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Generating Nanoparticles Using Supercritical Fluids

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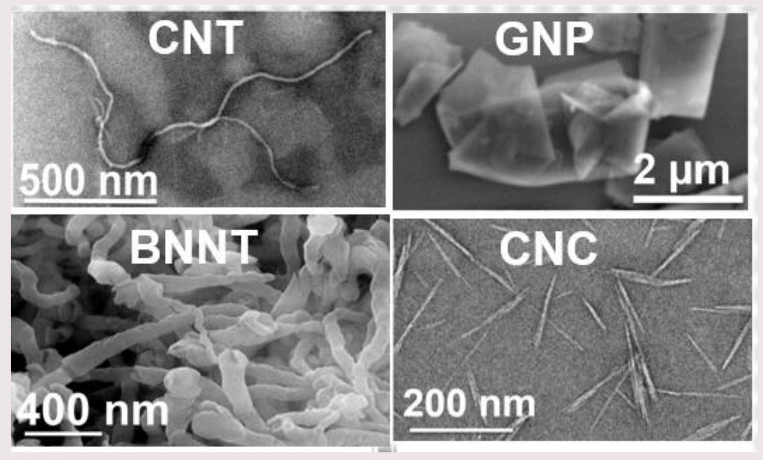
Objectives:

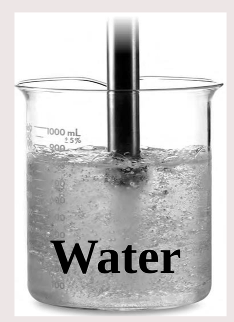
Introducing a novel process for bottom-up fabrication of engineered nanostructures by self-assembly of nanoparticles (NPs) through evaporation of particle-laden droplets. This entails two main steps:

1. Dispersion and stabilization of NPs in a solvent to make a colloidal suspension containing a single type or a hybrid system of NPs.
2. Atomization of the colloidal suspension and controlled formation of droplets that carry these NPs and deliver them on a substrate.

