New Journal of Chemistry

Electronic Supplementary Information

A stable and easily regenerable solid amine adsorbent derived from polyethylenimine-impregnated dialdehyde-cellulose/graphene-oxide composite

Xianyu Qiu [a], Xiaoqiong Wang [a], Shuixia Chen [*ab]

[a] *PCFM Lab, School of Chemistry, Sun Yat-Sen University, Guangzhou 510275, PR China*

[b] *Materials Science Institute, Sun Yat-Sen University, Guangzhou 510275, PR China*

Titration method to quantify the aldehyde content

The aldehyde content on the surface of DAC was estimated by the titration method as reported in the literature.[1] After freeze drying, 0.1 g of pieced DAC aerogel was weighed and placed into 0.25 M Hydroxylamine hydrochloride ($NH_2OH \cdot HCl$) solution (25 mL), which was adjusted to pH 4.5 with HCl. All the mixture was placed in dark, shaken in the thermostatic water bath at ambient temperature for 48 h. The conversion of aldehyde to oxime was determined by the consumption of 0.1 M NaOH solution, each sample was quantified twice.

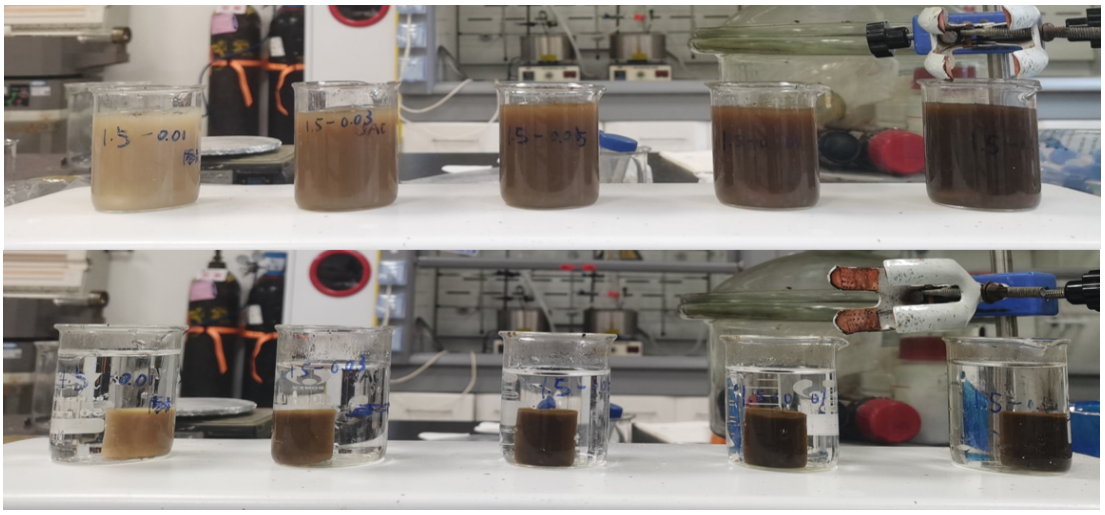

Fig. S1 Photo images of the self-assembly of DAC-GO with different concentration of GO (0.01 wt%, 0.03 wt%, 0.05 wt%, 0.07 wt%, 0.1 wt%) before and after periodate oxidation.

