Synthesis of cathode materials for lithium-ion batteries by continuous micro-flow spray pyrolysis

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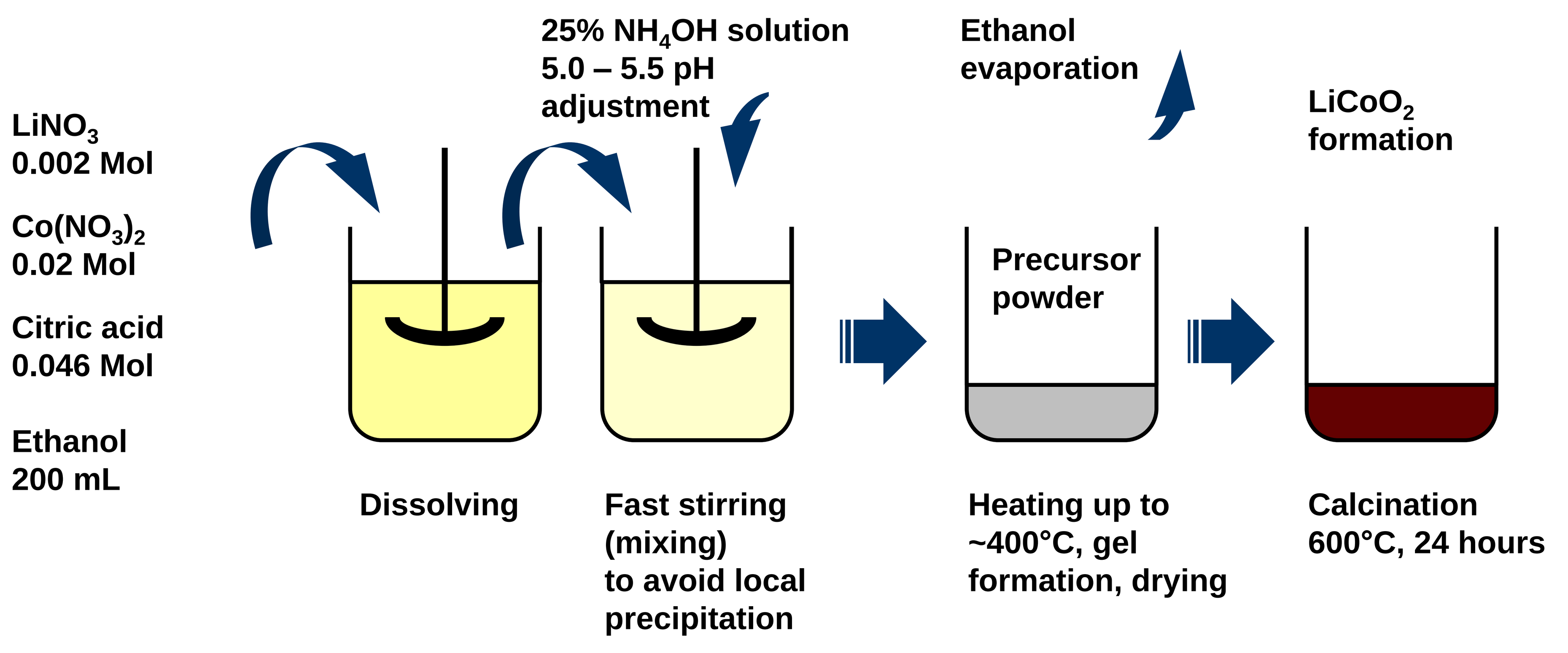
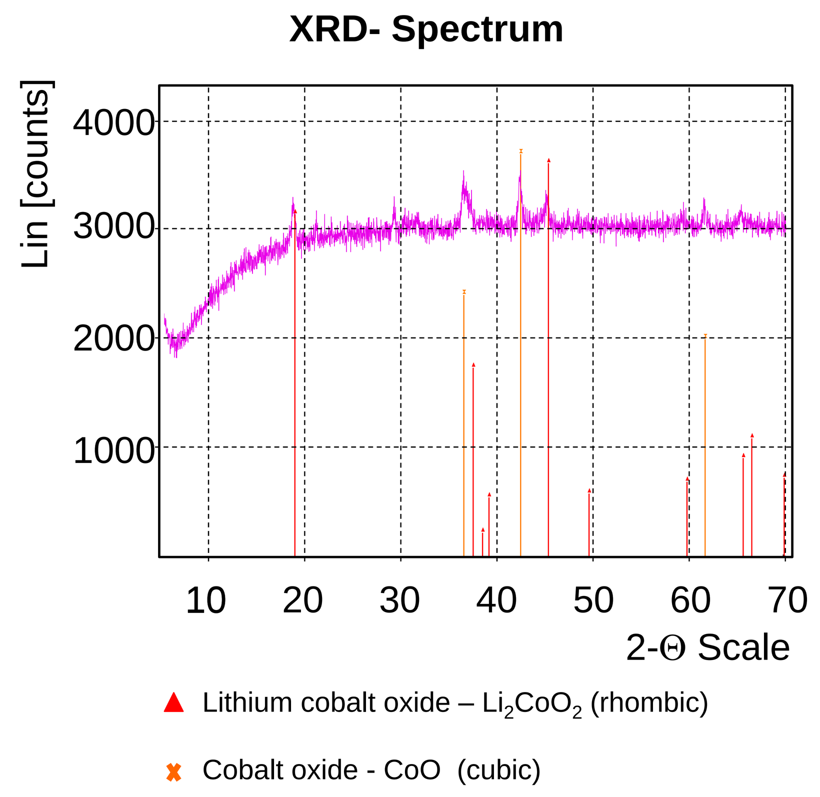
Introduction

