Flame Spray Pyrolysis of Electrode Materials for Energy Applications

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Introduction

Electrodes for PEM fuel cells, PEM electrolyzers and supercapacitors require starting powders with high conductivity, high surface area, well defined and sustainable pore structure/size distribution and long term stability/corrosion resistance. Flame spray pyrolysis (FSP) is evaluated as a powder production method for oxide based materials applied in such electrodes and appear ideal as single phase nanocrystalline, porous powders can be produced directly without the need for any further treatment.

Catalyst support materials for PEM FC/EC

Undoped and Sb-/Nb-doped titanium- and tin-based oxide powders for cathode catalyst support materials for PEM fuel cells and electrolyzers are prepared by FSP. The FSP powders show high surface area (BET): 90-110 m^2/g, as compared to commercial powders and powders derived from sol-gel/co-precipitation: 30-50 m^2/g.

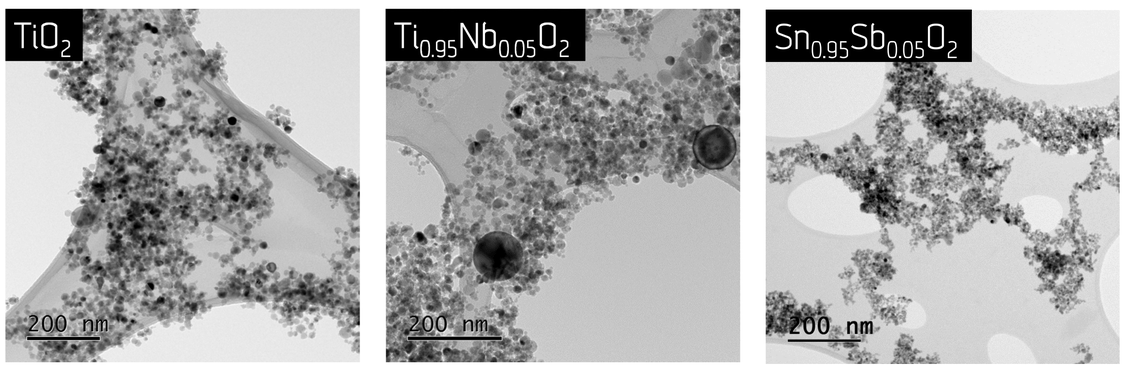

Figure 1: TEM micrographs indicating morphology and crystallite-/particle size.

Table 1: Crystal structures/crystallite size from X-ray diffraction/Rietveld method and surface area from nitrogen adsorption (BET) for TiO_2-based materials.

Composition		TiO_2	$Ti_{0.95}Nb_{0.05}O_2$
Phase composition	Anatase (wt%)	77.6	77.3
	Rutile (wt%)	22.7	-
	β-TiO_2 (wt%)	-	22.7
XRD crystallite size (nm)		A: 16.3 / R: 6.2	A: 16.8 / β: 8.8
BET surface area (m^2/g)		109	104

Table 2: Lattice parameters/crystallite size from X-ray diffraction/Rietveld method and surface area from nitrogen adsorption (BET) for SnO_2-based materials.

Composition		SnO_2	$Sn_{0.95}Nb_{0.05}O_2$	$Sn_{0.95}Sb_{0.05}O_2$
Lattice parameters (P42/mnm)	a (Å)	4.738	4.739	4.739
	c (Å)	3.189	3.189	3.187
XRD crystallite size (nm)		9.5	9.1	8.7
BET surface area (m^2/g)		91.5	102	102

- SnO_2 – IrO_2 (50:50 and 70:30 mol%): two-phase tetragonal system (XRD)
- Crystallite size (XRD): 5-7 nm
- Pore size distribution comparable to commercial carbon catalyst support
- Conductivity measurements
  - $Sn_{0.7}Ir_{0.3}O_2$: 0.013 S/cm
  - $Sn_{0.5}Ir_{0.5}O_2$: 0.155 S/cm
  - SnO_2 + Ir : 0.117 S/cm (reference powder 20wt% Ir):

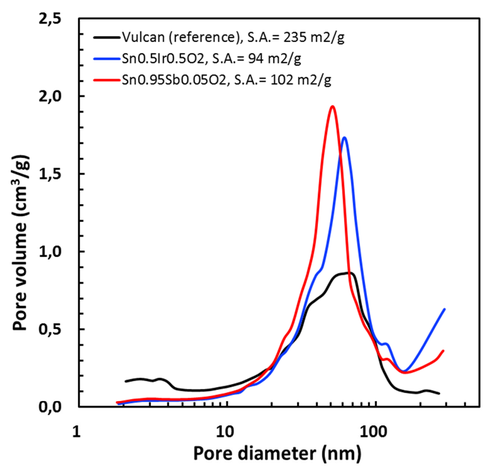

Figure 2: Pore distribution in SnO_2-powders.

Electrode materials for supercapacitors

Electrode materials based on manganese oxides for red-ox supercapacitors are prepared by FSP. The powder properties are tailored by varying the precursor/ solvent system. Less combustible precursor solutions and addition of slow burning organics (sucrose) results in reduced surface area and larger crystallites, due to increased residence/combustion time. In less oxidizing atmosphere (addition of sucrose and N_2 as dispersion/ sheet gas) MnO (5-7 %) is formed in addition to Mn_3O_4.