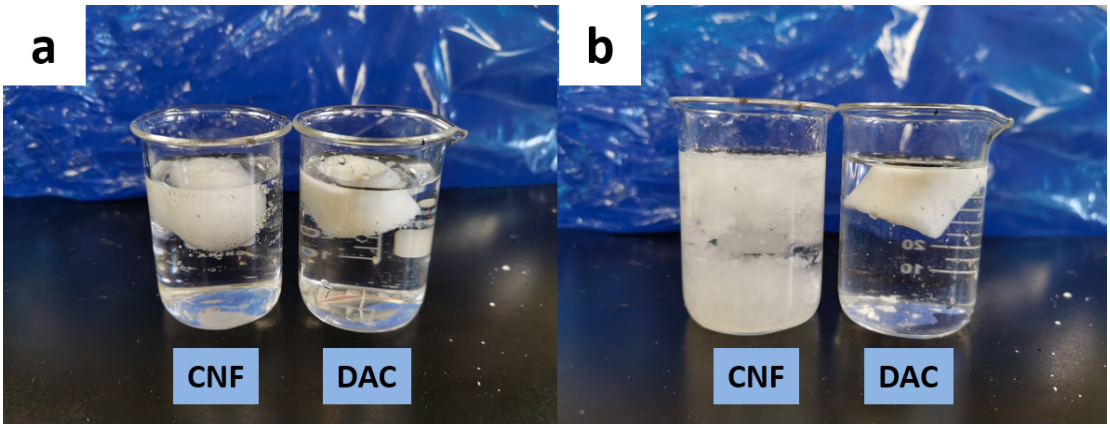

Fig. S2 Photo images of the CNF aerogel and DAC aerogel when hydrated again after freeze-drying: (a) hydrated samples at the beginning; (b) hydrated samples after shaking.

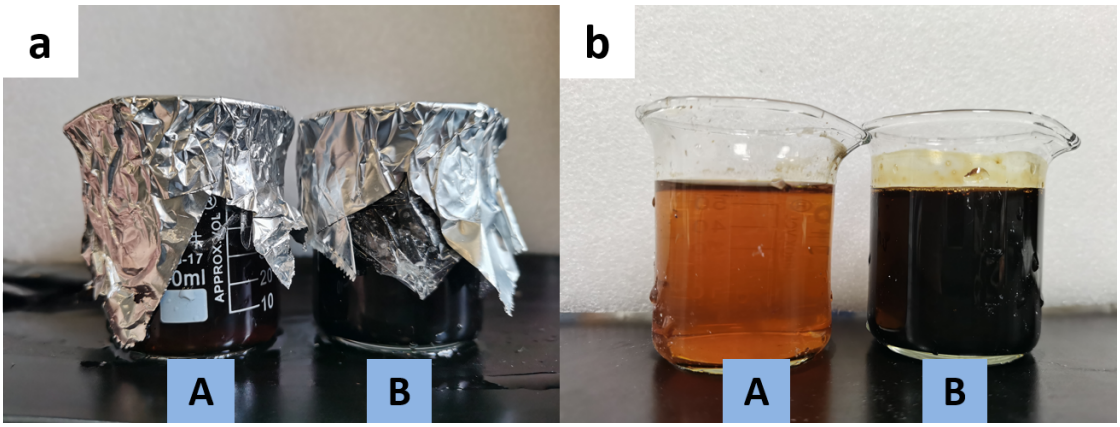

Fig. S3 Photo images of water solution with different concentration of GO before (a) and after (b) periodate oxidation: A: 0.05 wt% GO; B: 0.6 wt% GO.

Mechanical property of DAC-0.05GO gel in humid conditions

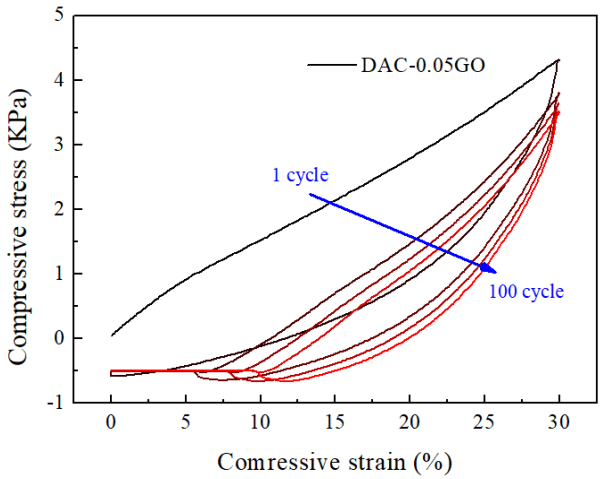

Fig. S4 Stress–strain curves during 100 cycles of compression-releasing at a constant strain of 30%, showing the stress–strain curves of selected cycles (0th, 30th, 60th, 100th).

