Large and fast reversible Li-ion storages in Fe$_2$O$_3$-graphene sheet-on-sheet sandwich-like nanocomposites

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Fe$_2$O$_3$ nanosheets and nanoparticles are grown on graphene by simply varying reaction solvents in a facile solvothermal/hydrothermal preparation. Fe$_2$O$_3$ nanosheets are uniformly dispersed among graphene nanosheets, forming a unique sheet-on-sheet nanostructure. Due to the structure affinity between two types of two dimensional nanostructures, graphene nanosheets are separated better by Fe$_2$O$_3$ nanosheets compared to nanoparticles and their agglomeration is largely prevented. A large surface area of 173.9 $m^2 g^{-1}$ is observed for Fe$_2$O$_3$-graphene sheet-on-sheet composite, which is more than two times as large as that of Fe$_2$O$_3$-graphene particle-on-sheet composite (81.5 $m^2 g^{-1}$). The sheet-on-sheet composite is found to be better suitable as an anode for Li-ion battery. A high reversible capacity of 662.4 mAh g$^{-1}$ can be observed after 100 cycles at 1 C. The substantially improved cycling performance is ascribed to the unique structure affinity between Fe$_2$O$_3$ nanosheets and graphene nanosheets, thus offering complementary property improvement.

Results

Characterizations of Fe$_2$O$_3$-graphene composites. Fig. 1a shows the crystal phase of Fe$_2$O$_3$ nanoparticle, Fe$_2$O$_3$ nanosheet, Fe$_2$O$_3$-graphene particle-on-sheet and Fe$_2$O$_3$-graphene sheet-on-sheet. All diffraction peaks in these...
samples can be assigned well to the standard Fe$_2$O$_3$ (PDF 33-0664). There is no clear observation of the (002) diffraction peak of graphene, because the (012) diffraction peak of hematite is strong, which can shadow the (002) peak at the similar 2$\theta$. Fig. 1b shows the Raman spectra of graphene nanosheet (GNS), Fe$_2$O$_3$-graphene particle-on-sheet and Fe$_2$O$_3$-graphene sheet-on-sheet. All three samples display similar two bands at $\sim$1320 and 1580 cm$^{-1}$, which correspond to the disordered (D) band and graphitic (G) band of carbon materials respectively. The intensity ratios of the D to G band (I$_D$ : I$_G$) are calculated to be 1.34 and 1.36 for Fe$_2$O$_3$-graphene particle-on-sheet and sheet-on-sheet composite, respectively, which are both larger than that of GNS (1.18). This enhancement of the disordered carbon content should be ascribed to the partial insertion of Fe$_2$O$_3$ nanoparticles or nanosheets into

Figure 1 | Structural analysis of Fe$_2$O$_3$-based products. (a) Powder X-ray diffraction (XRD) patterns of various Fe$_2$O$_3$-based products, (b) Raman spectrum. Nitrogen sorption isotherms of (c) Fe$_2$O$_3$-graphene particle-on-sheet composite, and (d) Fe$_2$O$_3$-graphene sheet-on-sheet composite, TGA curves of (e) Fe$_2$O$_3$-graphene particle-on-sheet and (f) Fe$_2$O$_3$-graphene sheet-on-sheet.
graphene layer, which is in accordance with previous reports about graphene-based nanocomposites. To explore the porous structure and specific surface area of Fe$_2$O$_3$-graphene sheet-on-sheet and particle-on-sheet composites, nitrogen sorption investigations have been carried out. The porous attribute of these nanocomposites are shown in Fig. 1c and 1d. Both adsorption and desorption curves exhibit an IUPAC IV type curve characteristic, which indicate that there are many mesopores in two Fe$_2$O$_3$-graphene composites. The surface area of Fe$_2$O$_3$-graphene sheet-on-sheet is determined to be 173.9 m$^2$ g$^{-1}$ by fitting the isotherms to the BET model. This value is substantially higher than that of Fe$_2$O$_3$-graphene particle-on-sheet (81.5 m$^2$ g$^{-1}$), indicating that graphene nanosheets are separated better in the Fe$_2$O$_3$-graphene sheet-on-sheet composite compared to particle-on-sheet composite. This is because nanosheet has a better structure suitability with graphene nanosheets due to their similar two dimensional nanostructures. Therefore there are more contact area between GNS and Fe$_2$O$_3$ nanosheet compared to that of the particle-on-sheet composite, which can effectively prevent the reassembly of GNS to graphite platelets. Moreover, nanosheets are more stable than nanoparticles because their sizes are larger than the particle sizes of Fe$_2$O$_3$ nanoparticles. In comparison, nanosized Fe$_2$O$_3$ particles may be easily agglomerated. The pore size distribution curves of Fe$_2$O$_3$-graphene sheet-on-sheet and Fe$_2$O$_3$-graphene particle-on-sheet are shown in the inset of Fig. 1c and 1d respectively. Based on the BJH calculation, the sheet-on-sheet composite displays a sharp distribution peak centered at 3.4 nm, which is similar to that of particle-on-sheet composite (3.3 nm). These similar pore size distributions indicate that the observed mesopores are mainly from graphene materials in two Fe$_2$O$_3$-graphene composites. According to thermal gravimetric
analysis in air as shown in Fig. 1e–f, the weight percentages of graphene were estimated to be 59.4 wt% for Fe$_2$O$_3$-graphene sheet-on-sheet and 54.9 wt% for Fe$_2$O$_3$-graphene particle-on-sheet. These values are both slightly less than the theoretical value (66.7 wt%), which is calculated based on the experimental conditions.

