

Supporting Information

CL-20 hosted in graphene foam as high energy materials with low sensitivity

Zhimin Li,^a Yu Wang,^b Yanqiang Zhang,^{*a} Long Liu,^a and Suojiang Zhang^{*a,b}

^a Beijing Key Laboratory of Ionic Liquids Clean Process, Key Laboratory of Green Process and Engineering, Institute of Process Engineering, Chinese Academy of Sciences, Beijing 100190, P. R. China

^b State Key Laboratory of Multiphase Complex Systems, Institute of Process Engineering, Chinese Academy of Sciences, Beijing 100190, P. R. China

Table of Contents

Section S1. General methods

Section S2. Preparation

Section S3. Thermal analyses

References

Section S1. General methods

Ethyl acetate and chloroform (>99.5%, Beijing Chemical Works) are of analytical grade and used as commercially obtained. Raman spectra were recorded by using a LabRAM HR800 (HORIBA Jobin Yvon, France) microscopic Confocal Raman Spectrometer with an Ar laser at a wavelength of 514 nm. X-ray diffraction (XRD) patterns were obtained by using a SmartLab (Rigaku, Japan) X-ray diffractometer. Field-emission scanning electron microscope (FE-SEM) images and energy dispersive spectroscopy (EDS) data were taken on a SU8020 (Hitachi, Japan) SEM unit. Differential scanning calorimetry (DSC) was conducted by using a Calorimeter (DSC1, Mettler-Toledo, Switzerland). Impact and friction sensitivities were conducted by using a standard BAM Fallhammer BFH-10 and a BAM friction tester FSKM-10 (Czech). Electrostatic spark sensitivity is measured by using the 50 percent probability of ignition energy (E_{50}). The energy is given by the formula $E=0.5CV^2$, where C is the capacitance of the capacitor, V is charge voltage. A series of samples were tested using the up and down method, and the electrostatic discharge sensitivity (E_{50}) was calculated according to the usual Bruceton formula.

Section S2. Preparation

The nickel foam ($10.0 \times 10.0 \times 1.5 \text{ mm}^3$) placed into a quartz tube was first heated to 1000 °C and maintained for 15 min under atmospheric pressure with the flow of H_2/Ar (100/200 sccm). Subsequently, the ethanol was introduced into the quartz tube with mixing gas of H_2/Ar (25/50 sccm) to synthesize the graphene in 20 min. After growth, the sample was rapidly cooled to room temperature with the protection of H_2 and Ar. At last, free-standing graphene foam was obtained by etching away the nickel foam with HCl (3 M) solution at 80 °C.

CL-20 was recrystallized via a solvent-nonsolvent method (ethyl acetate-chloroform). Chloroform (8 mL) was dropwise added into an ethyl acetate (2.5 mL) solution of raw CL-20 (1 g) which was slowly stirred in room temperature. Precipitation was filtered, washed with ethanol, and dried naturally.

As shown in Fig. S1, there are three steps in the preparation procedure of CL-20/GF composite. Firstly, an ethyl acetate (5 mL) solution of CL-20 (49 mg) was placed in a vial (10 mL). Secondly, a piece of GF (1 mg) was immersed into the solution. Finally, the CL-20/GF composite was obtained by slow evaporation of ethyl acetate in room temperature. As almost all the CL-20 was hosted in GF, the content of GF in CL-20/GF composite was calculated as 2 %, which was also confirmed by the weight of the obtained CL-20/GF (50 mg).

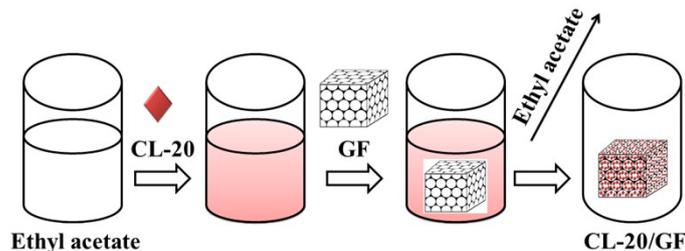


Fig. S1 Preparation procedure of CL-20/GF composite.

Section S3. Thermal decomposition

DSC was tested at different heating rates of 5, 10, 15 and 20 °C·min⁻¹ from 25 to 500 °C. Samples (about 0.5 mg) were placed in the closed aluminum containers, under dry nitrogen atmosphere with a flow rate of 30 mL·min⁻¹. The peak decomposition temperatures (T_p) of CL-20/GF and CL-20 obtained by DSC at various heating rates (β) were listed in Table S1. Based on the data of peak decomposition temperatures versus heating rates, the kinetic parameters were calculated by using the multiple non-isothermal reaction kinetic methods (Kissinger and Ozawa-Doyle).¹⁻³ The equations of the two methods are described in Formula S1. The values of E_a , $\lg A$, linear correlation coefficient (R) and standard mean square deviation (Q) calculated with the linear least-squares method are listed in Table S2.

$$\frac{d \ln(\beta / T_p^2)}{d(1/T_p)} = -\frac{E_a}{R} \quad (\text{Kissinger})$$

$$\lg \beta + \frac{0.4567 E_a}{RT_p} = C \quad (\text{Ozawa - Doyle})$$

Formula S1. Equations of the Kissinger and Ozawa-Doyle methods.

(T is the temperature, β the heating rate, E_a the apparent activation energy, A the pre-exponential factor, R the gas constant, C a constant)

Table S1 Peak decomposition temperatures at different heating rates for CL-20/GF and CL-20.

β (°C min ⁻¹)	T_p (°C)	
	CL-20/GF	CL-20
5	230	250
10	245	259
15	253	265
20	262	268

Table S2 Kinetic parameters of the decomposition process for CL-20/GF and CL-20.

methods	CL-20/GF				CL-20			
	E_a (kJ·mol ⁻¹)	lg (A/s ⁻¹)	R	Q	E_a (kJ·mol ⁻¹)	lg (A/s ⁻¹)	R	Q
Kissinger	90.1	6.91	-0.9975	0.0480	169.1	14.67	-0.9988	0.0340
Ozawa-Doyle	93.9	—	-0.9979	0.0207	169.2	—	-0.9989	0.0148
mean	92.0	6.91			169.2	14.67		

References

1. H. E. Kissinger, *Anal. Chem.*, 1957, **29**, 1702-1706.
2. T. Ozawa, *Bull. Chem. Soc. Jpn.*, 1965, **38**, 1881-1886.
3. R. L. Blaine and H. E. Kissinger, *Thermochim. Acta*, 2012, **540**, 1-6.