ADVANCED ENERGY MATERIALS

Supporting Information

for *Adv. Energy Mater.*, DOI: 10.1002/aenm.201200964

$Li_4Ti_5O_{12}$ Nanocrystals Synthesized by Carbon Templating from Solution Precursors Yield High Performance Thin Film Li-Ion Battery Electrodes

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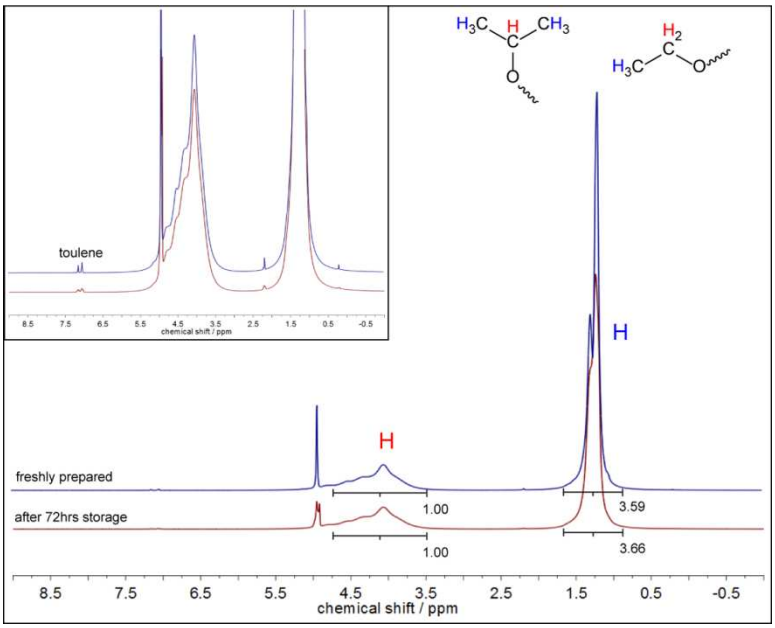

Figure S1. ^{1}H-NMR spectra of fresh precursor solution of LiOEt and Ti(O^iPr)$_4$ in toluene-d_8 (blue) and after storing for 72 h under ambient conditions (red).

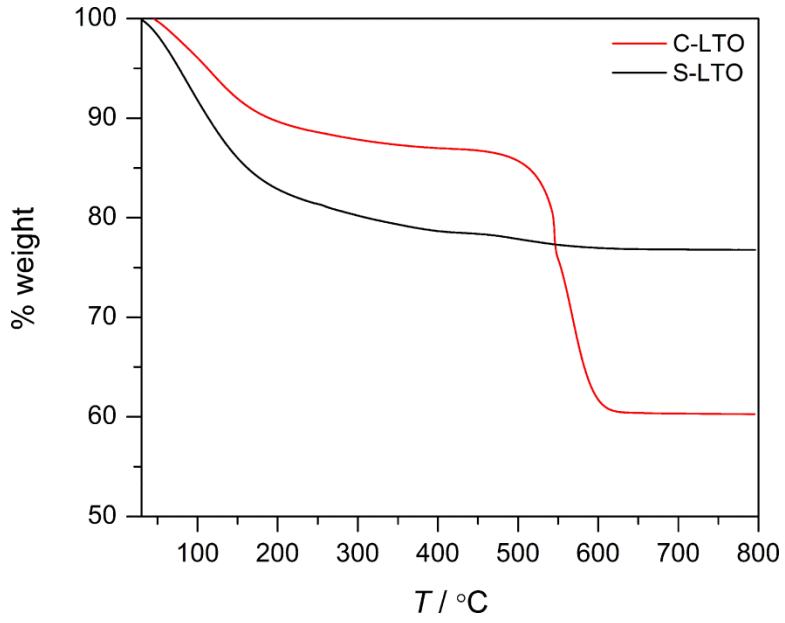

Figure S2. TGA traces of C-LTO nanocrystals and S-LTO aggregates.

