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CONTINUOUS SYNTHESIS OF NANOPARTICLES AT HIGH TEMPERATURES

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Introduction

Reproducibility is one of the most important goals in nanoparticle synthesis. The particle properties and hence the product quality often directly result from the particle size and particle size distribution. In batch-wise syntheses, difficulty in reproducibility is a common problem which can be adequately addressed by our technology. Moreover, continuous processes offer a number of advantages that are not achievable with conventional batch methods.

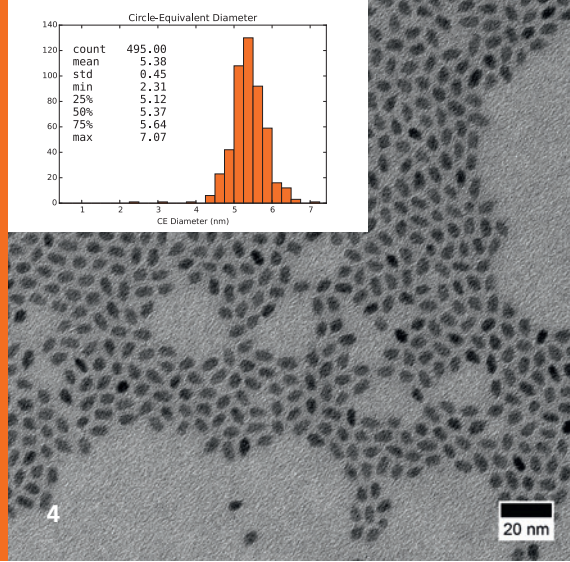
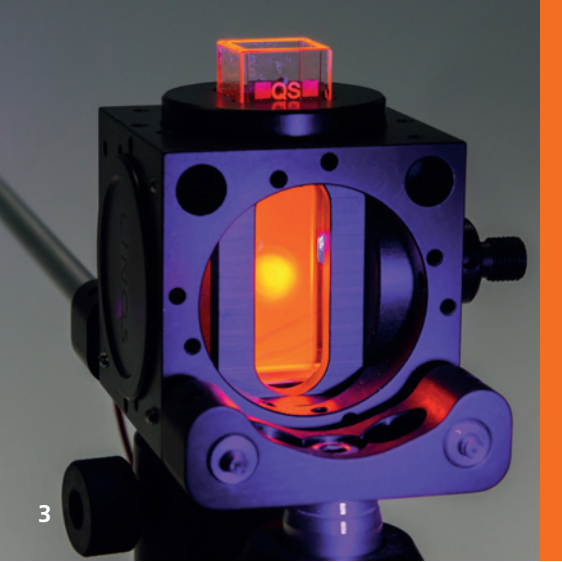
Modular Reactor for Nanoparticle Synthesis

A novel, modular reactor for continuous flow synthesis has been developed, designed for nanoparticle synthesis at elevated temperatures. The reactor has been tested with

liquid-phase reactions at temperatures of up to 400 °C, it enables nanoparticle synthesis routes requiring elevated temperatures and rapid mixing (“hot injection”), common conditions for the precise synthesis of high quality, monodisperse nanoparticles.

Application Example: CdSe Quantum Dots

One prominent material system for such “hot injection” syntheses are semiconductor quantum dots (QDs), colloidal nanocrystals that exhibit strong fluorescence with a size-dependent emission wavelength. The most common material is CdSe, but QDs can also be synthesized of other semiconductors, e.g. ZnS or InP. Additionally, the concept has been extended to core/shell architectures or particles with anisotropic shapes, e.g. CdSe/ZnS or CdSe/CdS core/shell quantum dots.



A key benefit of continuous syntheses is the tight process control and that the various process parameters such as flow rates and temperature can be adjusted to achieve the desired property, e.g. exciton wavelength in case of QDs. With our reactor, it is possible to tune the wavelength with sub-nanometer precision to the desired value in a stable process.

With the current design, flow rates of up to 10 ml/min, i.e. 14 L/d are possible. Obviously, the solid content depends on concentrations used. Easily achievable are about 100 g/d of solid material. Besides quantum dots, a whole variety of different materials such as metals or metal oxides have been synthesized with identical or very similar setups.

Modular Design

Central part of our reactor is a microfluidic mixer developed by Fraunhofer ICT-IMM and a temperature-controlled delay element. Integrated optical flow cells allow for in-situ optical detection at different positions, at high temperature, laying the ground for online process monitoring.

The reactor is modular by design and can be adapted to specific needs. Due to its compact size (approx. 20 x 30 x 50 cm³), the reactor module takes up little space in the laboratory or fume hood and can be integrated into existing setups.

For more complex synthesis routes that involve different temperature zones or

additional reagents, the reactor can be upgraded respectively.

Process Control

Precise inline and online analytics is a key issue to success. Therefore, the reactor has distinct analytical "windows" to monitor the product quality at different, essential time points. These are used to optically follow the particle growth and to manipulate the process according to the desired product properties and quality.

From Lab to Production Scale

Fraunhofer ICT-IMM has a long record of expertise in the field of microreaction technology, i.e. designing and building setups for continuous chemical processes used in flow chemistry. Scalability has been demonstrated for many cases and realized by both internal scale-up and parallelization, and external numbering-up. We are looking forward to establish and optimize continuous processes for our clients, tailored to their specific needs.

Examples of Nanoparticles synthesized by continuous processes at Fraunhofer ICT-IMM

Metals:

Au, Ag, Pt, Pd, Rh, Ni

Metal oxides:

Fe₃O₄, MnO, ZnO

Semiconductors:

CdSe, CdSe/ZnS, CdSe/CdS

Other Inorganic:

SiO₂, BaSO₄, Hydroxylapatite

Organic:

Polymers, Liposomes, Polymersomes

3 Fluorescent CdSe/CdS QDs

4 TEM image of CdSe QDs

