APPLICATION NOTE
LITHIUM MANGANESE PHOSPHATE

Particle Size Distribution according to ISO 13318-1 and ISO 13318-2

Introduction

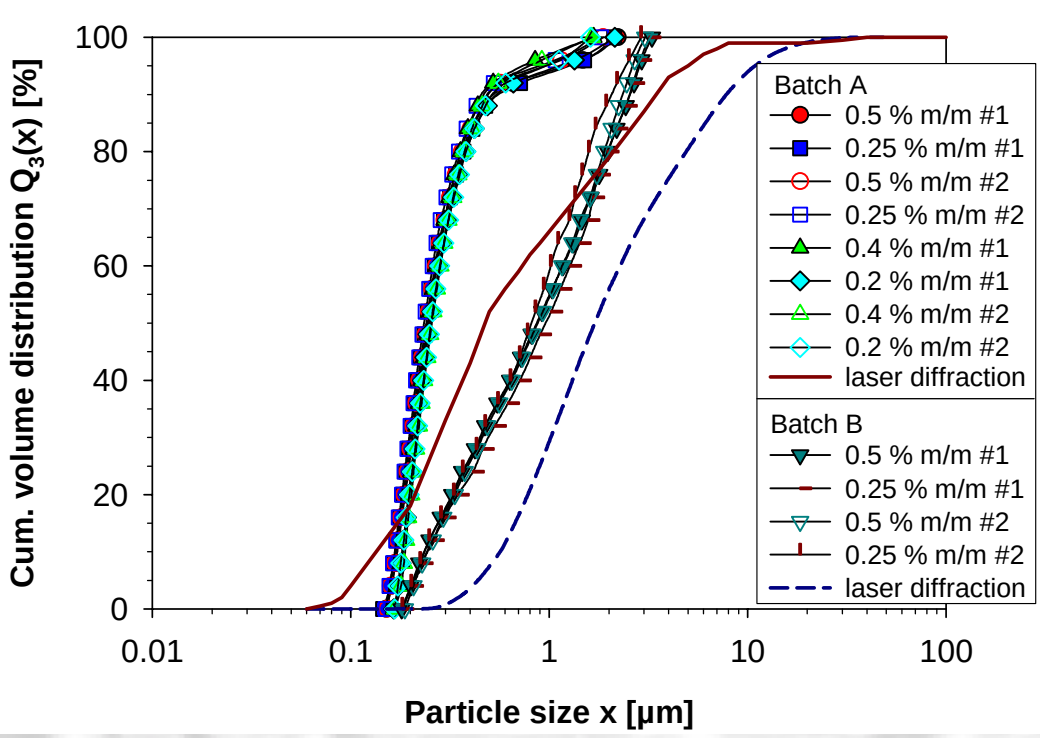
Lithium ion batteries are the device of choice to power consumer electronics. Conflicting demands for more power versus lifetime drives the need for improved materials and performance in each of the components making up a rechargeable battery. Due to its high theoretical capacity and stable phospho-olivine structure Lithium manganese phosphate ($LiMnPO_4$) is a promising candidate for application as cathode material.
Two different powders of Lithium manganese phosphate (Batch A and Batch B) were investigated in respect of particle size distribution to obtain information about the aggregation state. The particle size distribution is obtained by multisample analytical centrifugation based on STEP® -Technology.

Experimental

The $LiMnPO_4$ powders batch A and batch B were dispersed into ultra pure water to concentrations of 0.20 %, 0.25 %, 0.40 % and 0.50 % m/m. The samples were treated with an ultrasonic homogenizer directly after suspending and continuously stirred with a magnet stirrer. Before measurement the diluted suspensions were treated in an ultrasonic bath.
To investigate the variation in the particle size distribution of batch A as a function of the dispersion intensity an additional measurement was performed. The measurement with the Dispersion Analyser LUMiSizer® was conducted at 25°C at a constant centrifugal velocity or by a stepwise increase in velocity to cover the entire particle range.

