

Controllable synthesis of hollow spherical nickel chalcogenide (NiS₂ and NiSe₂) decorated with graphene for efficient supercapacitor electrodes

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Synthesis of graphene oxides. In a typical procedure, pristine graphite powers (3 g, 325mesh) was put into an 80°C solution of concentrated H₂SO₄ (15 ml), K₂S₂O₈ (2.5 g), and P₂O₅(2.5 g). The mixture was kept at 80°C for 6h. Then, the mixture was diluted with 500 ml of deionized (DI) water. Then, the mixture was filtered and washed with water to remove the residual acid. The product was dried at 60°C overnight to obtain pre-oxidized graphite powder. This powder was added into cold (0°C) concentrated H₂SO₄ (115 ml). Then, KMnO₄ (15 g) was slowly added, with the temperature of mixture kept below 20°C. After the addition of 250 ml of water (keep the temperature below 50°C), the mixture was stirred for 2 h, and then an additional 700 ml of water was added. After that, 10 ml of 30% H₂O₂ was added into the mixture, and the solution changed into brilliant yellow, accompanied by the generation of bubbles. The mixture was filtered and washed with diluted HCl aqueous solution (10 wt.%, 1 L) to remove metal ions. The brownish yellow solution was centrifuged at 10,000 rpm, supernatant solution was decanted away, and the resulting material was subjected to multiple washings with water until the pH was 7.

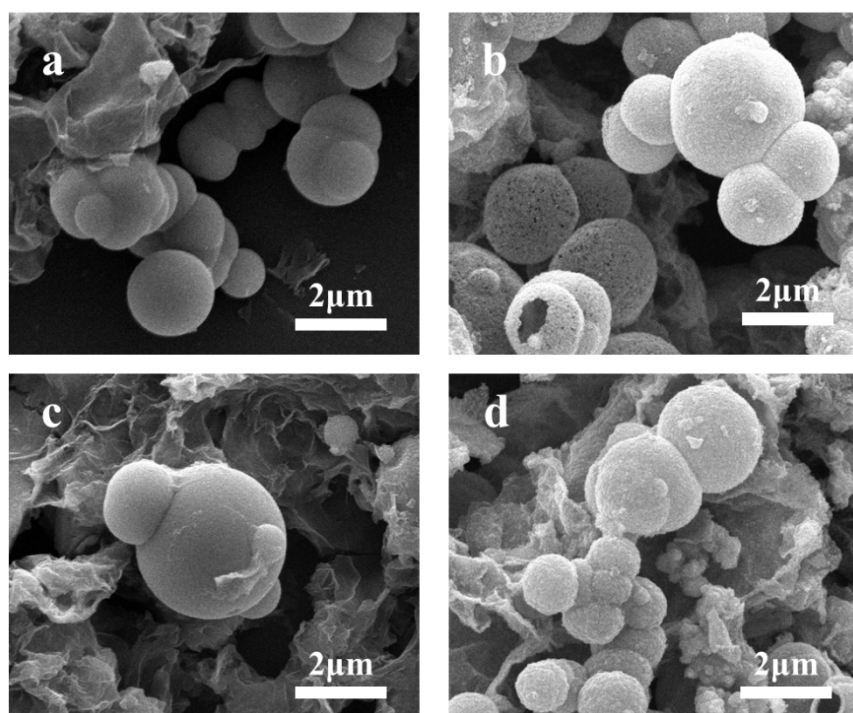


Fig. S1 SEM images of the product during prepare NiS₂ at different time: (a) 10%-NiS₂/GO; (b) 15%-NiS₂/GO; (c) 25%-NiS₂/GO; (d) 30%-NiS₂/GO.

