

THE Glatt POW(D)ER SYNTHESIS

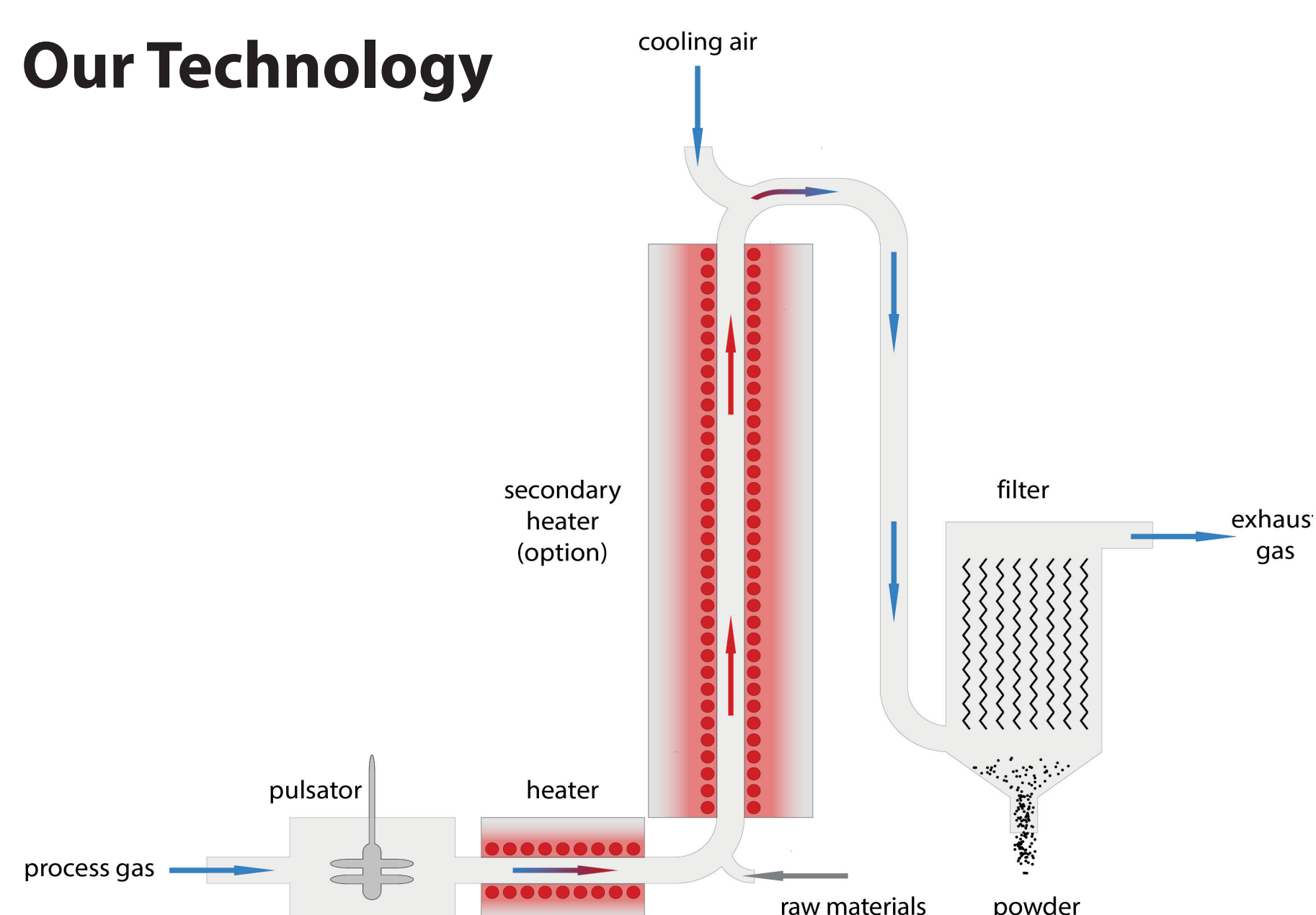
Tailored Cathode Materials in Sodium-Ion Batteries: From Tunnel-type Oxides to Layered Oxides to Polyanionic Compounds

