Upscaling Colloidal Nanocrystal Hot-Injection Syntheses via Reactor Underpressure

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Hot-injection technique approaches are convenient and fast one-pot processes, which are capable of providing colloidal nanocrystals with ultra-narrow size distributions. Effective time separation between nucleation and growth processes is facilitated by fast addition (*i.e.*, injection) of an elemental precursor or reducing agent to the hot reaction mixture. However, it is this fast addition of large volumes that presents a serious challenge for upscaling hot-injection protocols.
Here we focus on the possibility to upscale injection-based syntheses of colloidal nanocrystals without modifying the original protocol or using specially designed jet equipment. This work presents an easy and universal solution for linear upscaling of hot-injection synthesis (**Fig. 1**). [1] Applying a mild vacuum to the reaction mixture prior the injection enables an injection rate of 100-150 mL·s^{-1} such that large volumes of 200-500 mL can be introduced into the reaction flask within few seconds. We apply this underpressure-assisted approach to successfully upscale synthetic protocols for metallic (Sn) and semiconductor (PbS, $CsPbBr_3$ and $Cu_3In_5Se_9$) nanocrystals by one-to-two orders of magnitude to obtain tens of grams of nanocrystals per synthesis. We provide the technical details of how to carry out underpressure-assisted upscaling and demonstrate that nanocrystal quality is maintained for the large-batch syntheses by characterizing the size, size distribution, composition, optical properties, and ligand coverage of the nanocrystals for both small- and large-scale syntheses.
This work shows that fast addition of large injection volumes does not intrinsically limit upscaling of hot injection-based colloidal syntheses. An underpressure-governed hot-injection method enables a systematic optimization of nanocrystals and nanocrystal-based devices from a single source batch for research and development purposes and reinforce the commercial viability of electronic, photonic, and electrochemical devices that use large numbers of colloidal nanocrystals (*e.g.*, solar cells, lithium-ion batteries, thermoelectrics, phase-change memories, etc.).

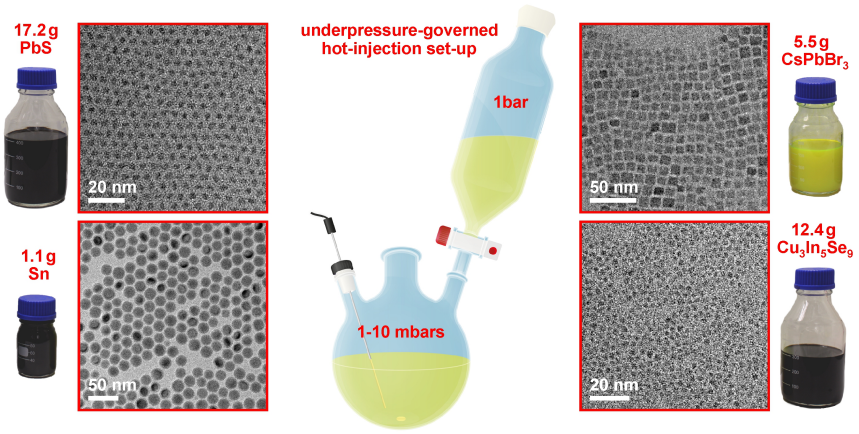

Fig. 1. Schematic illustration of underpressure-governed hot-injection set-up and various colloidal nanocrystals, obtained from single-batch large-scale syntheses

[1] Maksym Yarema, Olesya Yarema, Weyde M. M. Lin, Sebastian Volk, Nuri Yazdani, Deniz Bozyigit, and Vanessa Wood. Chemistry of Materials, 2017, 29, 796-803.