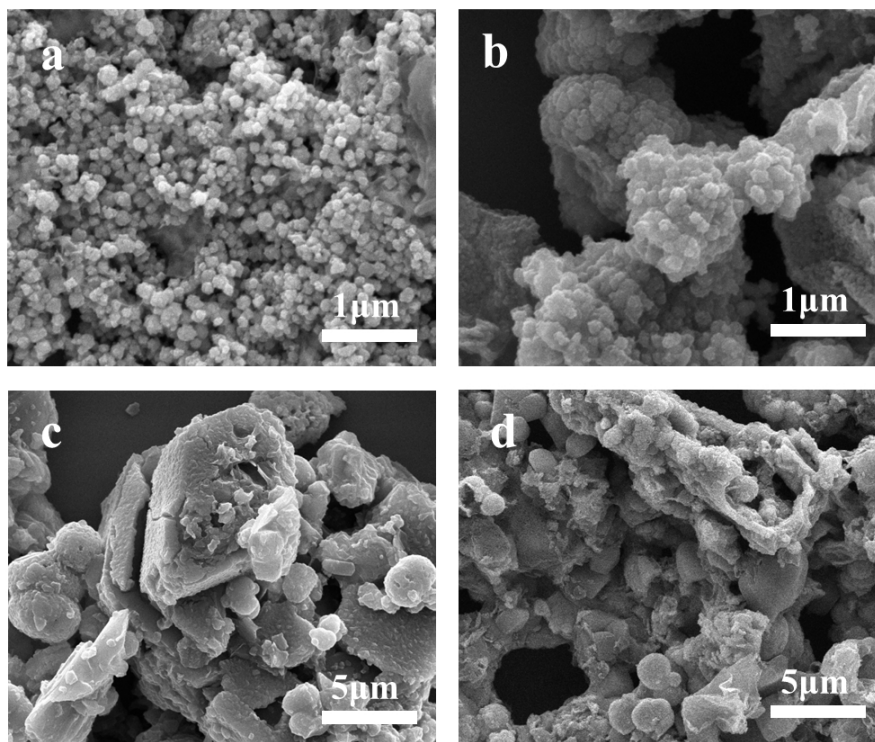


Fig. S2 SEM images of the product during prepare NiSe_2 at different time: (a) 1%- NiSe_2/rGO ; (b) 5%- NiSe_2/rGO ; (c) 7%- NiSe_2/rGO ; (d) 10%- NiSe_2/rGO .

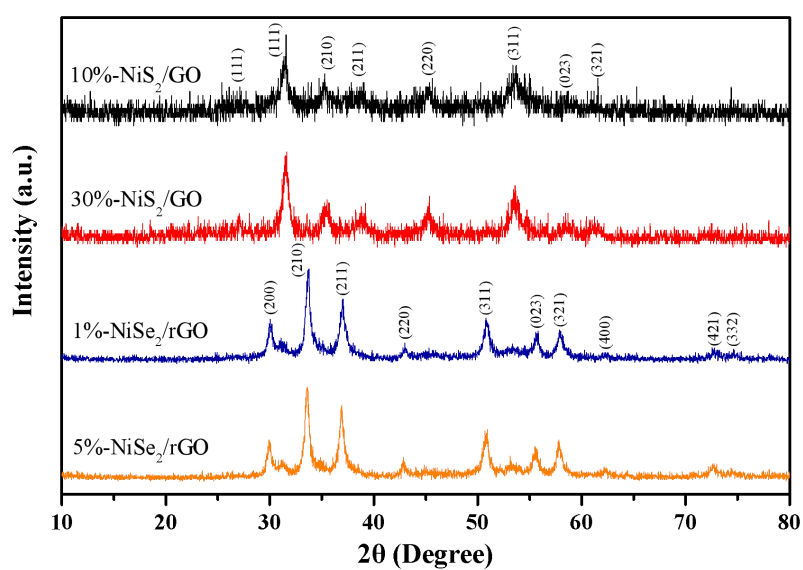


Fig. S3 XRD pattern of the product 10%- NiS_2/GO , 30%- NiS_2/GO , 1%- NiSe_2/rGO and 5%- NiSe_2/rGO .

5%-NiSe₂/rGO.