Introduction

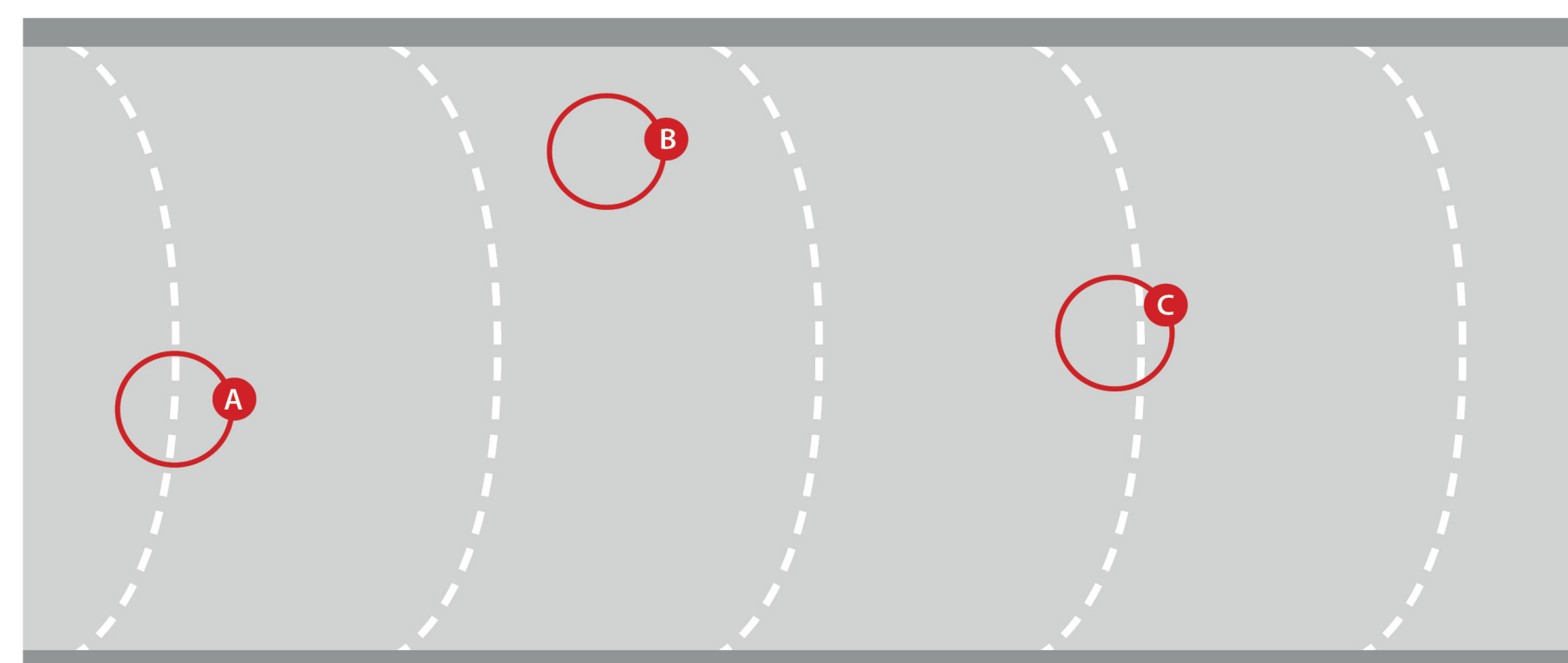
The growing demand for sustainable and secure energy storage solutions has intensified the interest in sodium-ion batteries (SIBs) as a promising alternative to lithium-ion technology. With sodium's natural abundance and wide geographic availability, SIBs offer not only cost-effective and scalable energy storage, but also a strategic opportunity for Europe to reduce dependency on critical raw materials primarily sourced from China. This shift is essential for establishing resilient and autonomous battery value chains within the European Union.

This contribution highlights recent advances in SIB technology, with a particular focus on material synthesis via spray pyrolysis, a scalable technology for producing high-performance electrode materials. Layered oxides and polyanionic compounds are explored as cathode materials, offering stable cycling and high voltage operation. By integrating material innovation with sustainable process engineering, this work underlines how sodium-ion batteries can play a pivotal role in Europe's transition to energy sovereignty and a greener future.

Our Technology



Pulsation makes the difference!