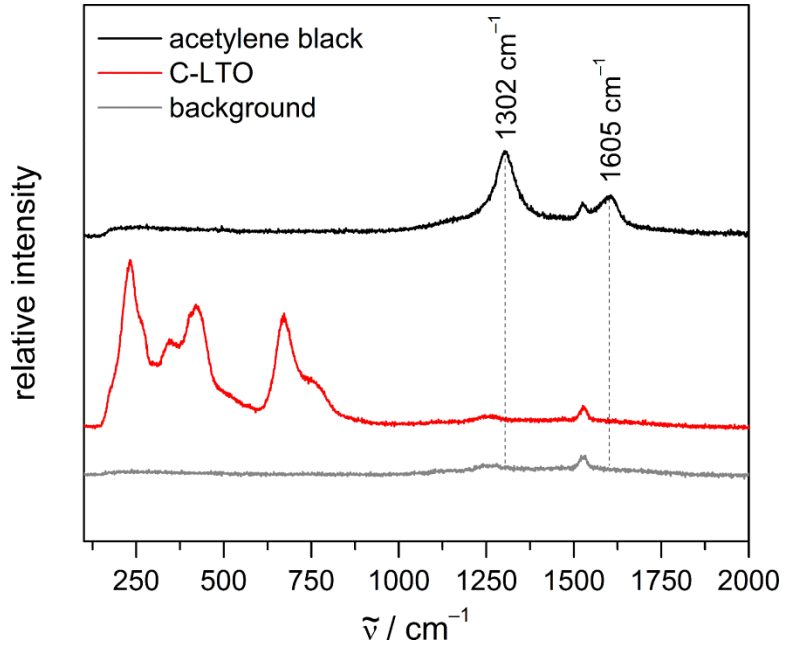

Figure S3. Raman spectrum of C-LTO showing that no carbon black remains on the surface after annealing. Raman features at *ca.* 1250 and 1520 cm^{-1} arise from the background.

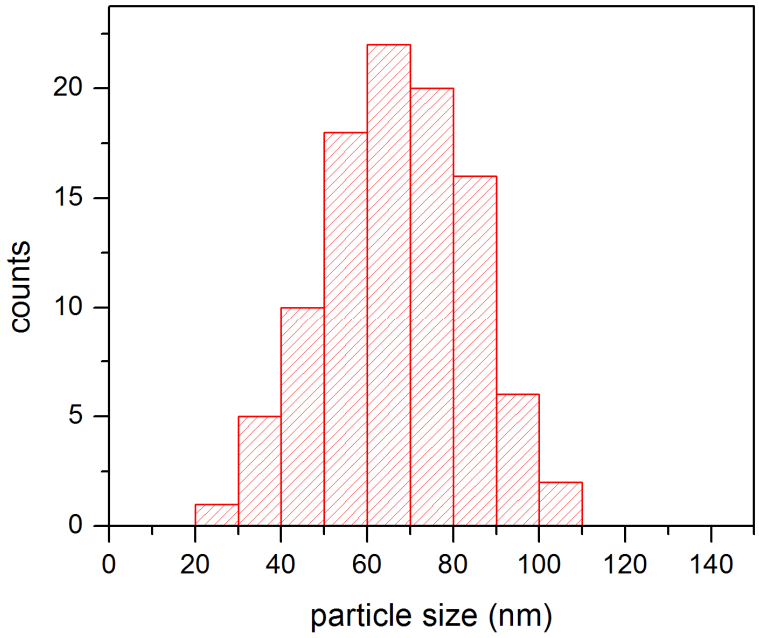

Figure S4. Particle size distribution histogram of C-LTO.