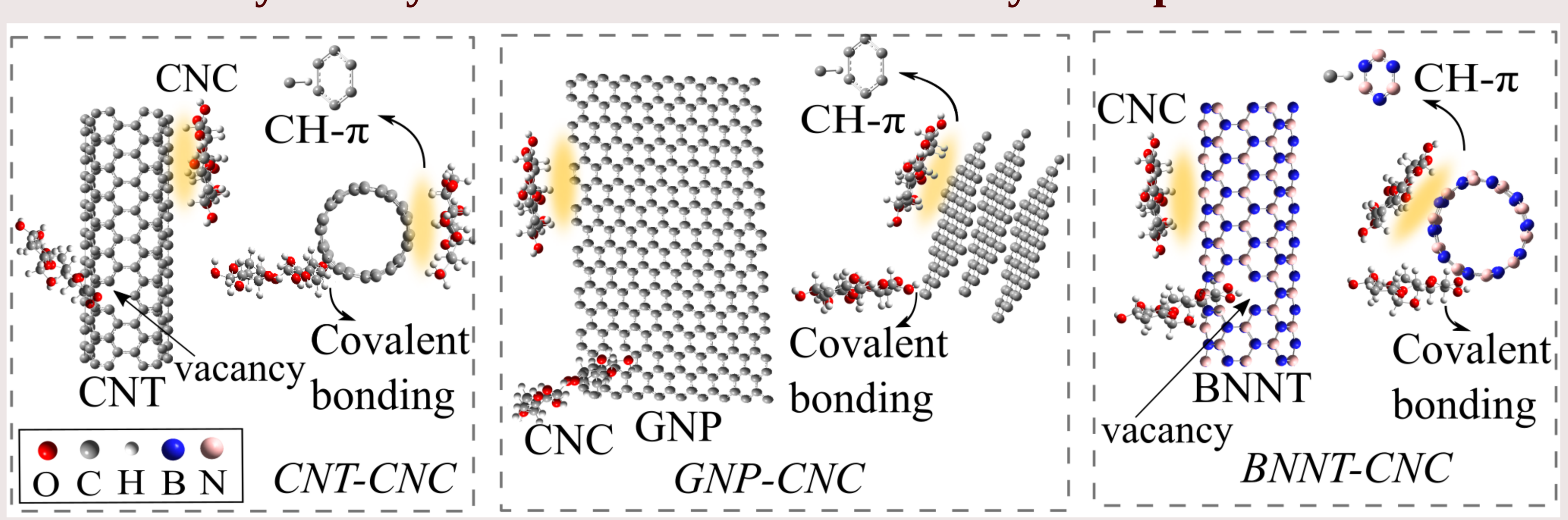
Individual Nanoparticles



Probe Sonication



Hybrid Systems of CNC and Secondary Nanoparticles



1. Preparation of Aqueous Nanoparticles Suspension

- NPs of different shapes, sizes and elemental compositions have been selected: Carbon Nanotubes (CNTs), Graphene Nanoplatelets (GNPs), Boron Nitride Nanotubes (BNNTs) to disperse in water using Cellulose Nanocrystals (CNCs).
- Aqueous suspensions of CNC and the secondary NP with different mass ratio and concentrations are prepared by probe sonication and hybrid NP systems (HNPS) are formed.

Interactions among Nanoparticles:

The molecules of CNC attach to CNT, GNP and BNNT through strong covalent bonds between hydroxyl groups on CNC and defected regions of the CNT, GNP and BNNT, as well as polar-π interactions between C-H in CNC and carbon rings in CNT and GNP and BN in BNNT to form HNPS.

Interactions among nanoparticles and solvent:

The balance of three forces determines the final assembly of nanoparticles:

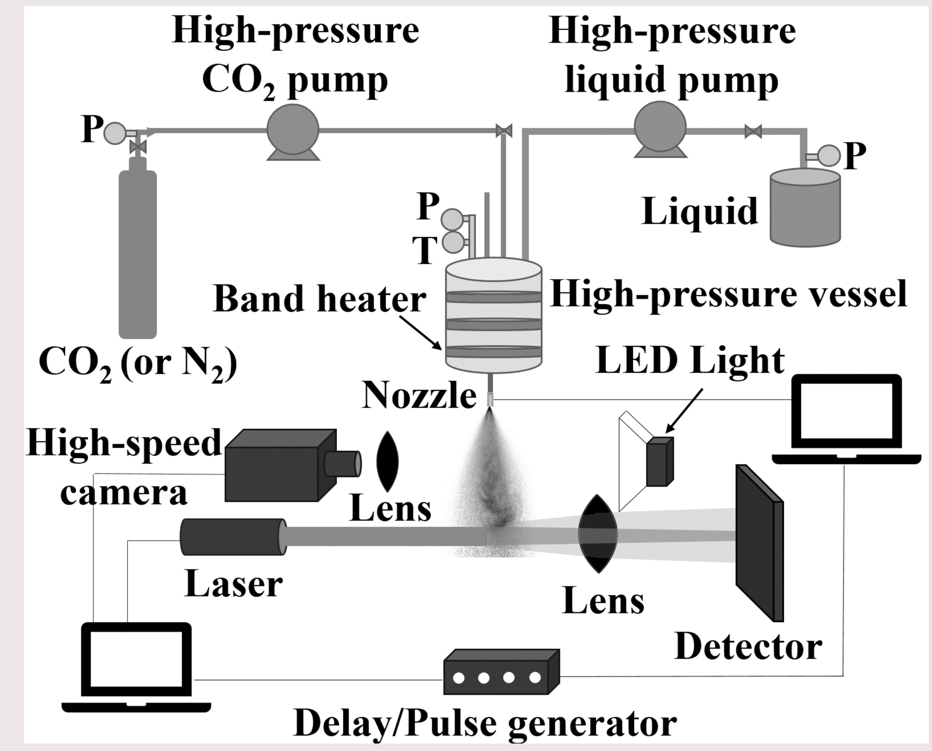
1. long-range electrostatic forces
2. Van-der-Waals interactions
3. Capillary forces

The prevailing force depends on the ratio of CNC to the secondary NPs in hybrid cases, as well as concentration of NPs.