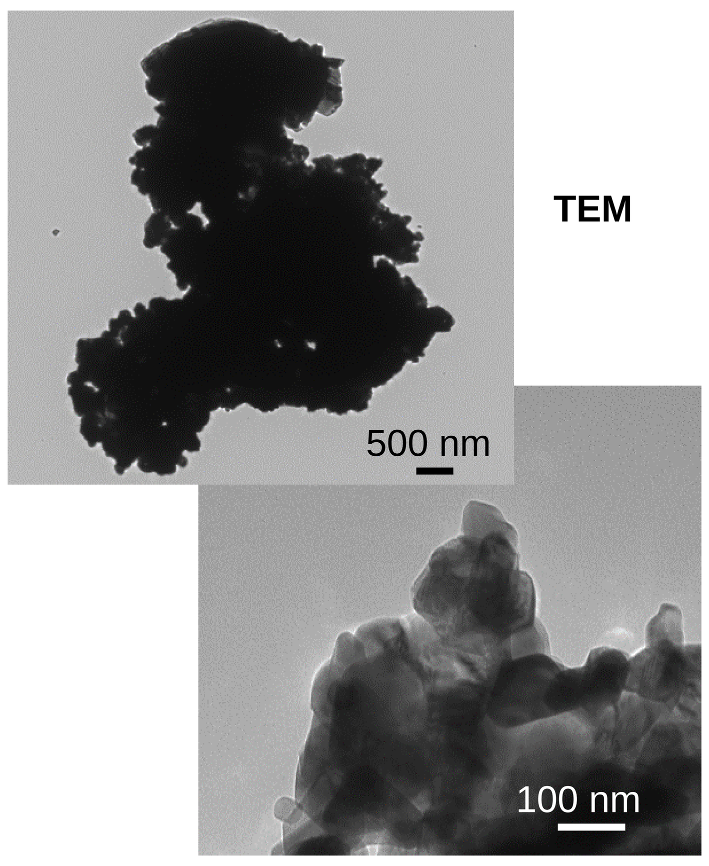
Li-ion batteries provide many advantages compared to traditional rechargeable systems (Pb-acid and Ni-MH), such as its higher energy density, lower self-discharge, long capacity cycle life and relatively maintenance free.[1-4] Due to the commercial advantages, a lot of research is done in developing new Li-ion electrode materials and improving existing ones. The most common and simplest synthesis method is the mixing of powders in their solid-state form and heating them to relatively high temperatures over long periods of time. Traditional batch methods of synthesizing the electrode material is costly and do not necessarily provide optimized electrochemical performance. Alternative continuous less energy intensive methods would help reduce the costs on the preparation of the electrode materials. In general, the spray pyrolysis method showed suitable cathode oxide materials are formed in shorter periods of time, resulting in small homogenous particles with narrow particle size distribution.[1] Other synthesis methods include the formation of sol-gel products, that are further heat-treated or directly used with a spray pyrolysis method which in turn would produce the desired cathode oxide material.

As a proof of concept this study investigated the synthesis of cathode oxide materials suitable for lithium-ion batteries using new continuous operating micro-flow processes. This new processes consist of fast micro-mixing of the starting materials to form a gel continuously or skipping the intermediate gel formation step by immediate continuous spray pyrolysis after mixing.