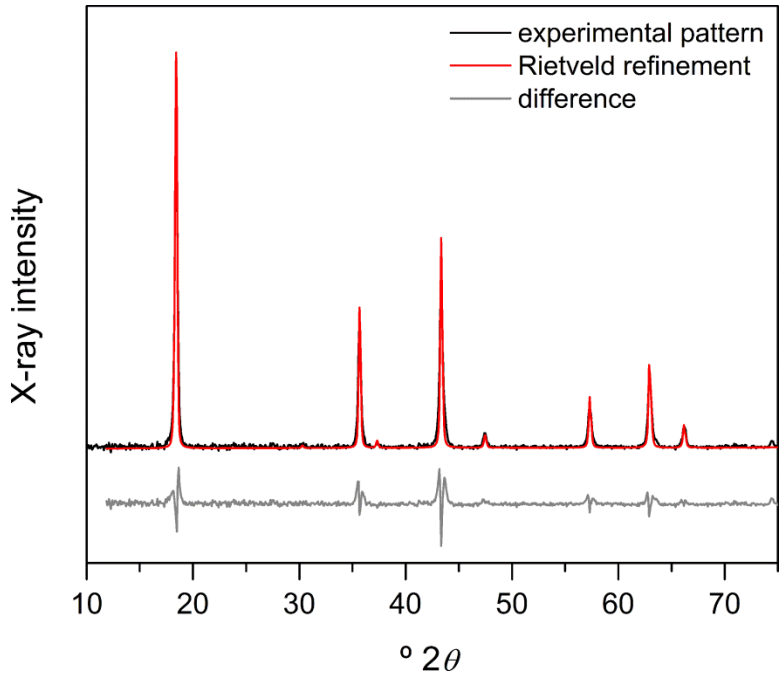

Figure S5. X-ray diffraction pattern and Rietveld refinement of C-LTO nanocrystals.

Table S1. Refinement parameters for C-LTO nanocrystals

Space Group	$Fd\bar{3}m$
Scale	4.896×10^{-4}
Lattice parameter, *a* (Å)	8.362
Unit Cell Mass (g•mol^{-1})	1439.529
Unit Cell Volume (Å^3)	584.654
Crystallite Size, Lorentzian (nm)	58.01
Crystal Density (g•cm^3), calculated	4.089
Crystal Linear Absorption Coefficient (cm^{-1})	452.192
Wt%-Rietveld	100.00
R_{exp}/ R_{exp}' (*a*)	2.81/3.69
R_{wp}/ R_{wp}'	2.94/1.49
R_p/ R_p'	2.26/1.70
R_{Bragg}	0.521
GoF	2.48
DW_d	0.78

(*a*)-Primed parameters are background corrected.

Table S2. Atomic coordinates and isotropic thermal parameters for C-LTO nanocrystals

Atom	Wyckoff Site	*x*	*y*	*z*	SOF	B_{eq}
Li	8*a*	0	0	0	1	6.131
Li	16*d*	0.6250	0.6250	0.6250	0.167	3.319×10^{-4}
Ti	16*d*	0.6250	0.6250	0.6250	0.833	7.082
O	32*e*	0. 38867	0. 38867	0. 38867	1	–0.9752