References:

1. Reverchon, E. J. I.; research, e. c., Supercritical-assisted atomization to produce micro-and/or nanoparticles of controlled size and distribution. **2002,** *41* (10), 2405-2411
2. Deegan, R. D.; Bakajin, O.; Dupont, T. F.; Huber, G.; Nagel, S. R.; Witten, T. A. J. N., Capillary flow as the cause of ring stains from dried liquid drops. **1997,** *389* (6653), 827

Schematic of the Supercritical Spray System

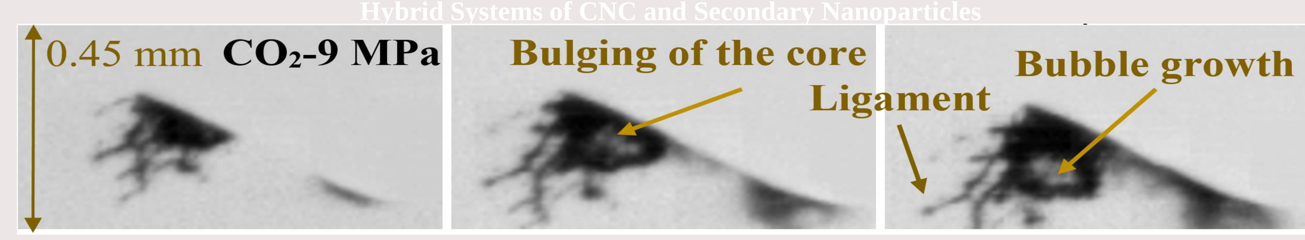


2. Formation and Delivery of Particle-Laden Droplets

Supercritical Assisted Atomization (SAA) utilizes dissolved supercritical fluid to enhance the atomization by triggering two simultaneous mechanisms:
1. Reducing liquid surface tension, 2. Enabling dissolved gas atomization
The combined effects result in controllable creation of micron-size droplets with narrow size droplet distribution [1].
The supercritical fluid of choice is CO_2 (SC-CO_2), due to: 1. High solubility of various materials in SC-CO_2, 2. Moderate critical temperature/pressure of CO_2

- ❖ Tuning spray parameters e.g. injection pressure/temperature, Gas-to-Liquid ratio, and axial distance from the nozzle enables controlling droplet size and distribution.