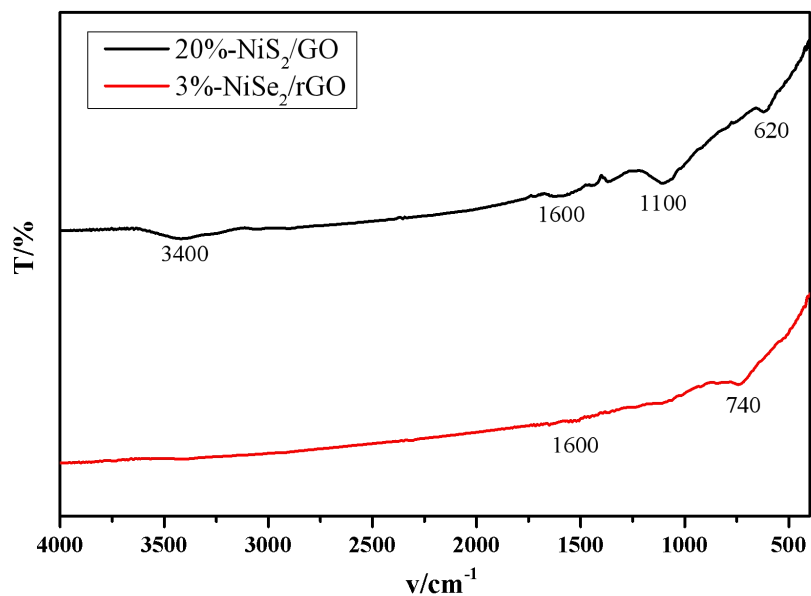


Fig. S4 FT-IR for 20%-NiS₂/GO and 3%-NiSe₂/rGO.