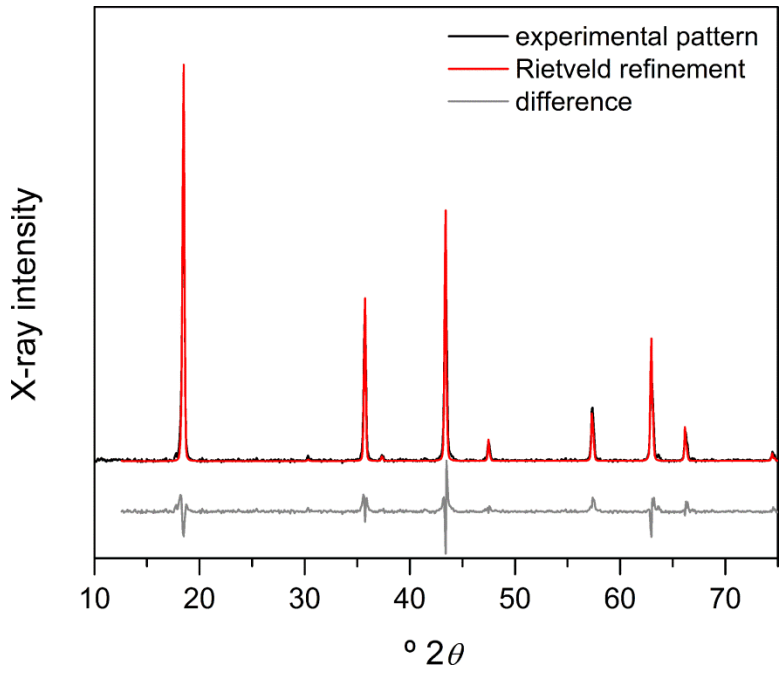

Figure S6. X-ray diffraction pattern and Rietveld refinement of S-LTO aggregates.

Table S3. Refinement parameters for C-LTO nanocrystals

Space Group	$Fd\bar{3}m$
Scale	4.315×10^{-4}
Lattice parameter, *a* (Å)	8.363
Unit Cell Mass (g•mol^{-1})	1439.529
Unit Cell Volume (Å^3)	585.458
Crystallite Size, Lorentzian (nm)	201.3
Crystal Density (g•cm^3), calculated	4.083
Crystal Linear Absorption Coefficient (cm^{-1})	451.571
Wt%-Rietveld	100.00
R_{exp}/ R_{exp}' (*a*)	1.34/2.07
R_{wp}/ R_{wp}'	1.54/2.49
R_p/ R_p'	3.16/2.67
R_{Bragg}	0.775
GoF	2.15
DW_d	0.83

(*a*)-Primed parameters are background corrected.

Table S4. Atomic coordinates and isotropic thermal parameters for C-LTO nanocrystals

Atom	Wyckoff Site	*x*	*y*	*z*	SOF	B_{eq}
Li	8*a*	0	0	0	1	4.016
Li/Ti	16*d*	0.6250	0.6250	0.6250	0.167	-8.246
Ti	16*d*	0.6250	0.6250	0.6250	0.833	7.082
O	32*e*	0. 38587	0. 38587	0. 38587	1	–3.645

