Preparation of colloidal clusters via controlled hydrothermal carbonisation of polystyrene particles

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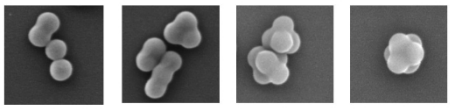
The controlled assembly of colloidal particles into finite clusters with well-defined geometries remains a key challenge in the design of hierarchical materials. In this work, we introduce a scalable strategy for generating dense clusters of cross-linked polystyrene nanoparticles via microwave-assisted hydrothermal carbonization (HTC) with glucose as the carbon precursor.
The approach exploits the dynamic interplay between carbon shell formation and particle destabilization during HTC. We show that the formation of acidic by-products leads to a decrease in pH and a concomitant increase in solution conductivity, progressively screening electrostatic repulsion and promoting particle aggregation. By systematically varying reaction parameters—including temperature, duration, initial pH, particle concentration, and stirring—we identify the key factors governing both carbon-coating kinetics and cluster-formation pathways.
Statistical modeling reveals that temperature, reaction time, and particle concentration strongly influence final conductivity and pH, which in turn control particle stability and aggregation regimes. The transition from diffusion-limited to reaction-limited aggregation enables tuning between open aggregates and compact, close-packed clusters, including dimers, trimers, and higher-order structures. Importantly, cross-linked particles prevent reshaping, allowing direct observation of cluster configurations formed during assembly.
We demonstrate that clustering can be finely controlled by balancing destabilization and surface re-stabilization induced by carbon deposition. Conditions such as moderate initial pH (~6.3) and controlled stirring favor the formation of monodisperse, discrete clusters, while higher temperatures, longer durations, and reduced stirring promote larger aggregates.
This work establishes hydrothermal carbonization as a versatile route to engineer colloidal clusters from simple isotropic building blocks, providing a tunable platform for fabricating colloidal “molecules” with potential applications in materials science and nanotechnology.

Keywords:
Self-assembly, clusters, carbonization, polystyrene colloids



SEM images of clusters prepared by hydrothermal carbonization of polystyrene particles