Comparison of LUMiSizer data with laser diffraction data

The particle size distributions for the $LiMnPO_4$ suspensions measured with the LUMiSizer® were calculated using the method “Constant Position”. The particle size distributions of batch A and batch b for different dilutions and repetitions (#1 and #2) obtained using a LUMiSizer® are displayed below and compared to two laser diffraction measurements.



Particle size distribution of the $LiMnPO_4$ particles in water obtained with the LUMiSizer and laser diffraction [1] for different concentrations and repetitions.

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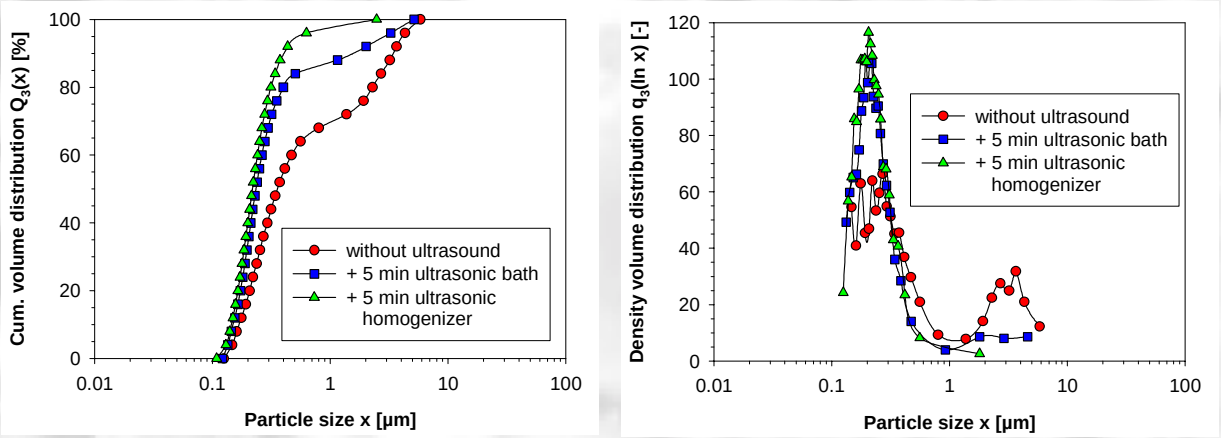
The repetitions and the different dilutions measured with the LUMiSizer® for each $LiMnPO_4$ suspension prove the excellent reproducibility. The median of batch A is about 0.25 µm and of batch B about 0.9 µm. The data of laser diffraction are shifted to larger particle sizes for both samples. The $LiMnPO_4$–powder is heterogeneous and strongly agglomerated. Therefore, the different distributions measured with two techniques are reasonable because laser diffraction measures the diffraction volume on the projected surface, while sedimentation gives a hydrodynamic diameter. Laser diffraction cannot measure particles below 1 µm so accurately because there is less diffraction (almost scattering which cannot be detected well by a classical laser diffraction measurement setup). During calculation with Mie-Theory a bimodal distribution can be virtually created (batch A laser diffraction), because the extinction coefficient has a maximum around 1 µm.

Influence of ultrasonic sample treatment

The following figure represents the results for different sample preparations before measurement.



Cumulative volume distribution Density volume distribution

Particle size distribution of the LiMnPO4 particles in water obtained with the LUMiSizer

With increasing dispersing intensity the particle size distribution becomes narrower and the content of coarse particle decreases. The minimum particle size remains the same. The maximum particle size is smallest after treatment with the ultrasonic homogenizer.

References

- Particle Size Distribution by Space or Time Dependent Extinction Profiles obtained by Analytical Centrifugation (concentrated systems), T. Detloff, T. Sobisch, D. Lerche, Powder Technology 174 (2007), 50–55
- Particle Size Distribution by Space or Time Dependent Extinction Profiles obtained by Analytical Centrifugation, T. Detloff, T. Sobisch, D. Lerche, Part. Part. Syst. Charact. 23 (2006), 184-187
- STEP-Technology see www.lum-gmbh.com/pages/technology.htm
- [1] N. H. Kwon, Laboratory of Powder Technology, EPFL, CH-1015 Lausanne, Switzerland, private communication