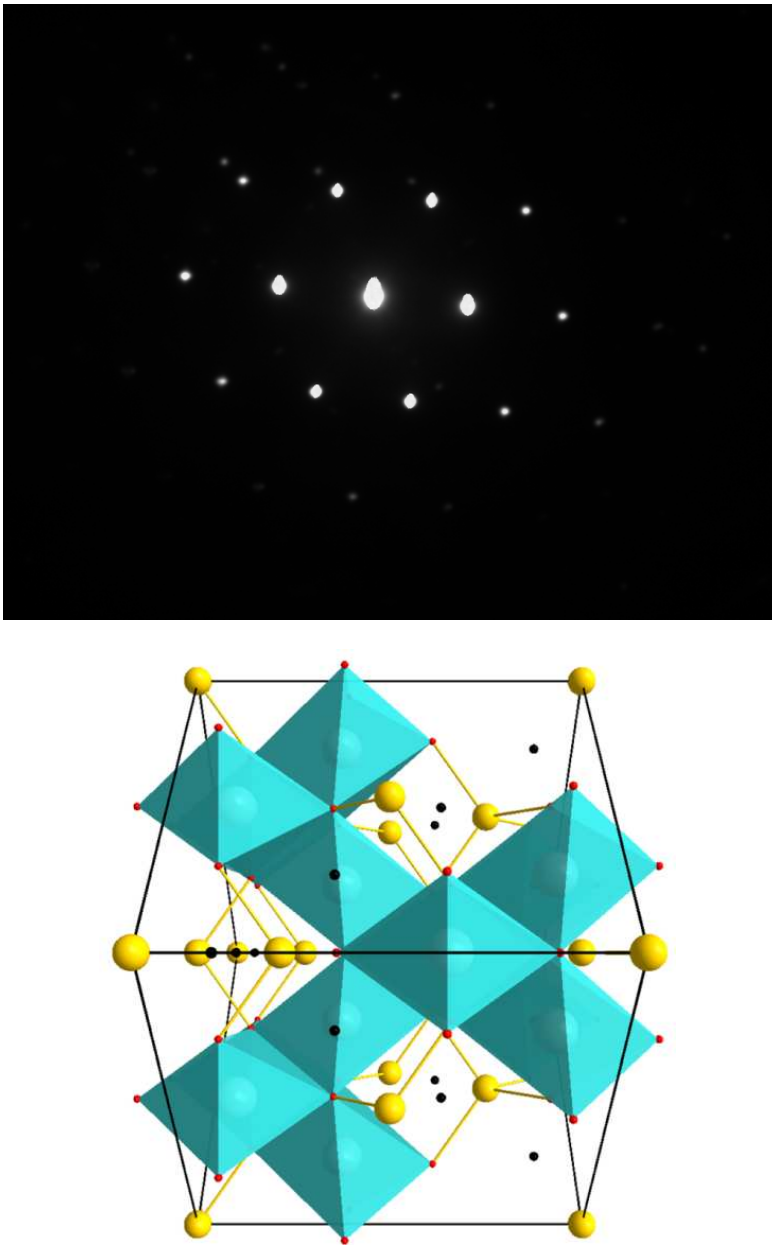

Figure S7. SAED pattern of C-LTO nanocrystals (top). The view is along [011], and diffraction spots correspond to the {111} family of planes. Unit cell of $Li_4Ti_5O_{12}$ viewed along [011] (bottom). Yellow spheres represent Li on 8*a* tetrahedral Wyckoff sites, light blue polyhedra represent $(Li/Ti)O_6$ octahedra on 16*d* sites, and black spheres represent the 16*c* octahedral sites. These sites are empty in $Li_4Ti_5O_{12}$, but occupied in the fully lithiated material $Li_7Ti_5O_{12}$.

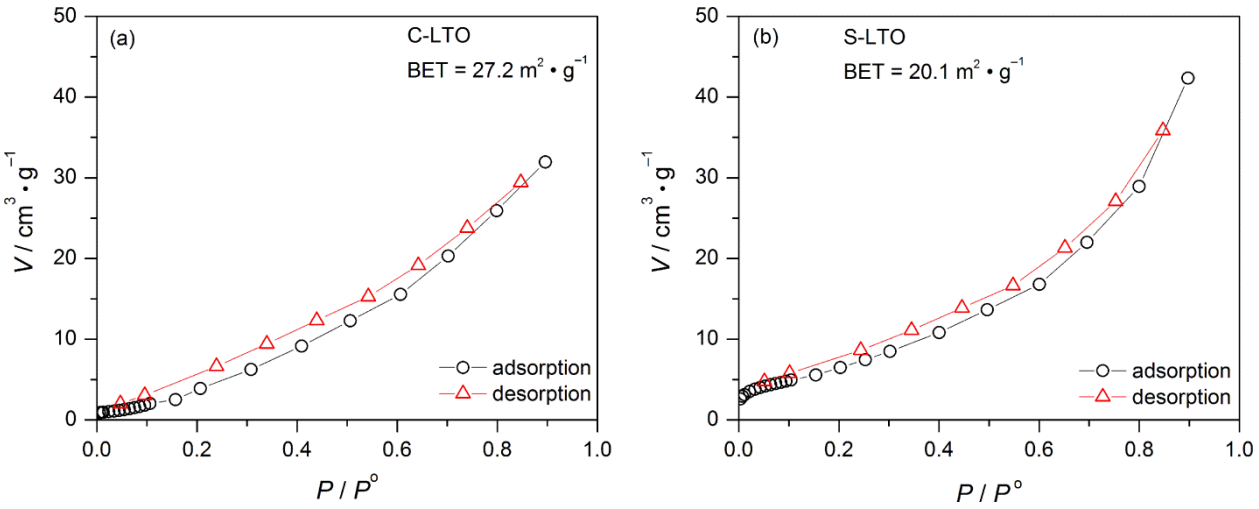

Figure S8. N_2 sorption isotherms for C-LTO (a) and S-LTO (b).