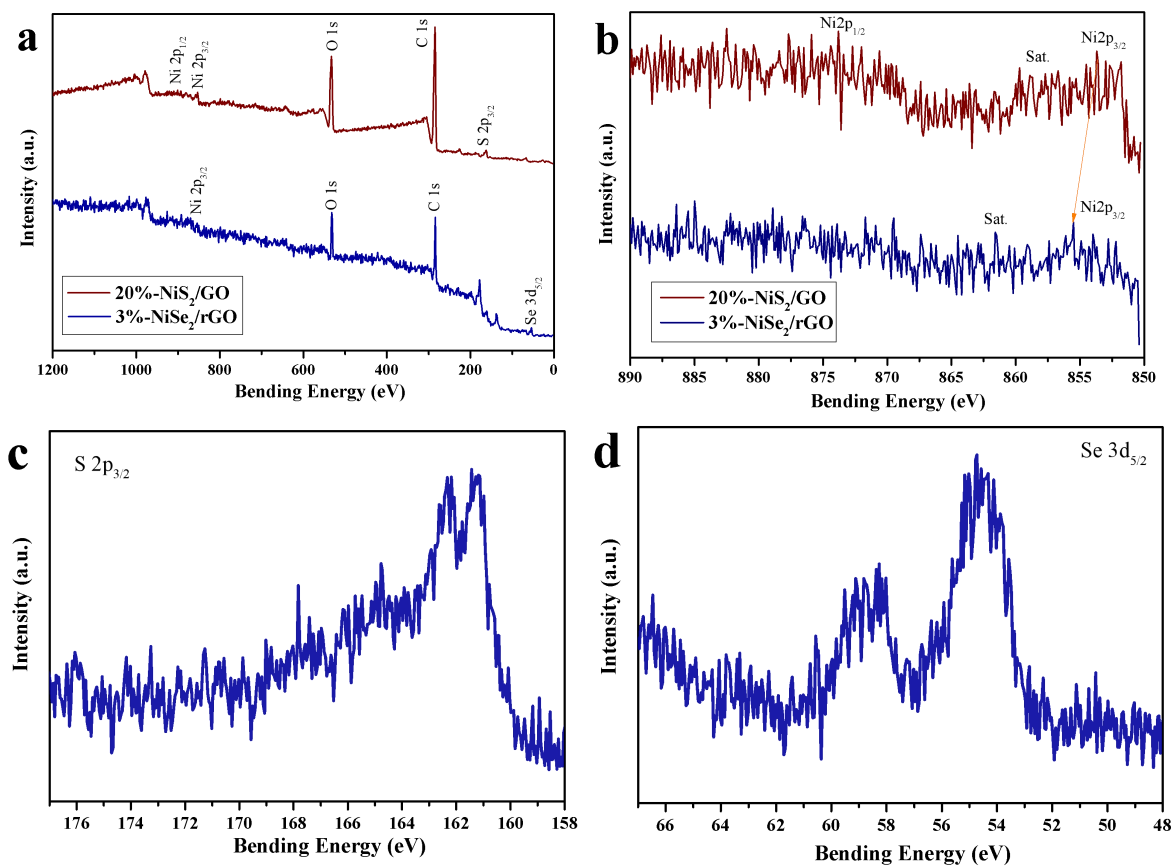


Fig. S5 XPS spectra for: (a) 20%-NiS₂/GO, 3%-NiSe₂/rGO; (b) Ni 2p; (c) S 2p; (d) Se 3d.

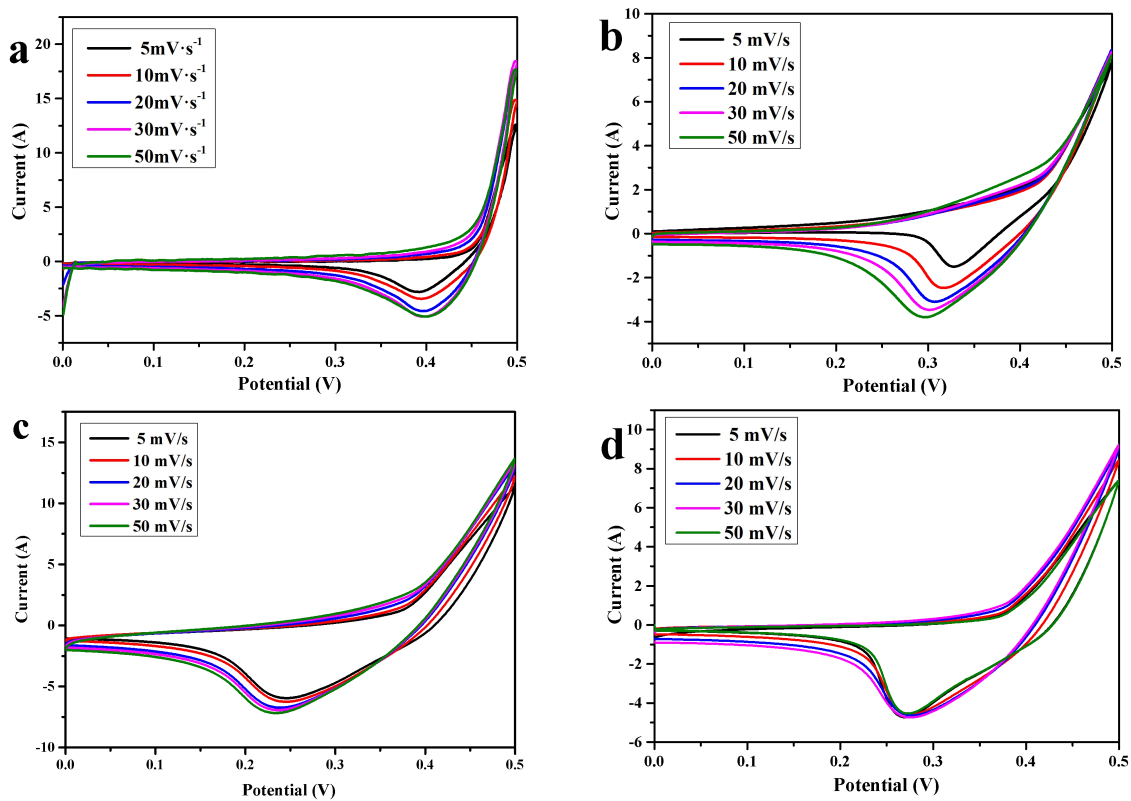


Fig. S6 CV curves of the electrodes at different scan rates: (a) NiS_2 ; (b) 10%- NiS_2/GO ; (c) 20%- NiS_2/GO ; (d) 30%- NiS_2/GO .