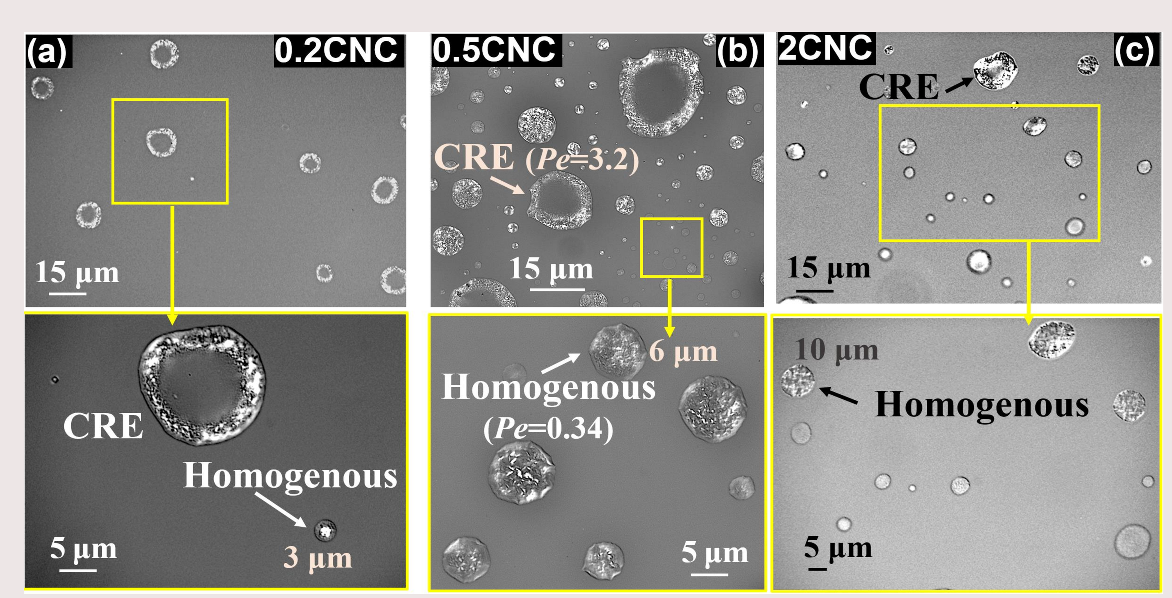
Liquid Jet Breakup



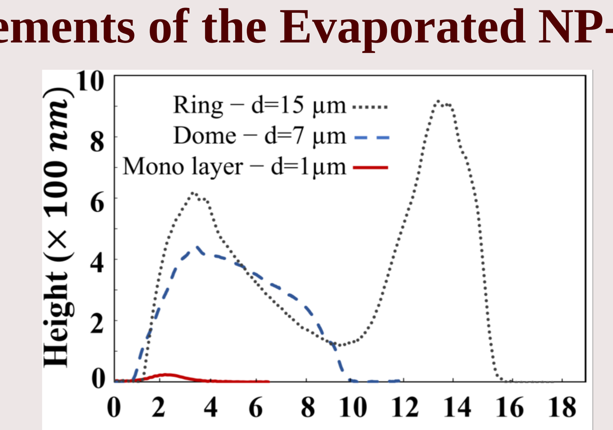
Characterization of Final Self-Assembled Nanostructures

Single NP system: size of droplet and concentration of NP in the solvent define the final pattern for colloidal suspension containing one NP type.