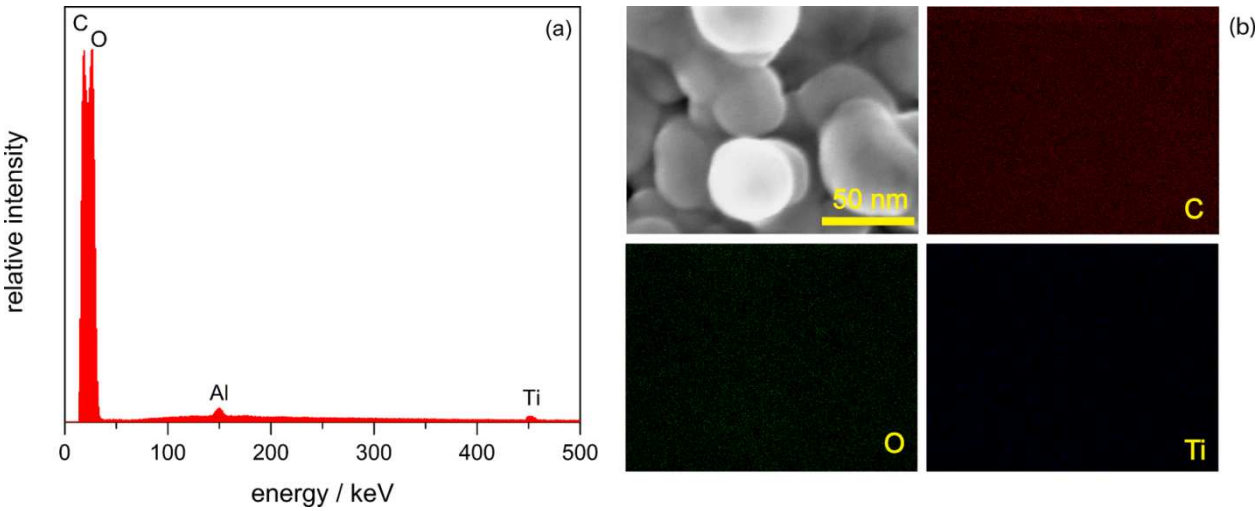

Figure S9. EDX spectrum (a) and map (b) of C-LTO nanocrystals.