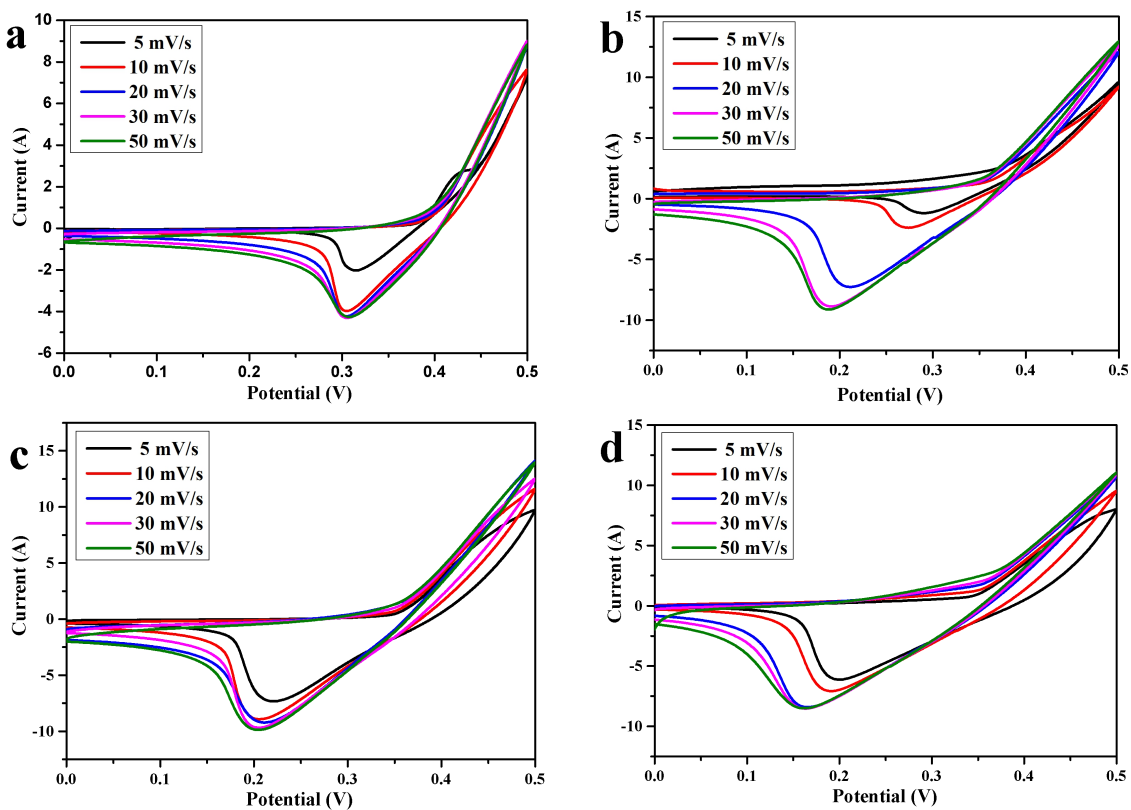


Fig. S7 CV curves of the electrodes at different scan rates: (a) NiSe₂; (b) 1%-NiSe₂/rGO; (c) 3%-NiSe₂/rGO; (d) 5%-NiSe₂/rGO.

