The low-temperature chemical synthesis of an LTO based material for Li-ion batteries

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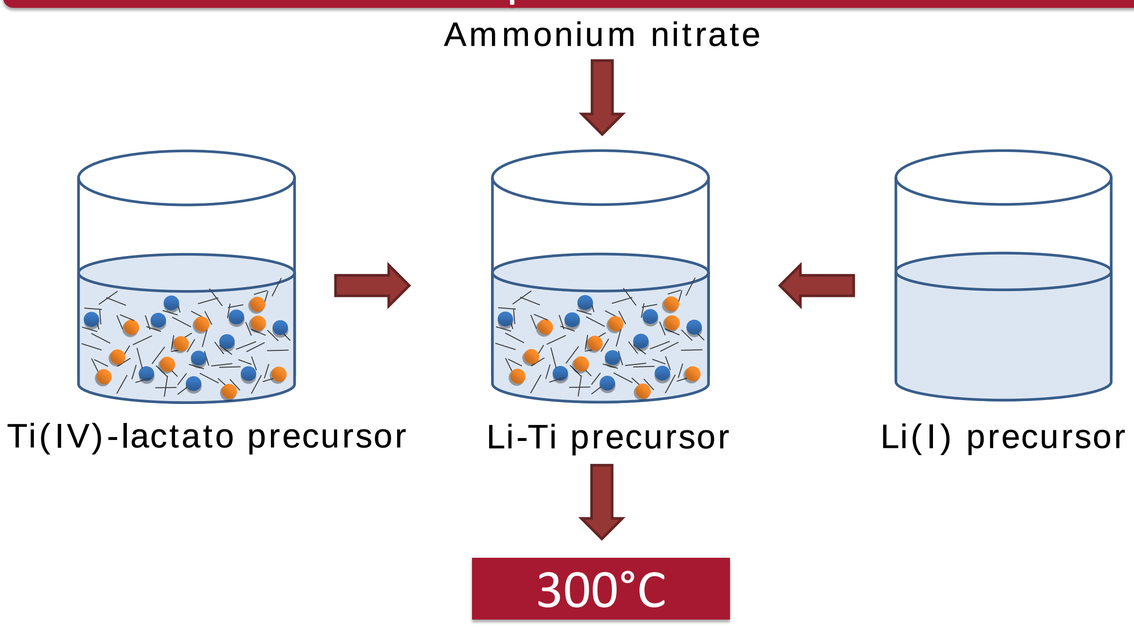
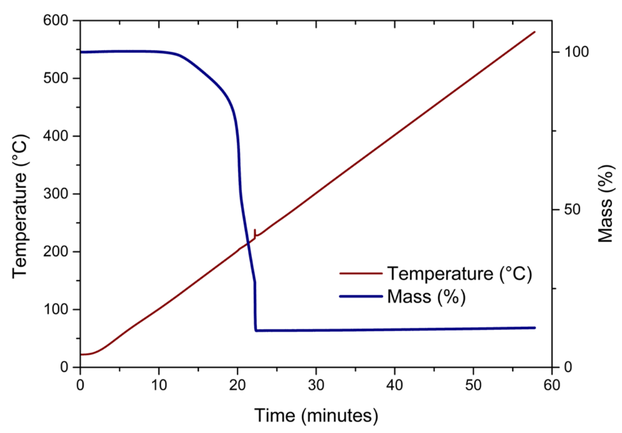
Introduction

- Lithium ion batteries lead the current rechargeable battery market because of their high energy and power density.
- Spinel $Li_4Ti_5O_{12}$ (LTO) is a promising anode material because of its high stability and safety.
- Reported synthesis methods employ high temperatures (~800°C) and long calcination times (3-24h).
- In the combustion synthesis method, a precursor is heated to a relatively low process temperature, where the precursor ignites and generates the necessary energy for conversion and crystallization to the desired oxide.
- This study presents a new approach to the combustion synthesis method for the preparation of LTO. Optimizing the process parameters allows to synthesize LTO at a temperature as low as 300°C.