Fig. 2a–e show SEM images of various Fe$_2$O$_3$-based products. A large number of pristine Fe$_2$O$_3$ nanoparticles are shown in Fig. 2a. Their particle sizes are $\sim 30$–50 nm, which is substantially smaller than that of pristine Fe$_2$O$_3$ nanosheets ($\sim 100$–250 nm in size) as shown in Fig. 2b. In the presence of graphene, Fe$_2$O$_3$-graphene particle-on-sheet and sheet-on-sheet composites both have curled paper-like structure (Fig. 2c and 2d–e). It can be shown that Fe$_2$O$_3$ nanoparticles and nanosheets are uniformly distributed on graphene nanosheets. Fig. 2e shows clearly that Fe$_2$O$_3$ nanosheets are wrapped by graphene nanosheets, and therefore on the other hand, graphene nanosheets are also separated by Fe$_2$O$_3$ nanosheets. Notably, compared to Fe$_2$O$_3$-graphene sheet-on-sheet, a similar method except for using deionized water as solvent to replace isopropanol was used to obtain pristine Fe$_2$O$_3$ nanoparticles. Fig. 2f shows the energy dispersive spectroscopy (EDS) of Fe$_2$O$_3$-graphene sheet-on-sheet composite. A few elements such as C, O, and Fe are present in the composite. The Si element is observed because it was used as a substrate to disperse SEM sample, thus removing the carbon effect from the common carbon support. The carbon contents in the sheet-on-sheet and particle-on-sheet were determined to be 59.7 wt% and 57.9 wt%, which are slightly different from the results indicated from TGA analysis. It is believed that the latter should be more accurate than the EDS results for the determination of materials composition. The particle-on-sheet and sheet-on-sheet nanostructures can be further confirmed by TEM images of Fig. 3a and Fig. 3b–d, respectively.

These Fe$_2$O$_3$ nanoparticles and nanosheets are uniformly distributed on graphene nanosheets. There is no observation of Fe$_2$O$_3$ materials outside the graphene support even after strong sonication in ethanol used for TEM measurement. It should be ascribed to the binding effect of graphene, which can immobilize Fe$_2$O$_3$ materials and prevent their movement and agglomeration. Notably, it is clear from TEM images with higher magnifications in Fig. 3c–d that the obtained Fe$_2$O$_3$ nanosheets exhibit irregular sheet-like structure with the size of $\sim 100$–250 nm.