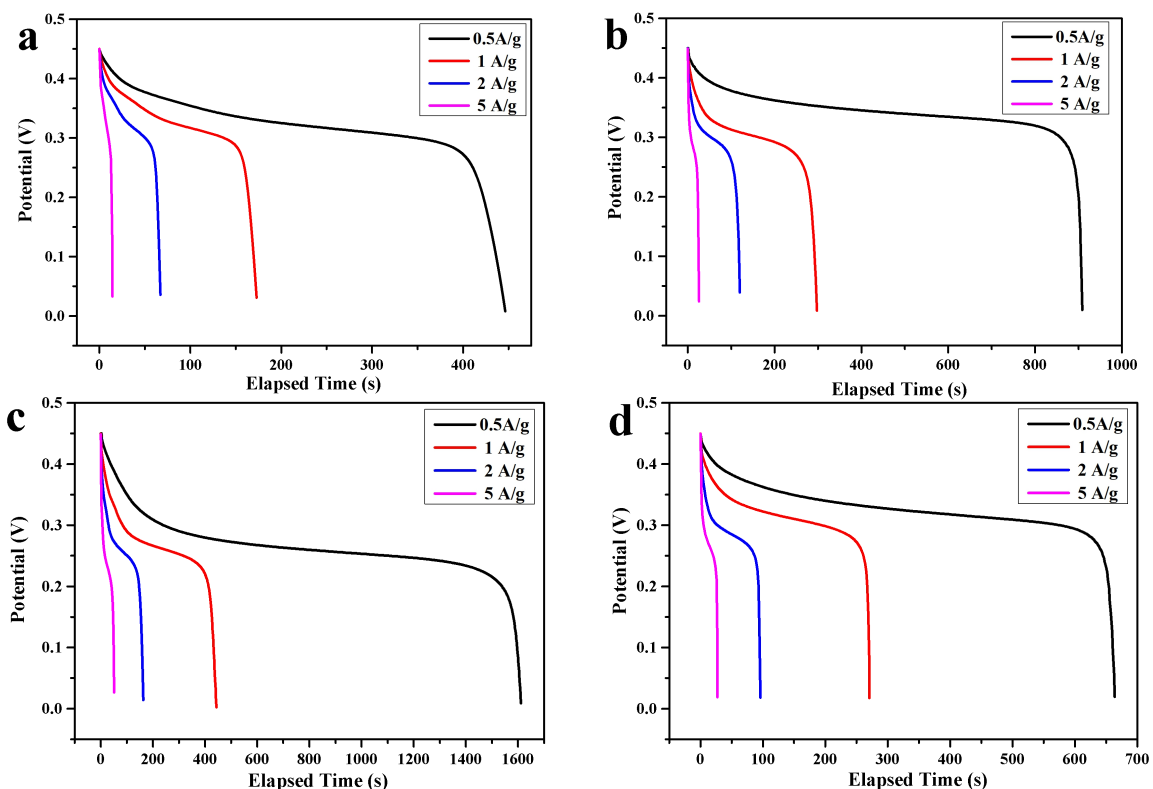


Fig. S8 Discharge curves of the electrodes at various current densities: (a) NiS₂; (b) 10%-NiS₂/GO; (c) 20%-NiS₂/GO; (d) 30%-NiS₂/GO.