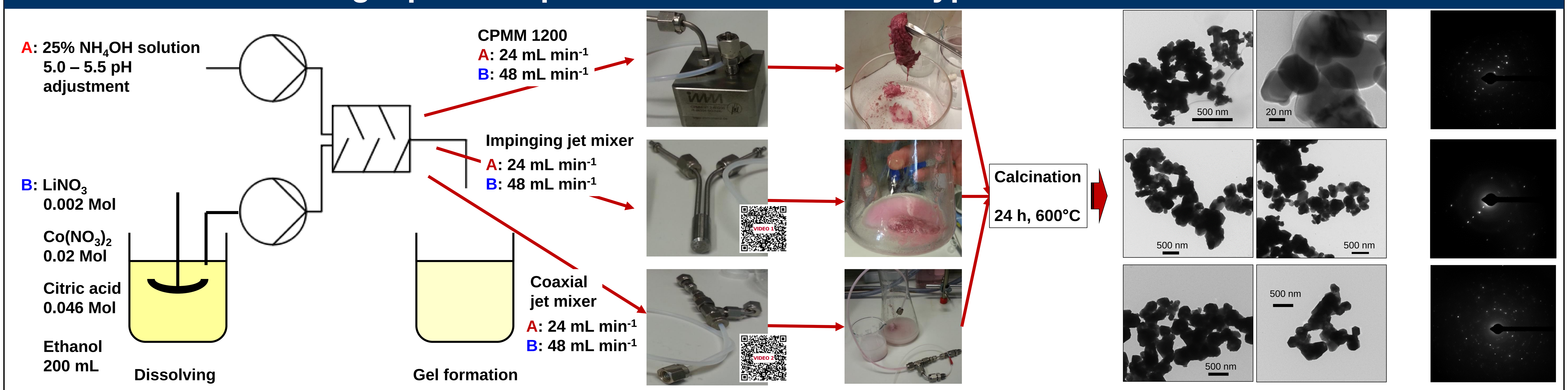
Batch synthesis of $LiCoO_2$ nano-particles