Polarized Microscopy of the Evaporated Droplets

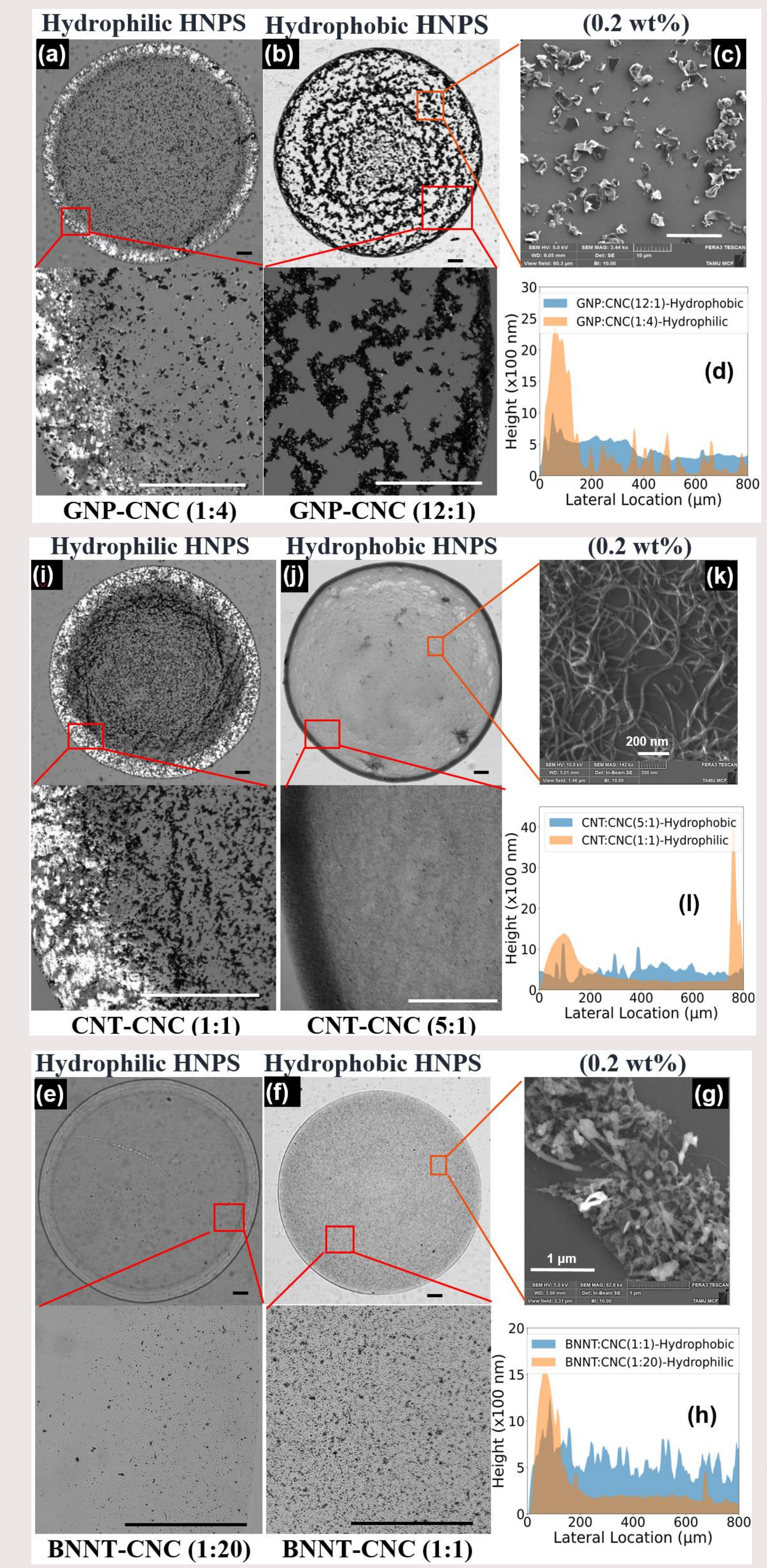


Profile Measurements of the Evaporated NP-Containing Droplets

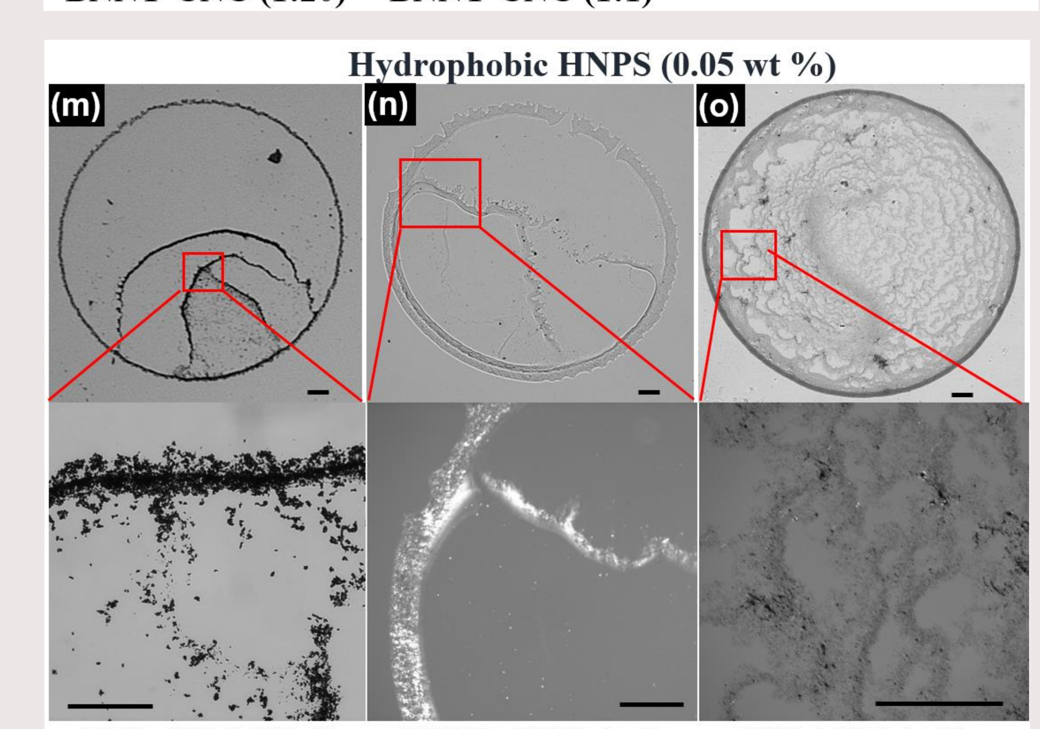


- The NP patterns can be controlled to form a ring [2], uniform or a ring with NP wires regardless of the shape and type of NP.
- For the Hybrid NP system: Mass ratio of involved NPs and their concentration in solvent defines the assembly of final nanostructure.

Different Engineered NP Patterns



Effect of Nanoparticles Ratio



Effect of Nanoparticles Concentration