**Exploration of the effect of experimental conditions on Fe$_2$O$_3$ morphologies.** Fig. 4 shows SEM images of the obtained Fe$_2$O$_3$ products by varying the reaction solvent. A large number of thick platelets were obtained by using 10 mL ethanol with 0.07 mL water (Fig. 4a). If 10 mL propanol with 0.07 mL water was used as the solvent, the obtained product exhibited sheet-like structure with larger particle sizes of several hundred nanometers to a few micrometers (Fig. 4b). As reported previously, a trace amount of water can facilitate the crystallization of Fe$_2$O$_3$. Therefore various amounts of water were also explored in the preparation. Fig. 4c shows Fe$_2$O$_3$ platelets with a smaller size of $\sim 100$ nm prepared from 10 mL isopropanol with increased amount of water (0.7 mL). In comparison, agglomerated Fe$_2$O$_3$ sheets and platelets were obtained by using 10 mL isopropanol in the absence of water (Fig. 4d). Fig. 5 show SEM images of Fe$_2$O$_3$-graphene composites grown from 10 mL propanol (Fig. 5a–b) and the mixed solvent of 10 mL propanol and 0.07 mL water (Fig. 5c–d). Although several Fe$_2$O$_3$ sheets can be shown in Fig. 5a and 5c, some nanoparticle products were also observed in Fig. 5b and 5d with higher magnifications. Compared to these benchmarked products by varying reaction solvents, it is...
clear that the main products of Fe$_2$O$_3$ nanoparticles and nanosheets (as shown in Fig. 2) have more uniform particle size and morphology control. There is also less agglomeration of Fe$_2$O$_3$ materials in the corresponding composites. Based on the above observations, it is believed that the mixed solvent of water and isopropyl is a very important factor for the synthesis of sheet-like Fe$_2$O$_3$. The organic solution may offer a suitable reaction environment for the growth of sheet-like crystal. The growth process of Fe$_2$O$_3$-graphene sheet-on-sheet or particle-on-sheet composites is illustrated in Fig. 5e. Fe$_2$O$_3$ nanosheets were obtained in the organic solvent with a trace amount of water, however nanoparticles were formed in a pure aqueous solvent. These Fe$_2$O$_3$ nanosheets or nanoparticles were formed among graphene nanosheets, forming a sandwiched sheet-on-sheet or particle-on-sheet nanostructure, which can separate graphene nanosheets against their reassembly. Because Fe$_2$O$_3$ nanosheet structure is basically also two dimensional structures similar to graphene nanosheet, therefore it has a large contact area with graphene and better structure affinity. It is believed that Fe$_2$O$_3$ nanosheet is a better spacing material compared to Fe$_2$O$_3$ nanoparticles, which can be confirmed by higher surface area in sheet-on-sheet composite compared to particle-on-sheet composite (Fig. 1c–d).

**Electrochemical properties of Fe$_2$O$_3$-graphene composites.** To explore the Li-ion storage properties of Fe$_2$O$_3$-graphene composites, carbon black, and polyvinyl difluoride (PVDF) were mixed at the weight ratio of 8:1:1 as working electrodes. The CV curves of Fe$_2$O$_3$-graphene sheet-on-sheet and particle-on-sheet electrodes were performed at a scan rate of 0.1 mV s$^{-1}$ as shown in Fig. 6a. There are two cathodic peaks at $\sim$1.56 V and $\sim$0.68 V for the sheet-on-sheet composite, which can be attributed to the stepwise reduction of Fe$^{3+}$ to Fe$^0$ and the formation of solid electrolyte interface (SEI) film around the electrode$^{29,30}$. Meanwhile, two anodic peaks at $\sim$1.90 V and $\sim$2.35 V are observed, corresponding to the stepwise oxidation process of Fe$^0$ to Fe$^{3+}$ and Fe$^{3+}$ to Fe$^{4+}$, respectively$^{34,35}$. Compared to the sheet-on-sheet composite, the particle-on-sheet composite displays two cathodic peaks and anodic peaks at similar positions with substantially weaker intensity. It is indicated that the sheet-on-sheet composite has a better electrochemical activity due to larger surface area and shorter diffusion route for lithium insertion and extraction reactions. The overall reversible electrochemical reactions process can be described by the following equation$^{32}$.

$$\text{Fe}_2\text{O}_3 + 6\text{Li}^+ + 6\text{e}^- \leftrightarrow 2\text{Fe} + 3\text{Li}_2\text{O}$$