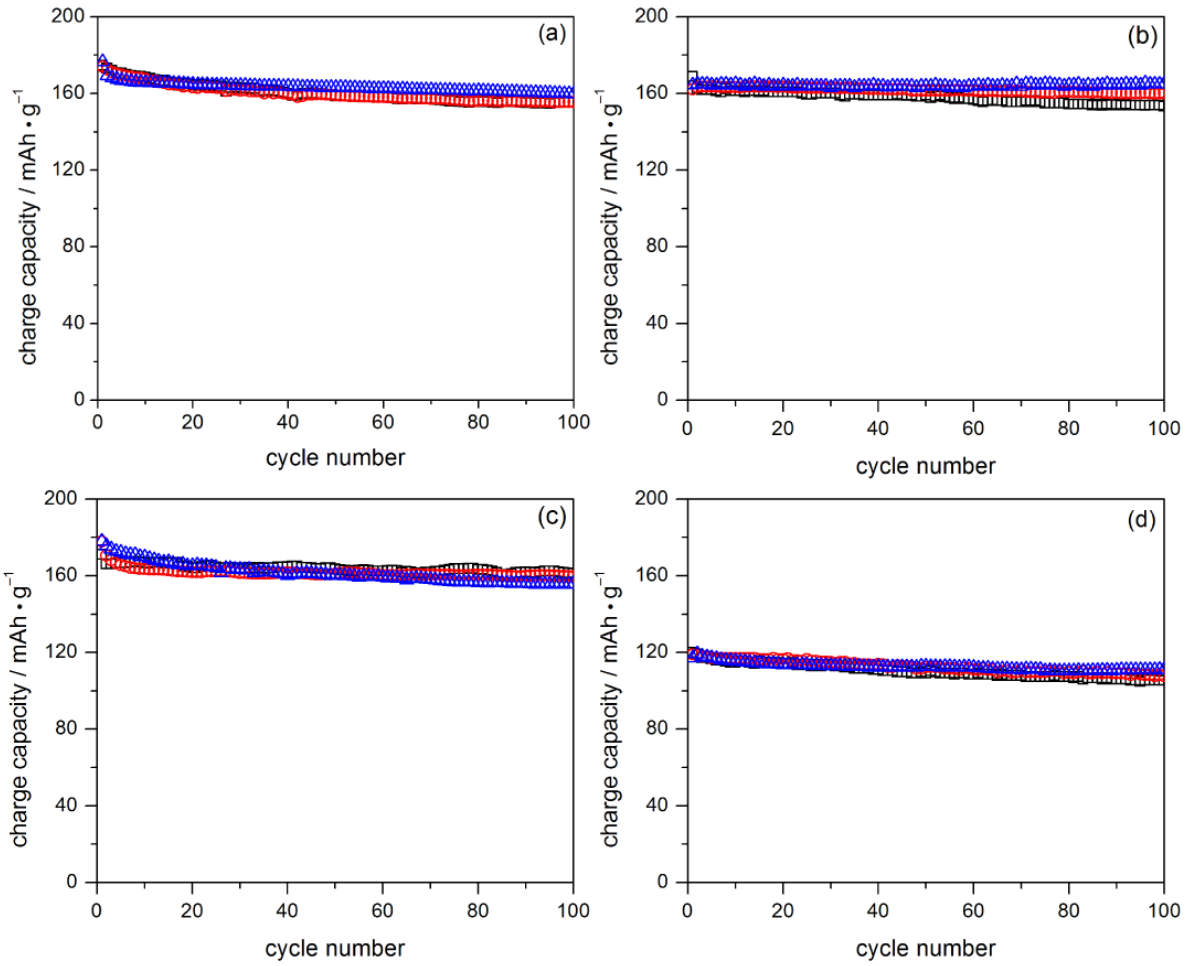

Figure S10. (a) Gravimetric charge capacity of C-LTO cells cycled galvanostatically at 1 *C;* (b) Gravimetric charge capacity of C-LTO cells cycled galvanostatically at 10 *C;* (c) Gravimetric charge capacity of S-LTO cells cycled galvanostatically at 1 *C;* (d) Gravimetric charge capacity of C-LTO cells cycled galvanostatically at 10 *C.*

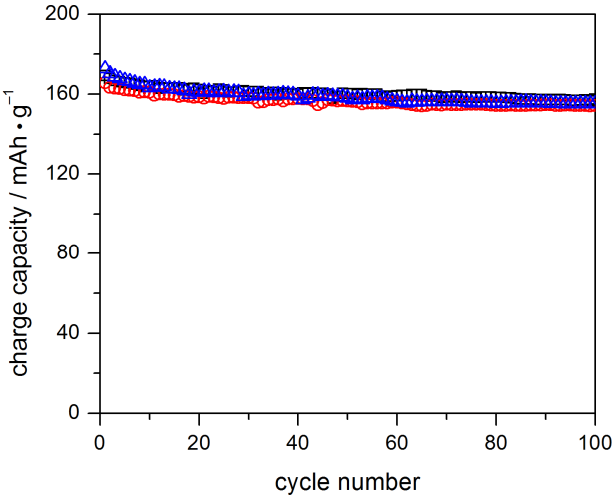

Figure S11. Gravimetric charge capacity of C-LTO cells (4mg/cm^2 active material loading) cycled galvanostatically at 1 *C*.

