Electronic Supplementary Material (ESI) for Nanoscale:

Preparation of Graphite Intercalation Compounds Containing Oligo and Polyethers

Hanyang Zhang [a] and Michael M. Lerner [a]

[a]*Department of Chemistry, Oregon State University, Corvallis, OR 97331, United States*

Section 1. Domain Size

The domain size along the stacking direction (*c*-axis) for each product was determined using the modified Scherer relation[38] for the first 3-4 low angle (00l) reflections observed in PXRD. The (003) reflection generally has a shoulder peak due to excess crystalline polymer and was not used in the calculations. Table S1 shows the data for one sample.

Table S1. PXRD peak data for Na-PEGDME1k-GIC*

β (rad)	θ (rad)	ln(1/cosθ)	lnβ
0.00384	0.06631	0.00220	-5.5624
0.00368	0.13266	0.00883	-5.6041
0.00394	0.26704	0.03609	-5.5355
0.00429	0.33632	0.05765	-5.4507

* β is full width at half maximum (FWHM), θ is the peak position. Linear regression for lnβ *vs.* ln(1/cosθ) gave an intercept of -5.5982.

The domain size was then determined using Equation (S1):

$$\exp(\ln\beta)_0 = 0.89\lambda / L \qquad \text{(S1)}$$

where $(\ln\beta)_0$ is the intercept of lnβ *vs.* ln(1/cosθ) and λ for Cu Kα radiation is 0.15406 nm.

Section 2. Additional Raman spectra info

All GIC products exhibit very low Raman D/G band intensity ratios (I_D/I_G). Fig. S1 shows spectra for selected products and synthetic graphite. The native synthetic graphite showed $I_D/I_G = 0.26$, the Na-PEG6k-GIC product obtained using this graphite showed a weaker, broader D-band with $I_D/I_G = 0.08$. No discernible D-band intensity

was observed found for Na-PEGDME1k-GIC spectra obtained from SP-1 graphite. The D-band peak at ≈1,350 cm^{-1} indicates sp^3 carbon whereas the G-band peak at ≈1,580 cm^{-1} indicates sp^2 carbon.[43] These results confirm that the graphene sheets in the GIC products retain a high degree of graphitization and very low defect concentration, in contrast for example to nanocomposites obtained with reduced graphene oxide. The disappearance of the Raman D band has been reported previously for stage 1 or 2 donor-type GICs. [44]

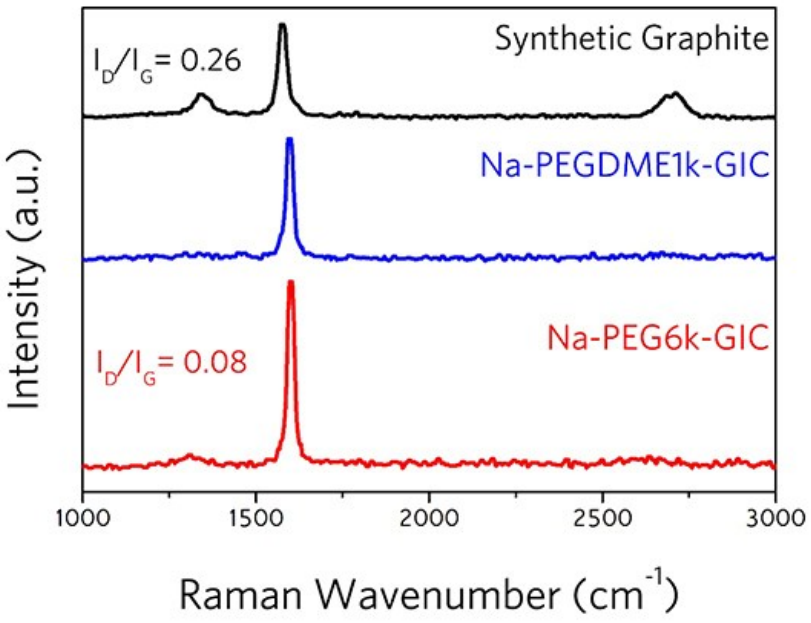

Fig. S1 Raman spectra of synthetic graphite and GIC products. D/G band intensity ratios (I_D/I_G) are calculated for synthetic graphite and Na-PEG6k-GIC sample; the Na-PEGDME1k-GIC shows no discernible D-band.

Section 3. Compositional Calculation

The dTGA plots of reaction products show two overlapping peaks. The lower temperature peak is attributed to polymer in the GIC phase, the mass percentage of intercalated polymer is equal to the integrated area of the low-temperature peak in the dTGA, and the higher temperature peak is from excess unreacted polymer. Fig. S2 shows selected peak fitting for Na-PEGDME1k-GIC and Na-PEG6k-GIC.

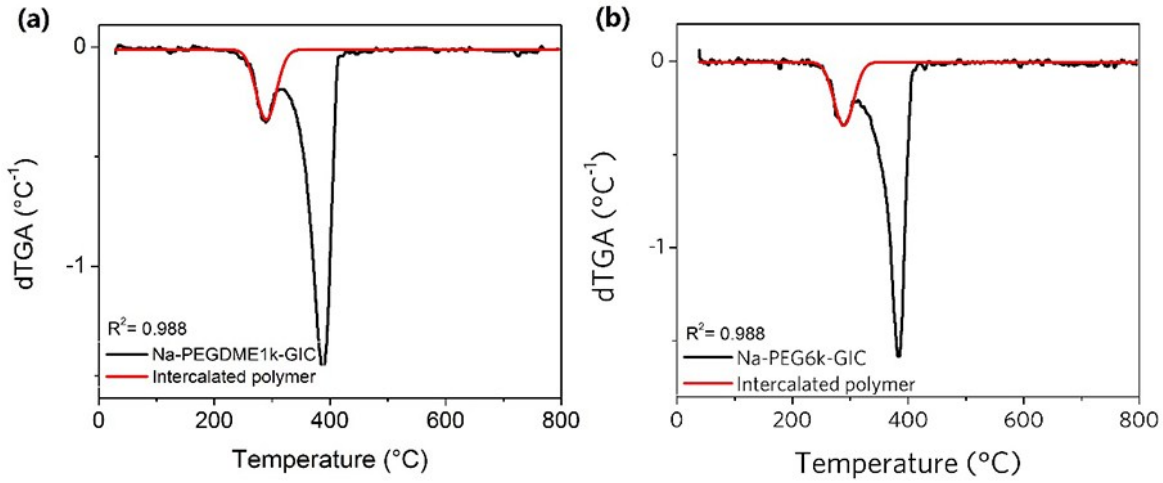

Fig. S2 dTGA curves with fit peaks for PEG intercalate,
(a) Na-PEGDME1k-GIC (b) Na-PEG6k-GIC

The graphite mass % in the products was obtained from the mass residual at 650 °C, under N_2 (Ar for Li products) flow.

Section 4. Packing Fractions

The packing fractions, *i.e.*, the fraction of occupied volume within the expanded galleries,[29] were derived by applying equation (S2):

$$\text{packing fraction} = (V_M + y \cdot 0.0493\ \text{nm}^3) / (x)\ (0.0261\ \text{nm}^2)\ (\Delta d) \quad \text{(S2)}$$

where the cationic volumes $V_{Li} = 0.0016$, $V_{Na} = 0.0060$, and $V_K = 0.0110$ nm^3, the van der Waals volume of one –CH_2CH_2O– repeat unit is 0.0493 nm^3,[45] x is the number of graphitic carbons per negative charge, and y is the molar ratio of –CH_2CH_2O– units to alkali metal cation.

References

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