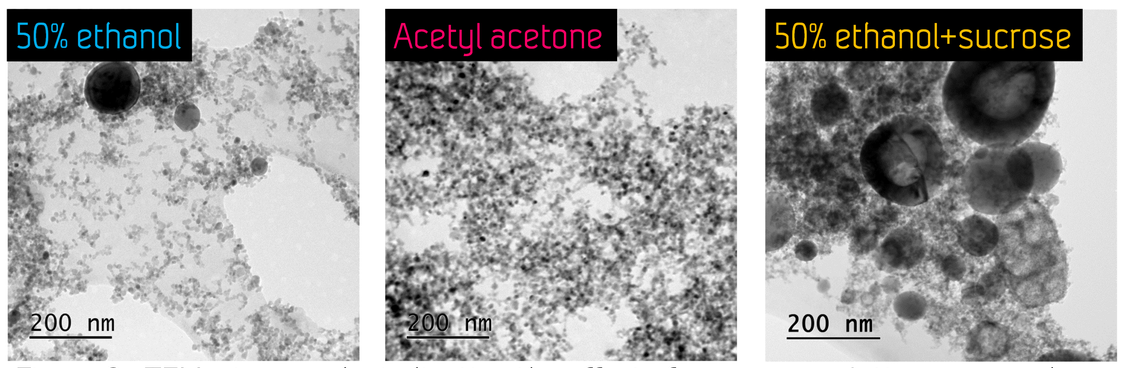

Figure 3: TEM micrographs indicating the effect of precursor solutions on powder morphology and crystallite- / particle size.

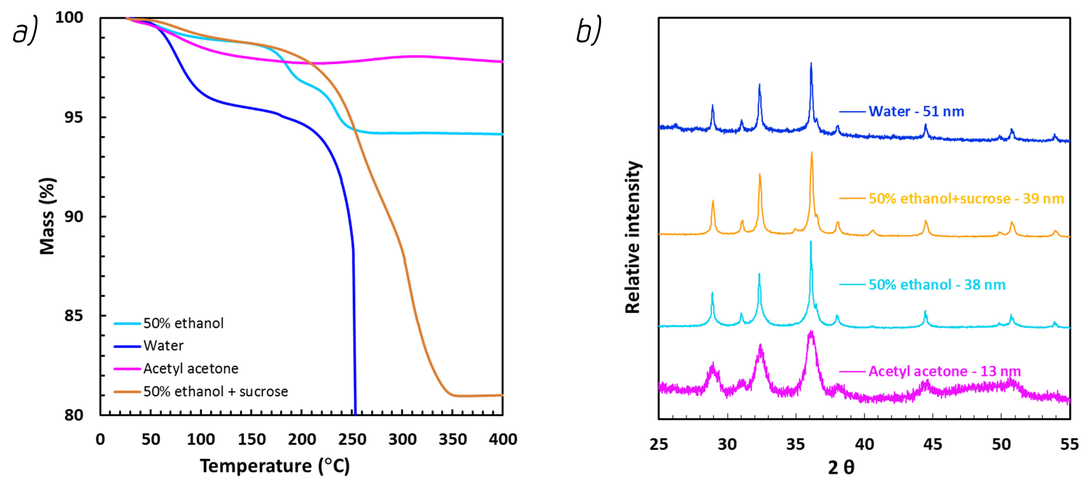

Figure 4: a) TGA and b) XRD indicating degree of combustion/crystallization.

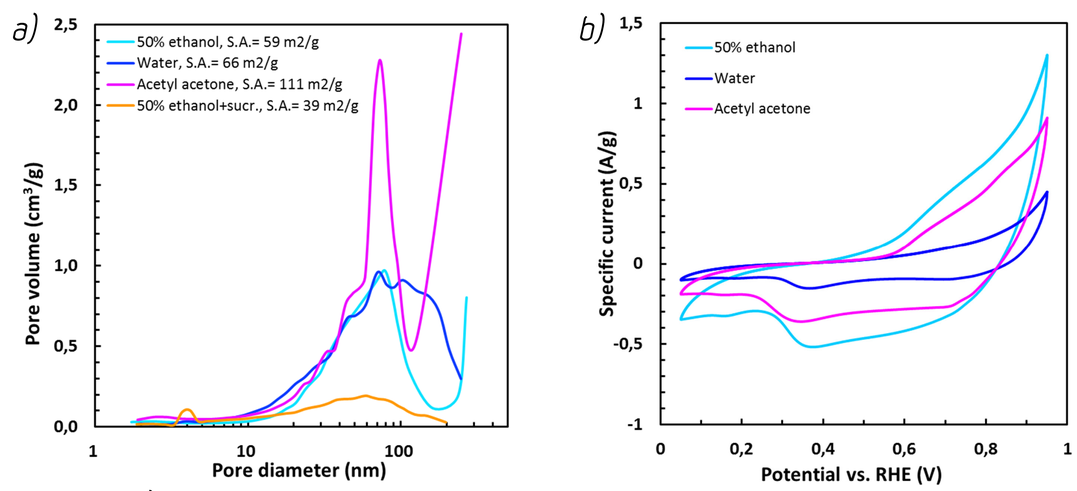

Figure 5: a) Pore size distribution and BET surface area from nitrogen adsorption/desorption measurement.
b) Voltammograms: effect of synthesis route on performance.

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