Fig. 6b shows the initial discharge (lithium insertion) and charge (lithium extraction) voltage profiles of various anodes at 0.1 C (1 C = 1000 mA g$^{-1}$). The Fe$_2$O$_3$-graphene sheet-on-sheet, Fe$_2$O$_3$-graphene particle-on-sheet, Fe$_2$O$_3$ nanosheet, Fe$_2$O$_3$ nanoparticle and bare graphene exhibited initial discharge capacities of 1652.8, 1421.7, 1140.8, 1413.3 mAh g$^{-1}$ and charge capacities of 1074.9, 890.4, 812.2, 901.6, 863.4 mAh g$^{-1}$ respectively. A Coulombic efficiency of 65% can be calculated for the Fe$_2$O$_3$-graphene sheet-on-sheet, which should be largely due to the irreversible capacity loss occurred in the formation of solid electrolyte interface (SEI) film. Notably, the charge capacity (1074.9 mAh g$^{-1}$) of Fe$_2$O$_3$-graphene sheet-on-sheet composite is slightly larger than the theoretical capacity (1007 mAh g$^{-1}$) of Fe$_2$O$_3$. This may be attributed to additional storage of Li-ions in the defects or micro-pores of the sheet-on-sheet composite induced by partial insertion of Fe$_2$O$_3$ into graphene. A more disordered carbon structure was observed in Fe$_2$O$_3$-graphene

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**Figure 4 | The morphological analysis of Fe$_2$O$_3$ products grown from the different solvent by SEM.** (a) 10 mL ethanol with 0.07 mL water, (b) 10 mL propanol with 0.07 mL water, (c) 10 mL isopropanol with 0.7 mL water, and (d) 10 mL isopropanol in the absence of water.
sheet-on-sheet composite as confirmed by Raman results in Fig. 1b. Compared to the particle-on-sheet composite, the sheet-on-sheet composite also has more active sites for lithium ion storage due to its larger BET surface area. Two voltage plateaus (~1.51 and 0.76 V) are observed in the discharge curve, which can be ascribed to the Li⁺ insertion into Fe₂O₃ anode and the formation of SEI film. There are also two voltage plateaus (~1.82 and ~2.27 V) in the charge curve, corresponding to the stepwise oxidation of Fe⁰ to Fe³⁺. These observations agree with the CV results as shown in Fig. 6a. 

Fig. 6c and 6d compare the cycling performances of the Fe₂O₃-graphene sheet-on-sheet, Fe₂O₃-graphene particle-on-sheet, Fe₂O₃ nanosheet, Fe₂O₃ nanoparticle and bare graphene at a constant small current rate of 0.1 C. Pristine Fe₂O₃ nanoparticle displayed a fast capacity fading from first-cycle 901.6 mAh g⁻¹ to 367.1 mAh g⁻¹ after 50 cycles. In comparison, Fe₂O₃ nanosheet showed a lower initial charge capacity than Fe₂O₃ nanoparticle, but much better cycling performance in 50 cycles. A higher charge capacity of 466.3 mAh g⁻¹ could be observed after 50 cycles. Bare graphene exhibited similar capacity fading to that of Fe₂O₃ nanosheet and a charge capacity of 468.5 mAh g⁻¹ was observed after 50 cycles. The cycling stabilities of two Fe₂O₃-graphene composites are both better than pristine Fe₂O₃ nanoparticles or nanosheets. It should be ascribed to the positive contribution from the graphene nanosheet support. It can be found that graphene play a positive role for improving the performance of Fe₂O₃ as anodes for Li ion batteries. Graphene can not only provide a large specific surface area for the insertion and extraction of lithium ions, but also buffer the volume change of Fe₂O₃ anodes during cycling. Compared to the particle-on-sheet composite, the sheet-on-sheet composite showed better cycling performances. The Fe₂O₃-graphene sheet-on-sheet has a high reversible charge capacity of 800.6 mAh g⁻¹ after 50 cycles, which is almost 1.65 times as large as that of Fe₂O₃-graphene.

Figure 5 | The morphological analysis of Fe₂O₃-graphene nanocomposites grown from the different solvent by SEM and Schematic illustration. (a, b) 10 mL propanol, and (c, d) 10 mL propanol with 0.07 mL water. (e) Schematic illustration of the growth process of Fe₂O₃-graphene sheet-on-sheet and particle-on-sheet composites.
particle-on-sheet (485.2 mAh g\(^{-1}\)) after the same cycle number. It is noted that the graphene content in Fe\(_2\)O\(_3\)-graphene sheet-on-sheet composite (59.4 wt%) is comparable to that of the particle-on-sheet composite (54.9 wt%) as indicated by TGA results.