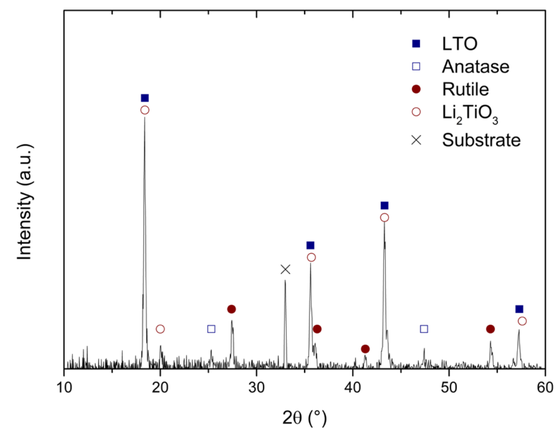
Experimental



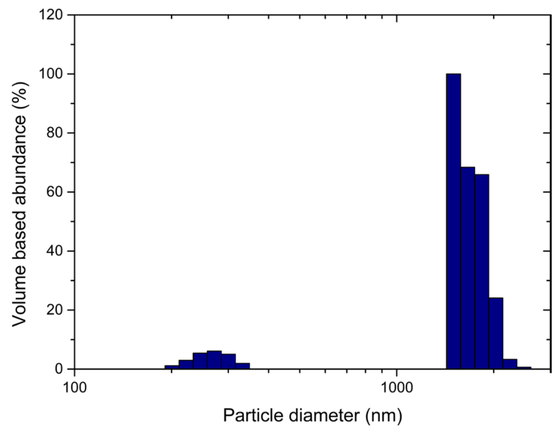
Results



TGA: a sudden mass loss reaction occurs at 223°C when the precursor is heated under O_2. There is no further mass loss at higher temperatures. The exothermicity of the reaction is evidenced by the sudden increase of the observed temperature.

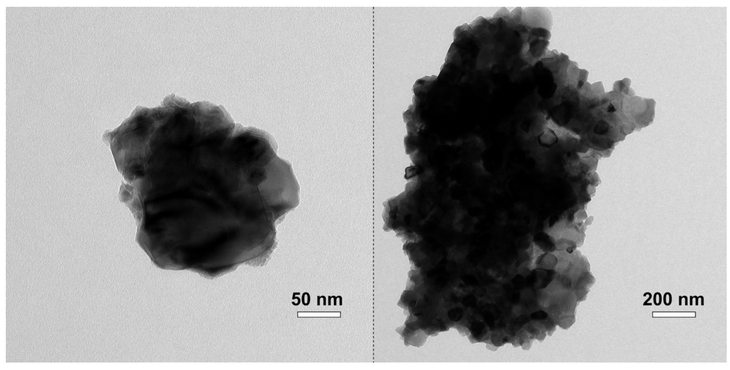


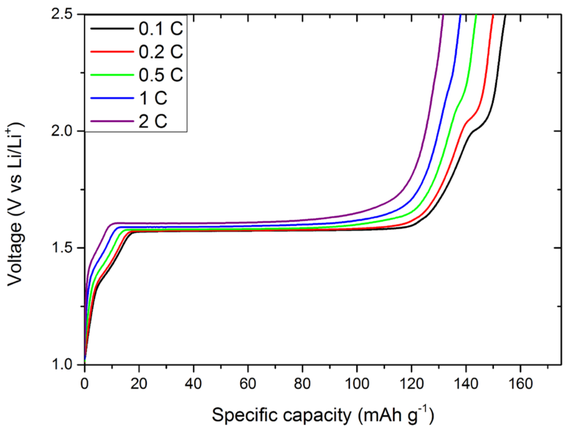
XRD: shows that LTO was formed (with impurities). Anatase is an electrochemically active impurity.