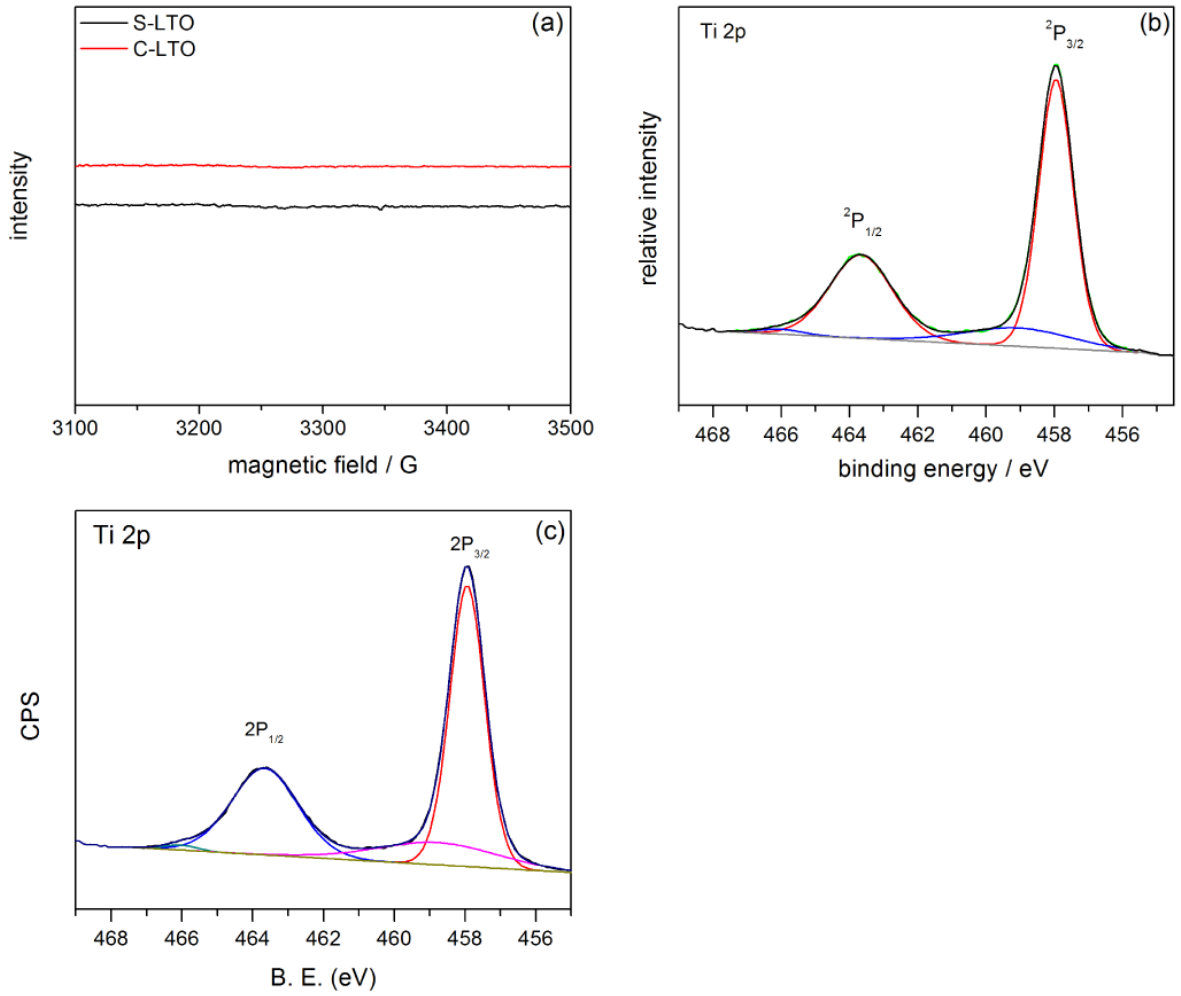

Figure S12. EPR spectrum (a) , Ti 2p XP spectrum of C-LTO nanocrystals (b) Ti 2p XP xpectrum of S-LTO nanocrystals.