The electrical conductivities of the obtained Fe\(_2\)O\(_3\)-based products were measured by a four-electrode method. Fe\(_2\)O\(_3\) is highly insulated because its electrical conductivity is too small to be measured. In comparison, the electrical conductivities of Fe\(_2\)O\(_3\)-graphene composites are greatly enhanced and 0.156 S cm\(^{-1}\) and 0.138 S cm\(^{-1}\) were determined for the Fe\(_2\)O\(_3\)-graphene sheet-on-sheet and Fe\(_2\)O\(_3\)-graphene particle-on-sheet respectively. Due to the increased electrical conductivity, it is meaningful to explore the rate performances of Fe\(_2\)O\(_3\)-graphene composites. As shown in Fig. 7a–b, the Fe\(_2\)O\(_3\)-graphene sheet-on-sheet also exhibited an excellent rate capability. High initial charge capacities of 833.9, 798.6 and 792.2 mAh g\(^{-1}\) were observed at high current rates of 1, 2 and 5 C, respectively. After comparatively fast capacity fading in the first few cycles, the sheet-on-sheet composite showed a very stable cycliability in the following cycles. High capacities of 662.4, 456.2, and 322.5 mAh g\(^{-1}\) are retained after 100 cycles at 1, 2, and 5 C, respectively. Fig. 7c shows the high-rate cycling performances of Fe\(_2\)O\(_3\)-graphene particle-on-sheet composite. After 100 cycles of discharge and charge, reversible capacities of 318.1, 170.4, and 138.4 mAh g\(^{-1}\) were observed at 1, 2, and 5 C, respectively.

Discussion

These high-rate cycling performances of Fe\(_2\)O\(_3\)-graphene particle-on-sheet are much worse than those of Fe\(_2\)O\(_3\)-graphene sheet-on-sheet. Because two composites have similar values of electrical conductivities and comparable graphene contents in the composites, the observed large difference between the high-rate performances of two composites should be largely ascribed to their structure difference. Fe\(_2\)O\(_3\) nanosheet has a better structure affinity with graphene compared to Fe\(_2\)O\(_3\) nanoparticles. Therefore the agglomeration of graphene is prevented better in sheet-on-sheet structure compared to particle-on-sheet structure as confirmed by the BET measurements in Fig. 1c–d. Compared with graphene-supported Fe\(_2\)O\(_3\) nanorice anode tested under similar test conditions\(^{29}\), the Fe\(_2\)O\(_3\)-graphene sheet-on-sheet composite also reveals enhanced Li\(^+\) storage properties especially at high current rates. For example, a charge capacity of 582 mAh g\(^{-1}\) was reported at 1 C after 100 cycles for graphene-supported Fe\(_2\)O\(_3\) nanorice composite\(^{29}\), however a higher charge capacity of 662.4 mAh g\(^{-1}\) was observed for the sheet-on-sheet composite in this work. It may be ascribed to the following points. The sheet-on-sheet composite should display the best structure affinity in all graphene-supported metal oxide morphologies because there are large intimate contact areas between these two types of two dimensional materials (Fe\(_2\)O\(_3\) nanosheets and graphene nanosheets). Graphene nanosheets are separated well by numerous Fe\(_2\)O\(_3\).
unique sheet-on-sheet nanostructure. Moreover, owing to the unique intrinsic promising properties of graphene nanosheets, which hinders the agglomeration of the latter. When used as an anode for Li-ion battery, the sheet-on-sheet nanostructure exhibited a high reversible capacity of 1074.9 mAh g⁻¹ with good cycling performance at 0.1 C. This composite also exhibited an excellent high-rate capability. A high reversible capacity of 622.4 mAh g⁻¹ was retained after 100 cycles at a high current rate of 1 C.

**Methods**

**Preparation of graphite oxide (GO).** Graphite oxide was synthesized from natural graphite by a modified Hummer’s method. 1 g natural graphite powder was mixed with 50 mL 65 wt% HNO₃ and 50 mL 98 wt% H₂SO₄ in an ice bath. After strong magnetic stirring for 30 min, 5 g KMnO₄ was added gradually and reacted for 2 h. 200 mL deionized water and 5 mL 30 wt% H₂O₂ were added dropwise to the mixture and the solution color changed to brilliant yellow, followed by washing with 15 mL 10 wt% HCl aqueous solution. The mixture was then centrifuged (12000 rpm) and washed with deionized water. Graphite oxides were obtained after drying on vacuum. After thermal reduction heating in a tube furnace in N₂ at 300 °C for 2 h, the resultant black powders were collected as graphene nanosheets.