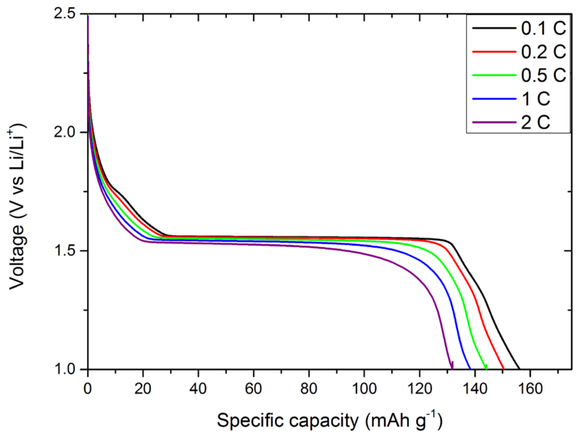


DLS: Submicron sized particles with a size of 200-300nm are present, along with bigger particles of 1-2µm.

TEM: The submicron and micron sized particles are in fact agglomerates of nanoparticles, resulting in a rough surface morphology



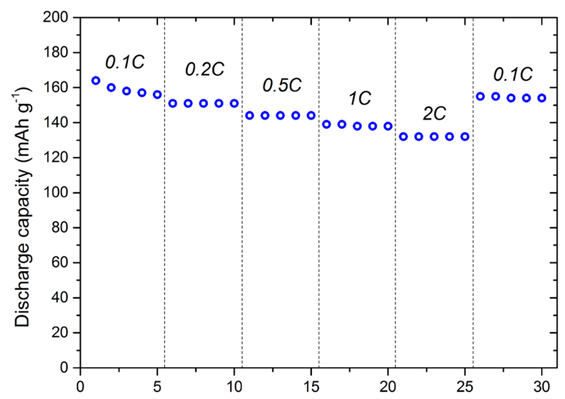


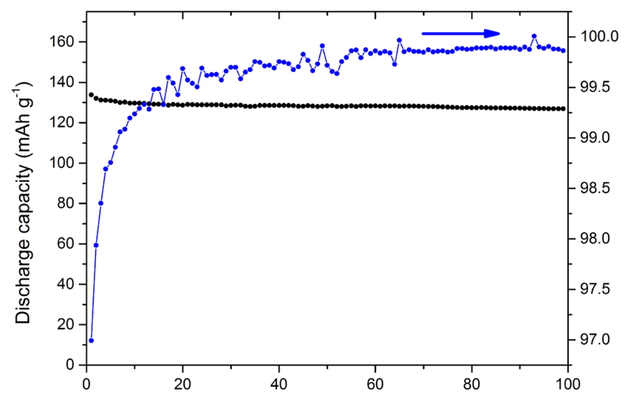


Galvanostatic cycling: a voltage plateau at 1.55V, which is characteristic for LTO, is prominent at every current density, which proves that LTO is the main electrochemically active material present in the sample. At lower C-rates, a voltage plateau at approximately 2V appears which can be related to anatase. With an increasing current density this plateau becomes smaller while shifting to higher potentials, ultimately disappearing at 2C.

In the first cycle at 0.1C, the material has a discharge capacity of 164mAh g^{-1} (theoretical capacity of 175mAh g^{-1}). In the fifth cycle, the capacity drops to 156mAh g^{-1}. At 2C, the material still shows a discharge capacity of 132mAh g^{-1}.

After 99 cycles at 1C, the material shows 94.9% of its discharge capacity of the first cycle. The coulombic efficiency of the first cycle is 97.0% and after 50 cycles, it never falls below 99.7% and values of 99.9% are common.





Conclusions

- An LTO based anode material was synthesized at a lower temperature and with shorter processing time than conventional synthesis methods (300°C for minutes compared to 800°C for hours).
- High energy costs are avoided.
- The product consisted of crystalline LTO with impurities.
- The material had an initial discharge capacity of 164mAh g^{-1} (175mAh g^{-1} theoretical capacity) at 0.1C. 85% of the capacity was retained when increasing the current twentyfold.
- Combustion synthesis is a viable method of synthesizing a performant LTO based material at a low temperature.

References

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