

, ?(Li₄Ti₅O₁₂)??., ?, Li₄Ti₅O₁₂,,, ?Li₄Ti₅O₁₂?, ?Li₄Ti₅O₁₂, Li₄Ti₅O₁₂?

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Despite the existence of widespread environmental regulations, pollution and global warming still present a tremendous challenge for humanity^{1,2}. Electrical vehicles (EV) and energy storage (ES) devices are gaining popularity for daily use³. However, since challenges persist, the scientific community must develop safe, long-lived, and fast charge-discharge batteries in order to promote the use of such vehicles and ES^{4,5}. Our research group^{6,7,8,9,10,11,12} as well as scientists^{13,14,15} around the world believe that LiFePO₄, lithium iron phosphate (LFP) and Li₄Ti₅O₁₂, lithium titanium oxide (LTO)-based batteries are ideal candidates to meet these needs for ES applications.

In this paper we report the development of a new eco-friendly and scalable method of forming a thin layer of nitrogen-doped carbons on an LTO surface, resulting in successfully filling all the nanopores of the particle with carbons. This technique allows the formation of a uniform 3D pathway of electronic conduction, inside and outside the LTO particle. That is the first example of N-doped carbons filling LTO porous particles. The particles are then protected by a carbon coating that is able to limit the degradation of the battery as well as reduction of the resistance of the electrode. Moreover, the charge capacity at 10C increased by 44%, and the specific energy density of the anode increased by the elimination of superfluous carbons in the electrode preparation.

As reported previously by our research group⁶, commercial LTO is formed by the agglomeration of primary particles of ~10 nm into secondary particles of a few micrometers. This LTO structure i.e. nanoporous micro-spheres, facilitates optimal performance when it is used as an anode material^{45,46}. However, another challenge arises from that structure: in order to optimize the coating and the electrochemical performances, we must develop a method to fill every pore of each secondary particle and fully coating every single particle. Such a method was not yet industrially developed.

Illustration of the carbon coating LTO process (Scheme was drawn by Ms. Eloïse Leroux).

(A) Surface of lithium titanium oxide (LTO) with a layer of N-doped carbon. (B) Cross-section of carbon-coated lithium titanium oxide (LTO-CC3). (C) Magnified images inside the cross-section. Carbon-filled pores are marked by red arrows. (D) Magnified images inside a pore filled by N-doped carbons.

Lithium titanium oxide

(A) Images of particles after fibbing. (B) Mapping of titanium. (C) Mapping of carbon. (D) Images of two primary particles allowing analysis of their junction (scale bar span 50 nm). Electron energy loss spectroscopy (EELS) profile showing the presence of carbons (300 eV and a count of 18000) between the two particles.

(A) Charge-discharge profiles at 0.2C and 25 °C of full cells. (B) Nyquist plots of the cells after formation (0.2 C, 25 °C). (C) Bar diagrams of capacity retention after fast charge. (D) Bar diagrams of capacity retention after fast discharge.

Here, we demonstrated that good electronically conductive materials have a major impact on fast charge performance while insulating material such as poly(styrene) impedes the diffusion of electrons and lithium despite providing good protection against degradation⁶.

Cycle-life performances of LFP-LTO cells (1C, 45 °C).

Acrylonitrile was passed over a bed of basic Al₂O₃. Azobisisobutyronitrile (AIBN) was purified by recrystallisation in methanol and dried under vacuum for 12 hours. The monomers were used immediately after purification. The carbon coated lithium iron phosphate (LFP) was purchased from Sumitomo Osaka Cement and the lithium titanium oxide (LTO) from Posco. PVDF, LiPF₆, and the carbonate solvents were obtained from BASF. The carbon Denka Black was from Denka. The SBR latex and CMC were obtained from Zeon Co. and DKS Co. respectively. All other chemicals from Sigma Aldrich and Acros were used as received. Water had a resistance of 18.2 MΩcm⁻¹. Same materials were reported as a state-of-art materials in our previous publication cited in refs.^{6,7}.

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