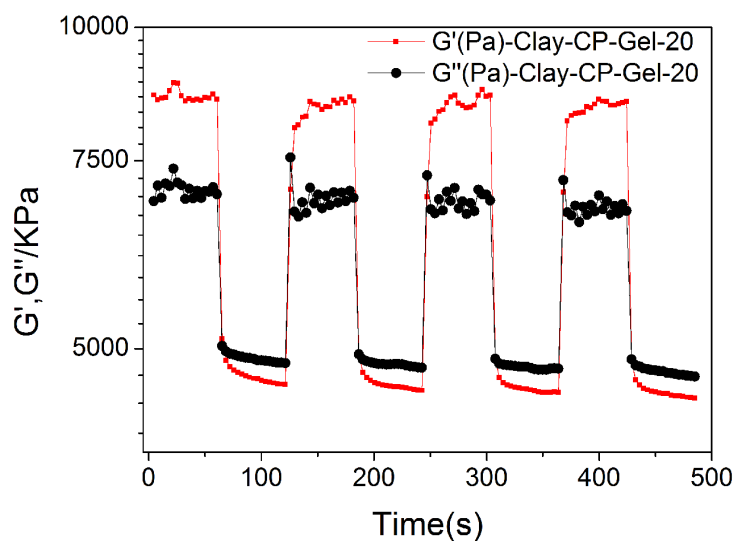


Fig. S2 The self-recovering nature of Clay-CP-Gel-20 (the gel consists of 2% clay and 20% DMAA in CP-52) by continuous step strain sweep test with alternate small oscillation force ($\gamma = 1.35\%$) and large one ($\gamma = 267\%$).

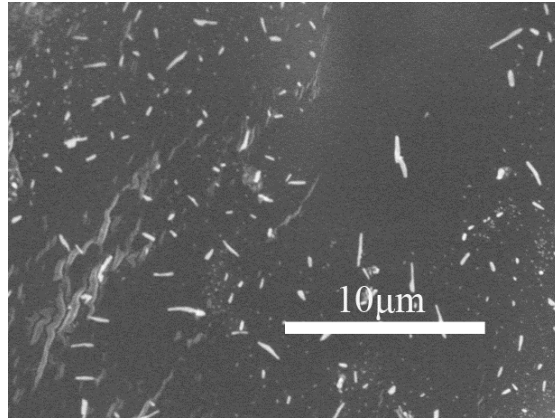


Fig. S3 Scanning electron microscopy image of the aggregated clay in Clay-CP-Gel-11.

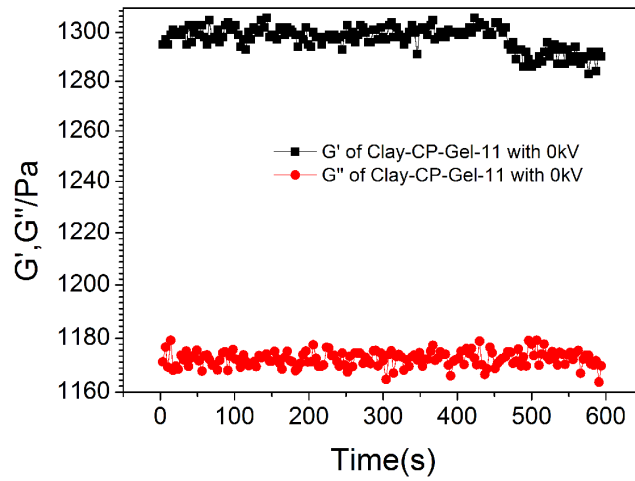


Fig. S4 The process of Clay-CP-Gel-11's elastic modulus and storage modulus change at a strain of 0.1% and a frequency of 1 rad.s⁻¹ under 0kV/mm electrical field.