Supplementary Information for

Facile synthesize of 3D-porous LiNbO₃ nanocomposite as a novel electrode material for lithium ion batteries

Experimental section

1. Synthesis

In a typical synthesis, the 3D-porous LiNbO₃ composites were prepared by citrate auto combustion route. The A.R. grade citric acid ($C_6H_8O_7.3H_2O$), (NH₄)₃NbO (C_2O_4)₃ and CH₃COOLi were used as starting materials. First, the citric acid was solved into the distilled water (200 mL) to acquire the citric acid solution, then the precursor solution was prepared by dissolving (NH₄)₃NbO (C_2O_4)₃ and CH₃COOLi to citric acid solution with the molar ratio as (NH₄)₃NbO (C_2O_4)₃: CH₃COOLi: $C_6H_8O_7.3H_2O = 1:1:3$. The mixed solution was kept on to a hot plate with continuous stirring at 80°C. During evaporation, the solution became viscous and finally formed a gel. The viscous gel were dried at 100°C in the vacuum drying oven to remove the residual moisture and then pressed to be circular tablets with 15 mm diameter.

The acquired Li-Nb-O circular tablets were placed in a glass vessel, which is transparent to microwave irradiation (microwave transmitter), and irradiated at 400W in a microwave (Mw) system. Owing to the rapid intermittent heat adsorption from the Mw treatment, after a few minutes, the citric acid inside the tablets was automatically ignited and released quantity of decomposed gas. Such reaction can introduce the porous morphology into the composites. Meanwhile, the large amount of heat released in this procedure facilitated the crystallization of LiNbO₃. The asprepared porous samples were used for further characterization.

In order to demonstrate the superiority of these 3D-porous LiNbO₃ composites, we prepared the pristine LiNbO₃ composites for comparison. The Li-Nb-O precursor of the controlled group was utilizing the same procedure without citric acid added. The acquired precursor was sintered for 10 h at 500 °C in a tube furnace to acquire the product.

2. Characterization

X-ray diffraction measurements were conducted on an X-ray powder diffractometer using Cu-K α radiation (D/Max-RA). Field-emission scanning electron microscopy (FE-SEM) experiments were conducted on a Hitachi S-4800 instrument. Transmission electron microscopy (TEM) experiments were carried out on a Titan 80-300 S/TEM operated at 150.0 kV.

Nitrogen sorption isotherms of the porous composites were collected at 77 K using Micromeritics ASAP2020. Brunauer–Emmett–Teller (BET) and Barrett–Joyner–Halenda (BJH) models are used for the specific surface area and porosity evaluation, respectively.

3. Electrochemical measurement

To test the electrochemical performance of acquired materials, the porous LiNbO₃ and pristine LiNbO₃ electrodes were prepared by mixing 80% of pristine LiNbO₃, 10% carbon black, and 10% PVDF dispersed in N-methylpyrrolidinone (NMP), respectively. The homogenous slurries were coated on a Cu substrate and dried at 60 °C for 1 h under vacuum. As formed electrodes were then pressed at a pressure of 2 MPa and further dried under vacuum at 100 °C for another 10 h. The mass loading was 3-4 mg cm⁻² on the current collect. All the cells were assembled in an argon-filled glove box. The constant-current charge and discharge measurements were conducted by a LAND CT2001-A at different current densities in a voltage range between 1.0 and 3.0 V (vs. Li/Li⁺). CV tests and EIS measurements were performed on CHI-660C electrochemical workstation..All of the electrochemical performance measurements were obtained at a constant temperature of 25 °C.



Fig. S1 BJH pore size distribution of 3D-porous coral-like LiNbO₃ from the adsorption branch.



Fig. S2 SEM image of 3D-porous coral-like LiNbO3 textures



Fig. S3 TEM image of 3D-porous LiNbO₃ textures



Fig. S4 Intial 40 cycles of the 3D-porous LiNbO₃ at 0.25 A g⁻¹



Fig. S5 Nyquist plot of 3D-porous LiNbO₃ anode compared with the pristine LiNbO₃ anode.