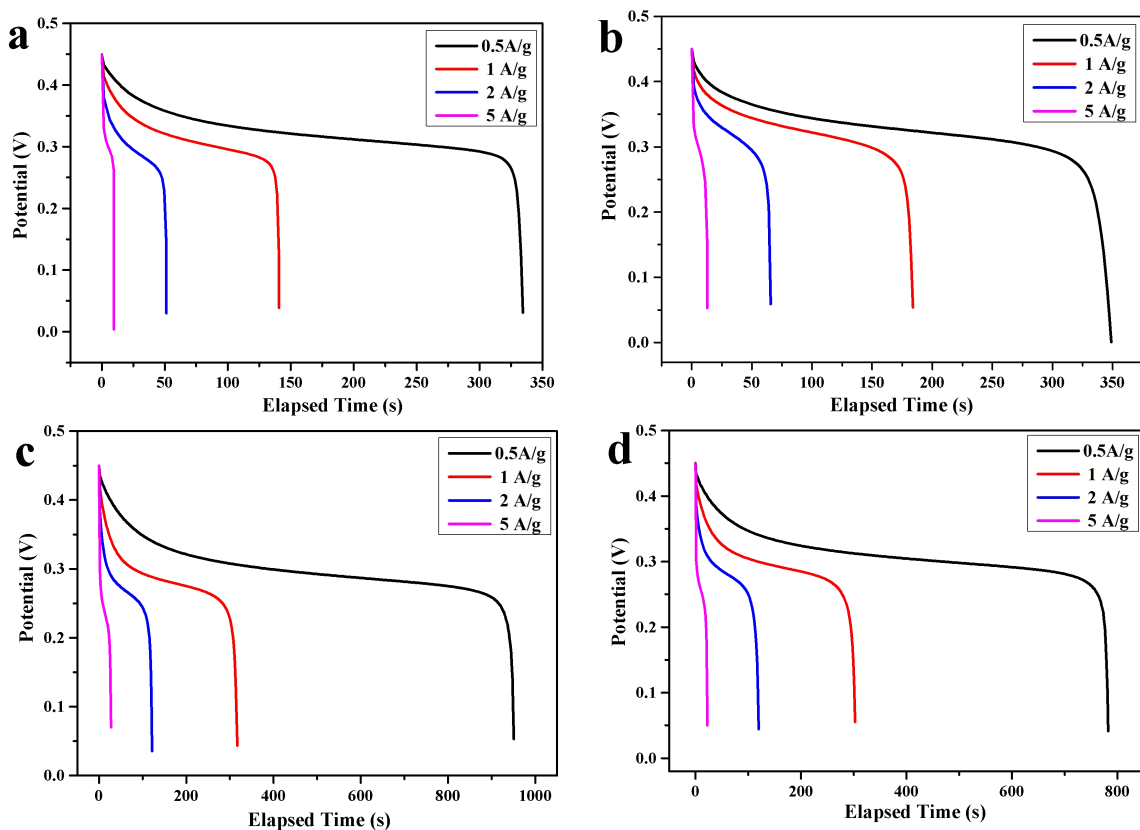


Fig. S9 Discharge curves of the electrodes at various current densities: (a) NiSe₂; (b) 1%-

NiSe₂/rGO; (c) 3%-NiSe₂/rGO; (d) 5%-NiSe₂/rGO.

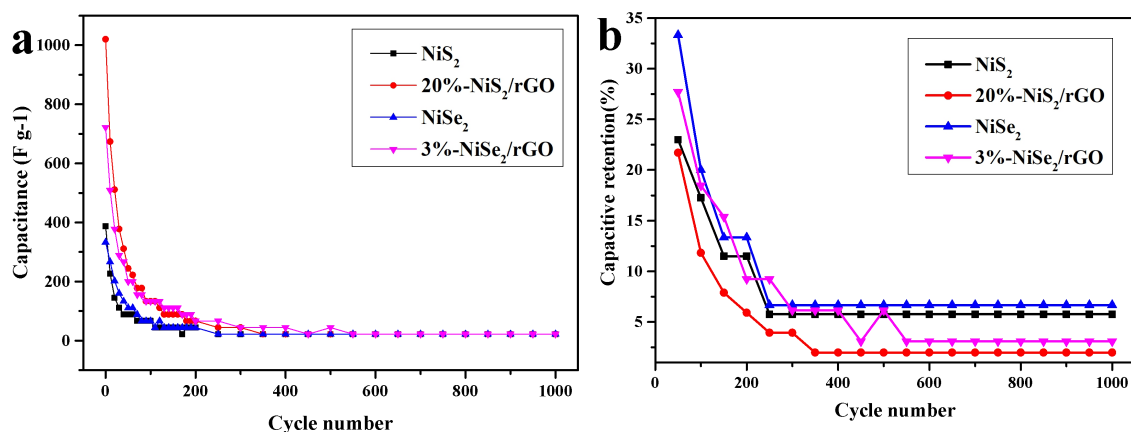


Fig. S10 (a) cycle performance curves of the four electrodes at a current density of 1 A·g⁻¹; (b) the capacitive retentions of the four electrodes during 1000 cycles.

Table S1 Performance comparisons with other works

Sample	Morphology and structure	Specific capacitance (0.5A·g ⁻¹)	Specific capacitance (1A·g ⁻¹)	Specific capacitance (2A·g ⁻¹)	Specific capacitance (5A·g ⁻¹)
15%-NiS ₂ /GO	Hollowspheres	1213 F·g ⁻¹	676 F·g ⁻¹	562 F·g ⁻¹	373 F·g ⁻¹
25%-NiS ₂ /GO	Hollowspheres	810 F·g ⁻¹	640 F·g ⁻¹	533 F·g ⁻¹	409 F·g ⁻¹
7%-NiSe ₂ /rGO	Nanospheres	659 F·g ⁻¹	622 F·g ⁻¹	412 F·g ⁻¹	213 F·g ⁻¹
10%-NiSe ₂ /rGO	Nanospheres	537 F·g ⁻¹	402 F·g ⁻¹	320 F·g ⁻¹	124 F·g ⁻¹