**Preparation of Fe₂O₃-graphene composites.** 0.027 g FeCl₃·6H₂O and 0.08 g CH₃COONa were dissolved in 10 mL isopropanol with a trace amount of 0.07 mL deionized water. 0.016 g graphene nanosheets (GNS) were also dispersed in 10 mL isopropanol and 0.07 mL deionized water by ultrasonication, and then mixed with previous FeCl₃ precursor solution (the theoretical weight ratio of Fe₂O₃ to GNS was 1:2). The mixture suspension was sealed in a 60 mL Teflon lined stainless steel autoclave and heated at 180 °C for 12 h. After cooling to room temperature, the precipitate (Fe₂O₃-graphene sheet-on-sheet) was collected after copious washing by deionized water. The Fe₂O₃-graphene particle-on-sheet composite was prepared by the same method except that 10 mL deionized water was used as the solvent to dissolve FeCl₃ and disperse GNS. Pristine Fe₂O₃ nanoparticles and nanosheets were also prepared by similar preparation process in the absence of GNS. Ethanol, propanol and isopropanol with various amounts of deionized water were also used to explore the solvent effect on the Fe₂O₃ products.

**Materials characterizations.** The obtained products were characterized by field-emission scanning electron microscopy (FE-SEM, JSM-6700F) with an energy dispersive X-ray spectrometer (EDS), transmission electron microscopy (TEM, JEOL JEM-200CX) and X-ray diffraction (XRD, Rigaku D/max-2550V, Cu Kα radiation). Raman spectroscopy was recorded on Renishaw in plus laser Raman spectrometer (excitation wavelength: 785 nm, excitation power: 3 mW, spot size: ~1.2 μm). The electrical conductivity was measured by a four-electrode method using a conductivity detector (Shanghai Fortune Instrument, EZ-2010). The specific surface area and porous structures were tested by an accelerated surface area and porosimetry analyzer (Micromeritics Instrument Corp, ASAP 2020 M + C, analysis adsorptive: N₂). Thermogravimetric analysis (TGA) was performed on a NETZSCH STA 409 PG/PC instrument in air.

**Electrochemical measurements.** The working electrodes were composed of 80 wt% of active material, 10 wt% of the conductivity agent (acetylene black), and 10 wt% of the binder (poly(vinylidene difluoride)), PVDF, Aldrich). The loading amount of the electrode on copper foil was kept at ~2 mg cm⁻². The thickness of electrode materials was ~20 micrometers. Lithium foil (China Energy Lithium) was used as counter and reference electrode. The electrolyte was 1 M LiPF₆ in a 50:50 w/w mixture of ethylene carbonate (EC) and diethyl carbonate (DEC). Electrochemical measurements were performed on a LAND-CT2001C test system. The Swagelok-type cells were discharged (lithium insertion) and charged (lithium extraction) at a constant current of active material, 10 wt% of the conductivity agent (acetylene black), and 10 wt% of the binder (poly(vinylidene difluoride)), PVDF, Aldrich). The loading amount of the electrode on copper foil was kept at ~2 mg cm⁻². The thickness of electrode materials was ~20 micrometers. Lithium foil (China Energy Lithium) was used as counter and reference electrode. The electrolyte was 1 M LiPF₆ in a 50:50 w/w mixture of ethylene carbonate (EC) and diethyl carbonate (DEC). Electrochemical measurements were performed on a LAND-CT2001C test system. The Swagelok-type cells were discharged (lithium insertion) and charged (lithium extraction) at a constant current.
(100 mA g\(^{-1}\), 0.1 C, 1 C = 1000 mA g\(^{-1}\)) in the fixed voltage range 5 mV to 3.0 V. Higher hourly rates (1, 2, or 5 C) were also used, and the first cycle discharging was kept at 0.1 C. Cyclic voltammetry (CV) was performed on a CHI660D electrochemical workstation at a scan rate of 0.1 mV s\(^{-1}\). Nyquist plots were collected on the same workstation for various electrodes at a discharged potential of 0.8 V versus Li\(^+\)/Li after 5 cycles from 100 kHz to 10 mHz.

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Author contributions

JK performed the experiments. J.K. and Y.W. designed the experiments, discussed the results and wrote the manuscript.

Additional information

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