Impact of pulse will create superfine droplets by secondary atomization

Pulsation creates a highly turbulent flow, homogenizing temperature and velocity in the gas stream and constantly changes the position of the particles to equalize the resident times

Impact of pulse and perpetual relative velocity between particle and gas stream will continuously break up boundary layers, guaranteeing high heat and mass transfer

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Further information can be found on our website
powdersynthesis.glatt.com

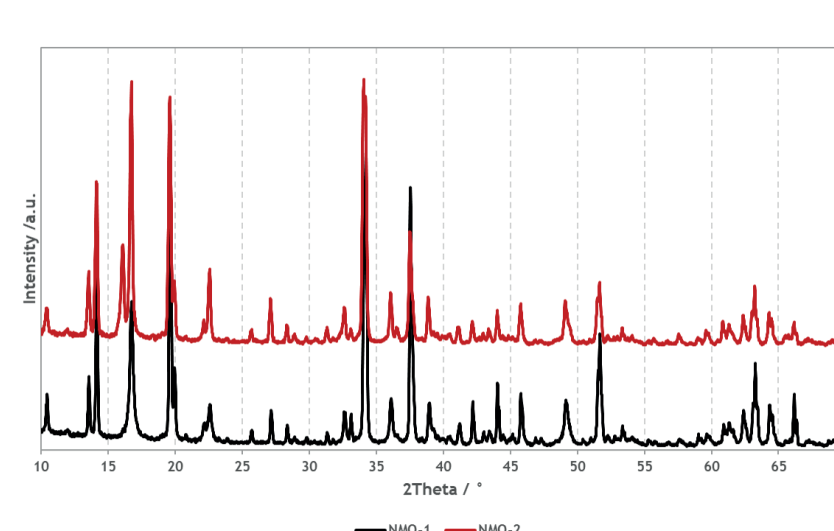


Preparation of the materials

The materials were produced in a two-stage process. Additional steps were applied for coated materials. In general, the synthesis was carried out from prepared solutions or suspensions. For this purpose, the corresponding raw materials were dissolved or suspended in deionized water, whereby the spray solution had a solids content of 20% in relation to the end product. The solutions for the oxidic compounds NMO, P2-NFMO and P2-NFMMO were sprayed at a reactor inlet temperature of approx. 640°C with spray rates of 40-50 g/min and a pulse frequency of 80 Hz. Thermal post-treatment was carried out at 850°C for several hours. The coating of P2-NFMO was carried out by suspending the material in ethanol with the addition of H_3PO_4 . The material was then calcined at 400°C. The NASICON-type NFP, $Na_3Fe_2(PO_4)_3$, was prepared by spraying a solution at an inlet temperature of 250°C with a spray rate of 40 g/min and a pulse frequency of 80 Hz. The resulting powder was then post-processed thermally at 600°C. For the production of NFPP, $Na_3Fe_2(PO_4)_2(P_2O_7)$, additional carbon precursors were used to obtain an appropriate carbon coating. Spraying was carried out at reactor inlet temperatures of 220°C with a spray rate of 30 g/min and a pulse frequency of 80 Hz. Thermal post-treatment was carried out under inert conditions at 500°C.

Results - Example NMO – $Na_{0.44}MnO_2$

Two samples of NMO were prepared using a nitrate combustion method (NMO-1) and a sol-gel-combustion route (NMO-2). Where as NMO-1 show a pure orthorhombic symmetry with space group Pbam, NMO-2 is a composite material of the tunnel-type $Na_{0.44}MnO_2$ and layered $Na_2Mn_3O_7$.



Material	D_{10} μm	D_{50} μm	D_{90} μm	S_{BET} m^2/g
NMO-1	3.95	15	34	1.3
NMO-2	2.23	18	57	0.89