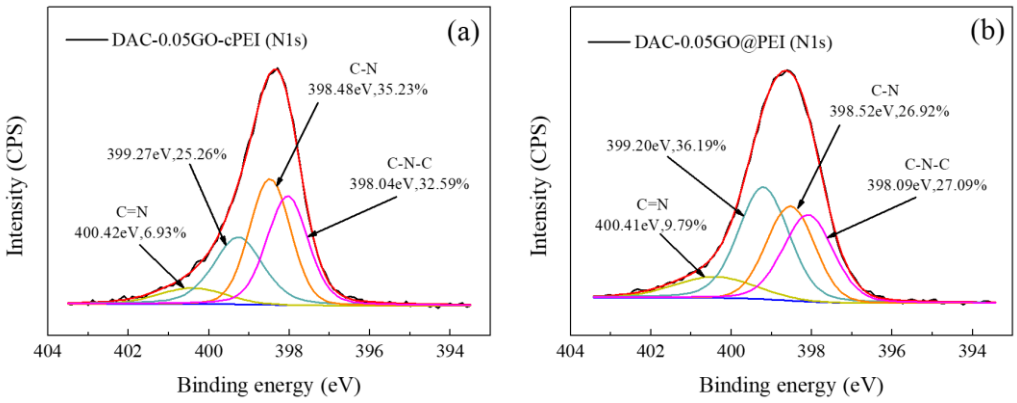

Fig. S5 XPS spectra of DAC-0.05GO after amination: (a) N1s spectrum of DAC-0.05GO-cPEI; (b) N1s spectrum of DAC-0.05GO@PEI.

Structural characterization

Nitrogen adsorption–desorption isotherms, characterized at 77 K in the range of

relative pressure from 10^{-6} to 1 by using adsorption instrument (JW-BK200C, JEGB SCI&TECH., China), were used to determine the surface area and pore size distribution of the samples. V_{Total} was calculated based on the nitrogen amount adsorbed at P/P_0 = 0.95. Before measuring, non-aminated aerogels were degassed under a vacuum at 120 ℃ for 6 h, aminated aerogels were degassed under a vacuum at 100 ℃ for 6 h. Specific surface area was calculated by using the Brunauer-Emmett-Teller (BET) method from the isotherm in the relative pressure range between 0.05 and 0.15. Pore parameters were calculated by using density functional theory (DFT) method.

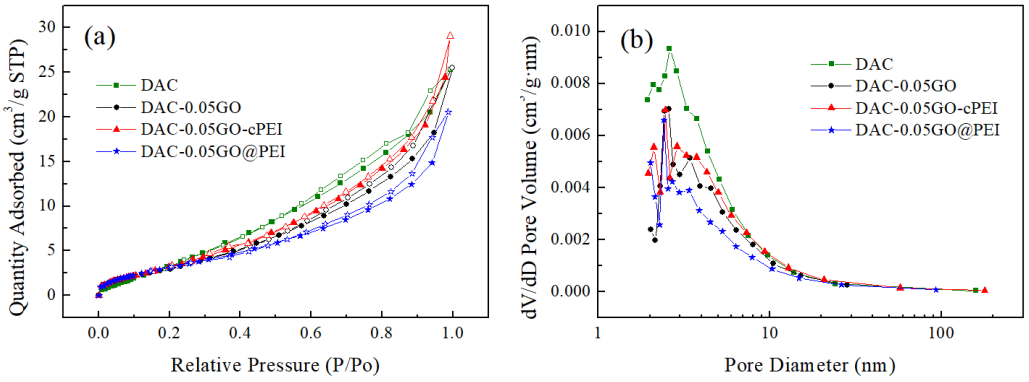

Fig. S6. The N_2 adsorption–desorption isotherms (a) and pore size distributions (b) of DAC, DAC-0.05GO, DAC-0.05GO-cPEI and DAC-0.05GO@PEI.

Table S1 Summary of pore parameters of the gels.

Samples	S_{BET} (m^2/g)	V_{Total} (cm^3/g)	D_{Pore} (nm)
DAC	11.487	0.039	10.625
DAC-0.05GO	10.271	0.039	13.894
DAC-0.05GO-cPEI	11.291	0.045	15.641
DAC-0.05GO@PEI	10.783	0.032	10.687

As it was shown in Fig. S6, all N_2 adsorption–desorption isotherms belonged to Type IV with hysteresis loops (Fig. S6a) and all the samples had similar pore size distributions (Fig. S6b), which showed that these materials possessed mesoporous structure. As shown in Table S1, the specific surface area and total pore volume of all the gels were close, indicating that composite or amination showed little influence on pore characteristic. Therefore, the slight-increased CO_2 adsorption amount on composite gel might be attributed to slight-increased space inside the support, while the increased CO_2 adsorption amount on DAC-0.05GO-cPEI might be attributed to increased effective amino sites.