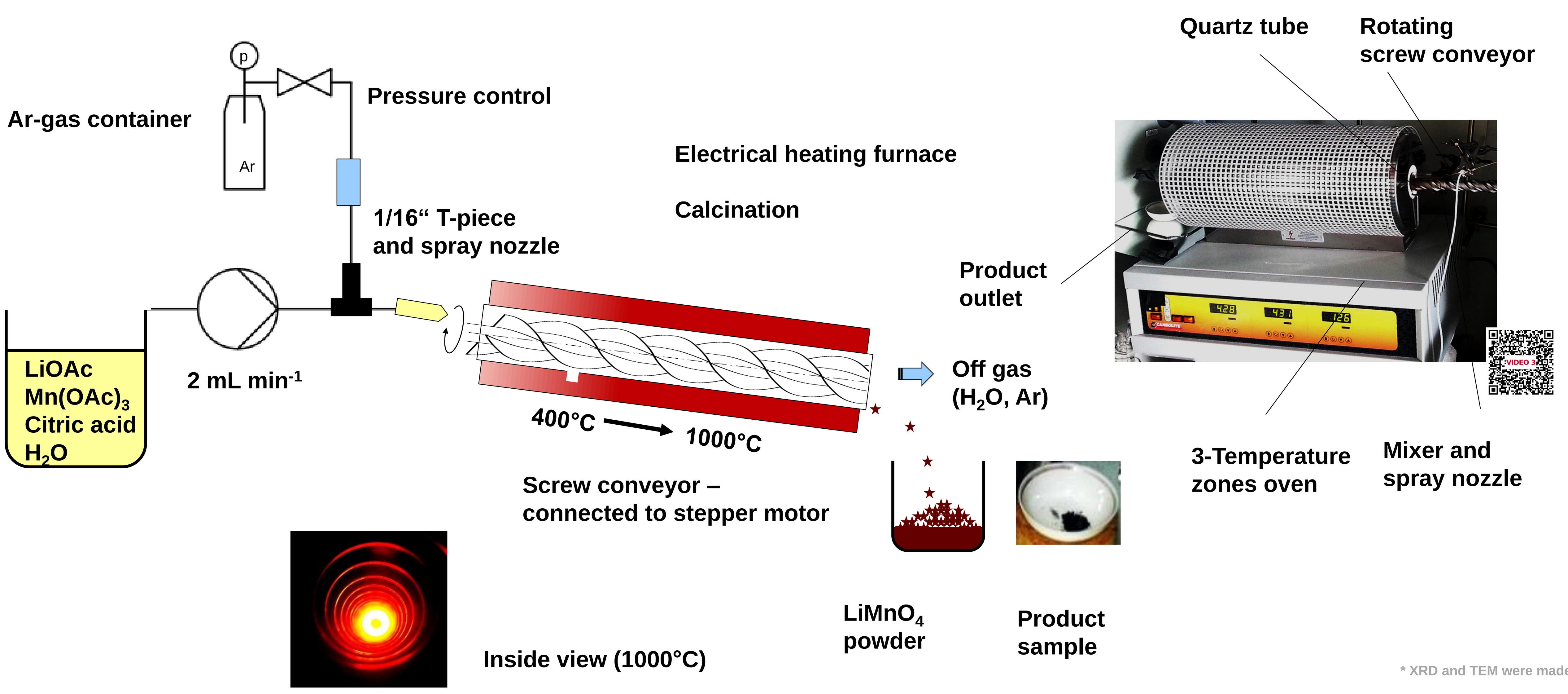


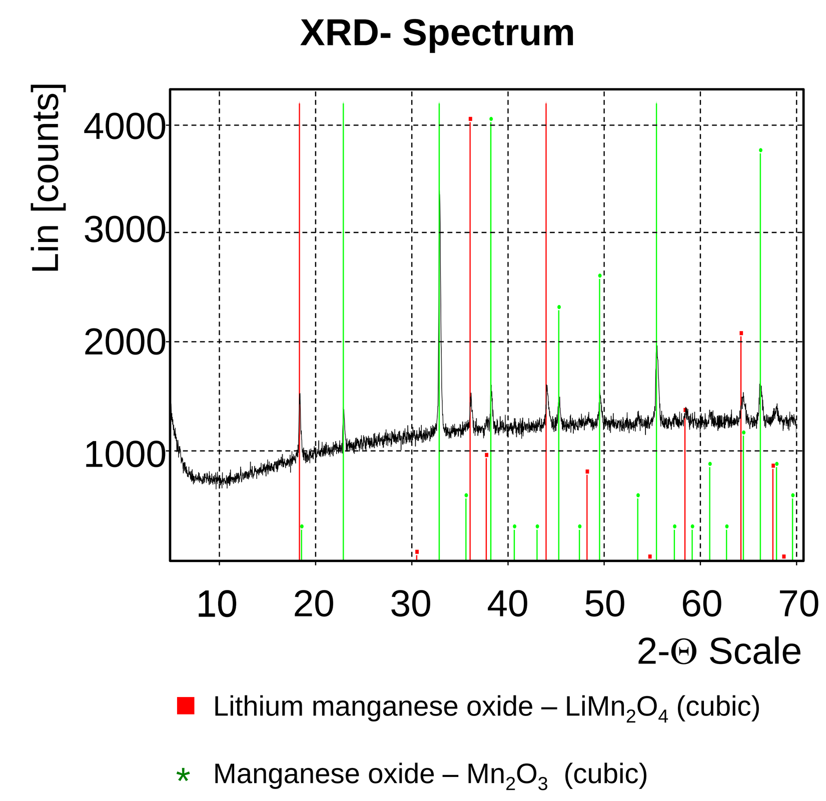


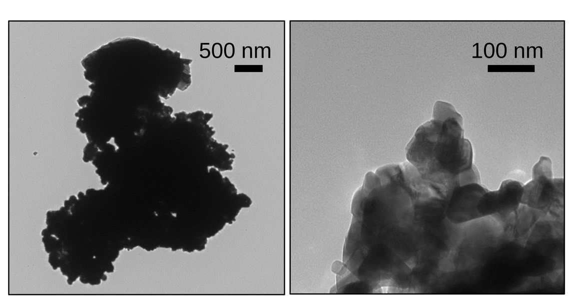
Continuous-flow sol-gel process performed with different types of micro mixers



Continuous-flow design of spray pyrolysis – bypassing a separate sol to gel formation







* XRD and TEM were made from different samples

Summary

- By using fast performing static mixers, e.g. different micro mixers (caterpillar micromixer, impinging jet mixer (both IMM) and coaxial jet mixer (JGU)) the time for sol to gel formation can be significantly reduced (see video sequence). After removing of the solvent, the gels were calcined at temperatures about 600°C for several hours to achieve crystalline $LiCoO_2$ nano particles .
- Due to the very high heat capacity, a conventional full steel hammer drill was used as the rotating screw. Induced heat fluctuations of the relatively cold carrier gas could be suppressed. The injection of the premixed reactant was done as spray at the upper end of the quartz glass tube, which allows the solvent to evaporate. The formed powder is the precursor for the $LiCoO_2$ crystals. .
- The powder X-ray diffraction pattern showed a crystalline material was produced that corresponded to the typical spinel material. There were some impurities present, which were noted on the diffraction pattern to be between 35 and 45 °2θ and no distinct separation between the two peaks at 65 ° 2θ. Impurities observed within this analysis setup resulted from possible contamination from the quartz glass rod and the steel screw..

References

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