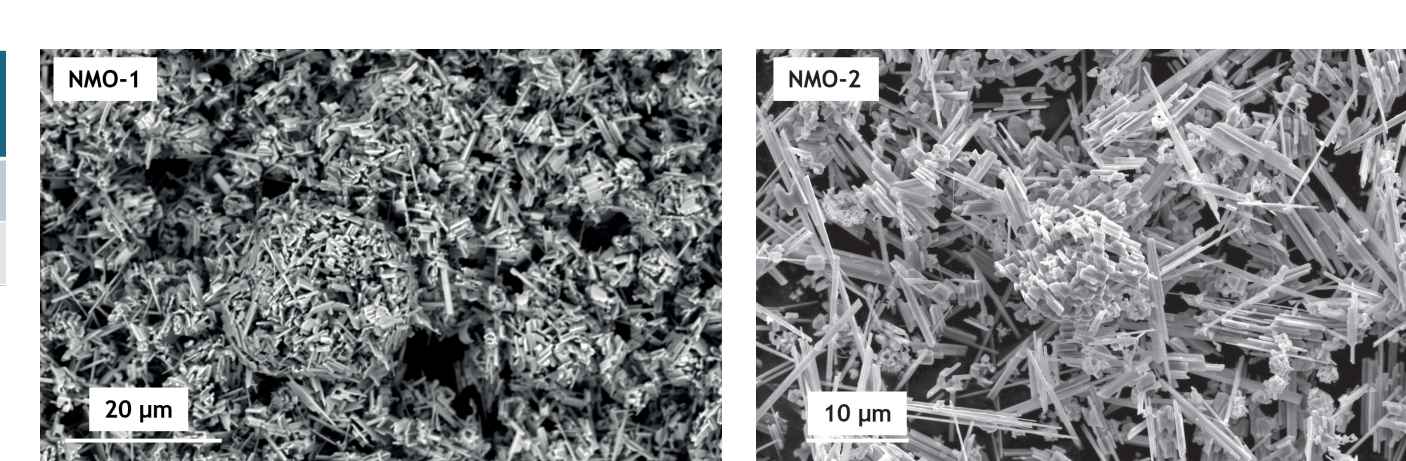
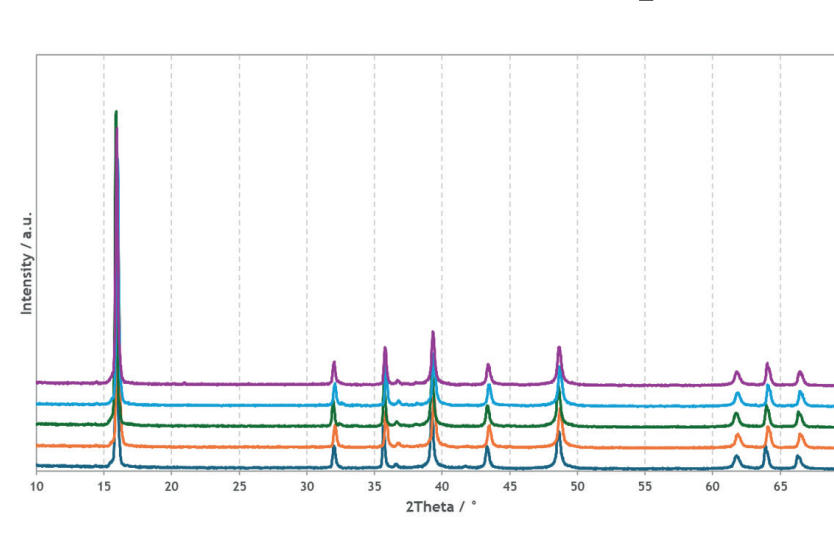


Fig.1 XRD pattern of the prepared NMO-powders.

Results - Example P2-NFMO – $Na_{0.66}Fe_{0.5}Mn_{0.5}O_2$



Material	D_{10} μm	D_{50} μm	D_{90} μm
NFMO as prepared	2.63	10.7	21
NFMO washed	1.56	7.7	16
NFMO + 0.5wt% H_3PO_4	1.29	6.8	15
NFMO + 1.0wt% H_3PO_4	1.67	8	18
NFMO + 2.0wt% H_3PO_4	1.56	7.9	18