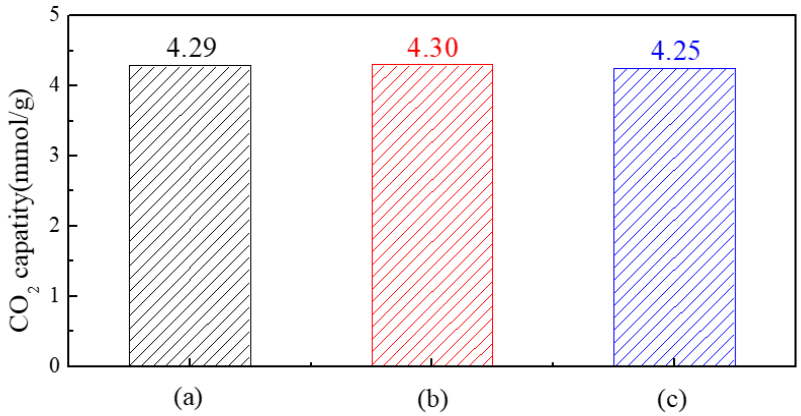

Fig. S7 CO_2 amount adsorbed on DAC-0.05GO-cPEI at 25 ℃ before conducting desorption test at 70 ℃ (a), 80 ℃ (b) and 90 ℃ (c).

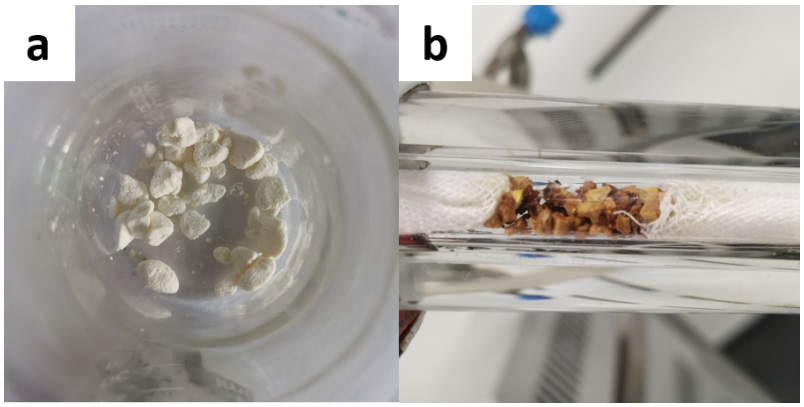

Fig. S8 Photo images of DAC-cPEI: (a) fresh sample; (b) regenerated sample after CO_2 desorption at 70 °C.

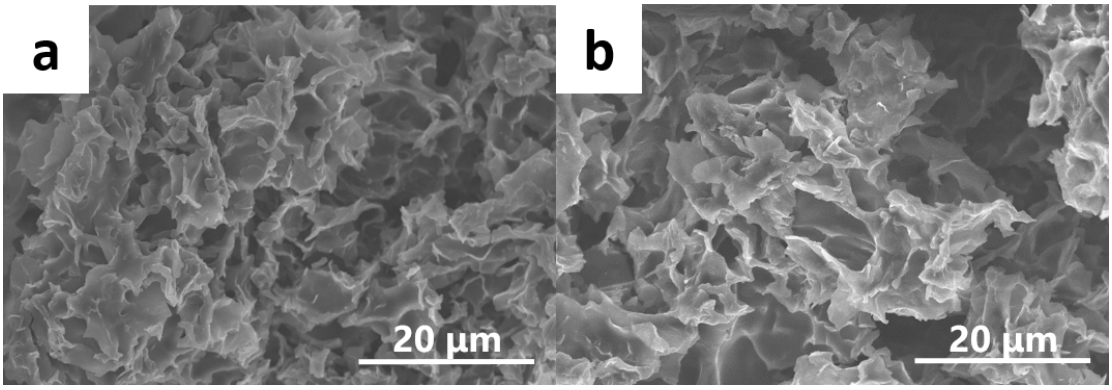

Fig. S9 SEM images of DAC-0.05GO-cPEI: (a) fresh sample; (b) regenerated sample.

References:

1 U. Kim, M. Wada and S. Kuga, 2004, **56**, 7–10.