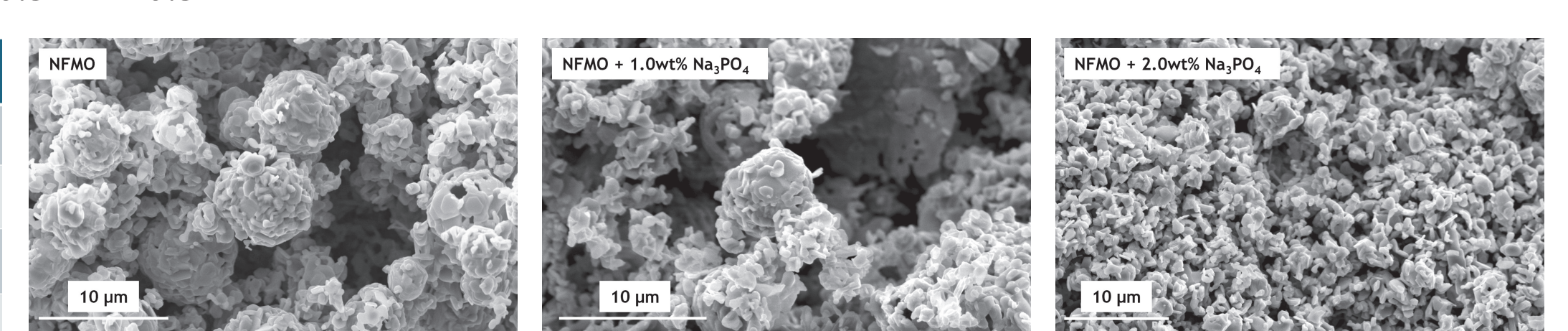
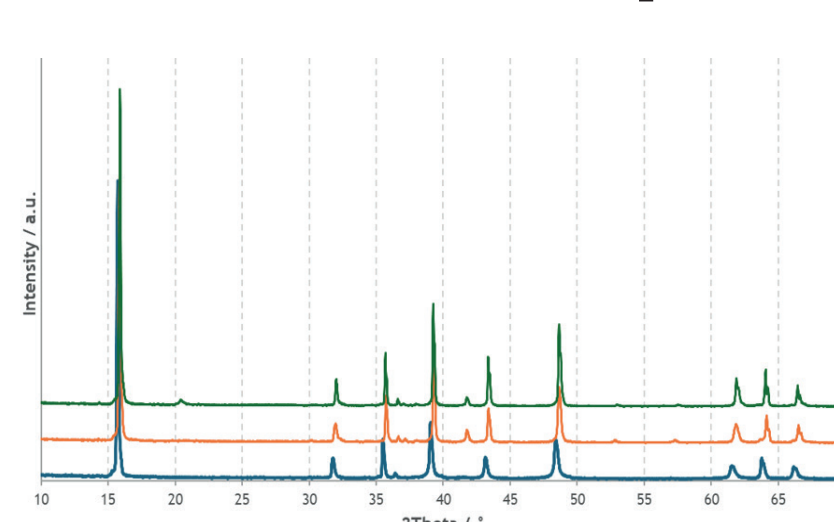


Fig.2 XRD pattern of uncoated and coated P2-NFMO that shows pure pphase material with hexagonal phase with space group $P6_3/mmc$.

Results - Example P2-NFMMO – $Na_{0.67}Fe_{0.4-x}Mn_{0.6}Mg_xO_2$ with $x = 0, 0.1, 0.2$



Material	D_{10} μm	D_{50} μm	D_{90} μm	S_{BET} m^2/g
NFMMO $x=0$	4.19	16	31	1.26
NFMMO $x=0.1$	2.76	8.84	16	1.39
NFMMO $x=0.2$	1.8	6.62	13	1.00

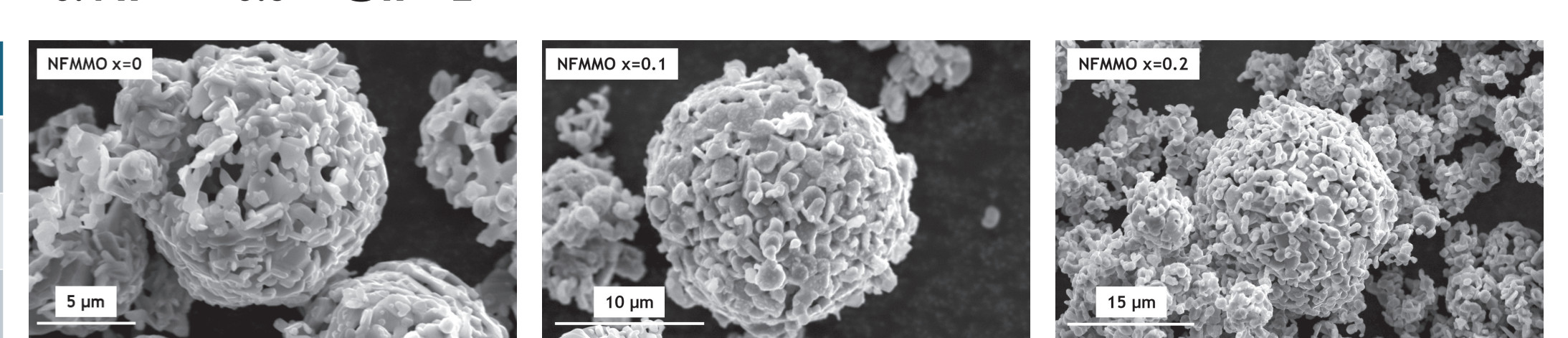
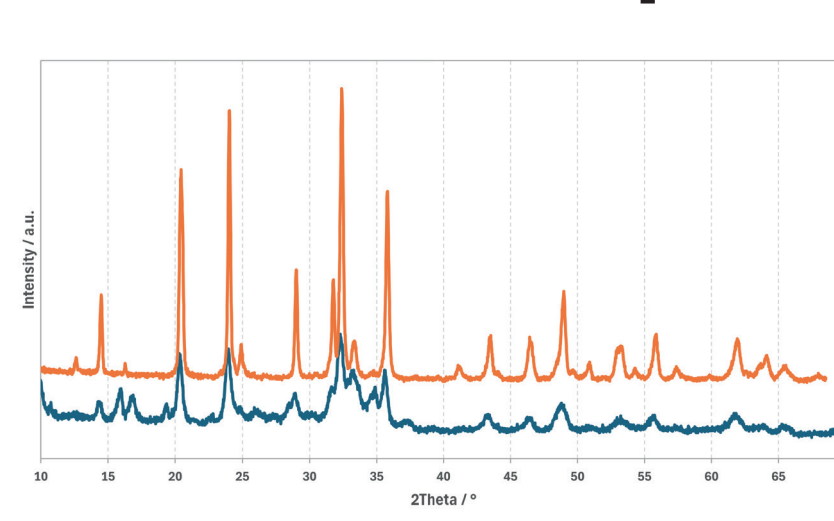


Fig.3 XRD pattern of the prepared $Na_{0.67}Fe_{0.4-x}Mn_{0.6}Mg_xO_2$ oxides. The materials show the typical hexagonal P2-structure with minor side phases of sodium deficient layered oxides, which is a result of an insufficient calcination protocol

Results - Example NASICON-type materials NFPP / NFP



Material	D_{10} μm	D_{50} μm	D_{90} μm
NFPP	8.3	25	51
NFP	4.0	19	36

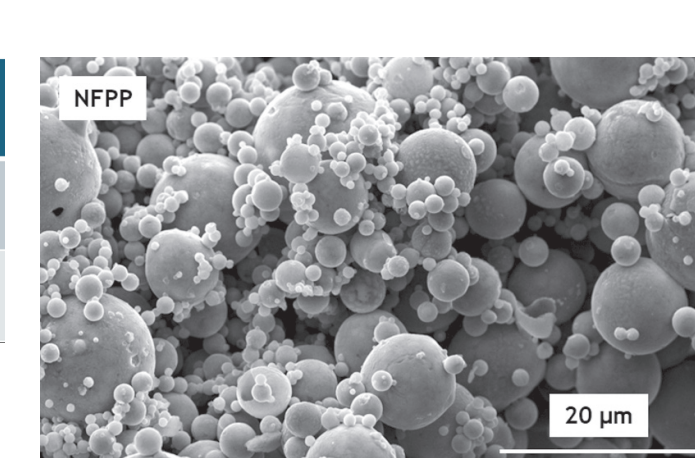


Fig.4 XRD pattern of the prepared NFPP and NFP. The diffraction spectrum of NFPP shows a composite material made of NFP-NFPP. A phase-pure material can be generated using an optimized calcination protocol. The diffraction spectrum of NFP indicates Fe_2O_3 as a minor component.

Advantages

- Adjustable particle size and narrow size-distribution: 50 nm - 50 μm
- Selectable reaction atmosphere: inert, oxidizing, reducing
- Drying, coating and calcination in one step starting from solutions, suspensions or solids
- Unique structures and chemical compositions like doped and undoped complex oxides, mixed oxides like spinel or mullite, doped materials
- Superior product properties by innovative pulsating gas technology up to 1300 °C
- Innovative electrical heating; heat recovery
- Flexible process variations like coating, synthesis, agglomeration and encapsulation
- Feasibility trials; process development; batch or continuous plants; contract manufacturing; scale-up