Diagnostics of Combustion Synthesis of Iron Oxide Nanoparticles: from gas phase intermediates to solid particulates

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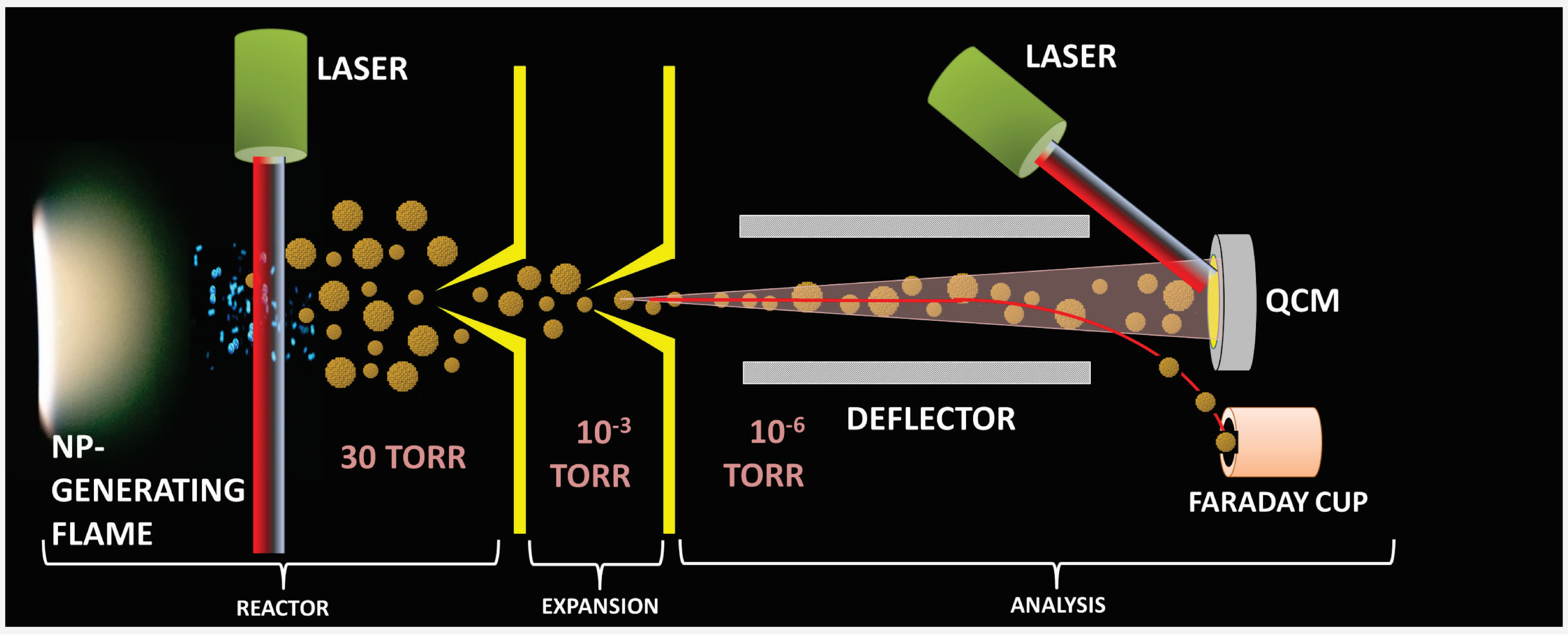
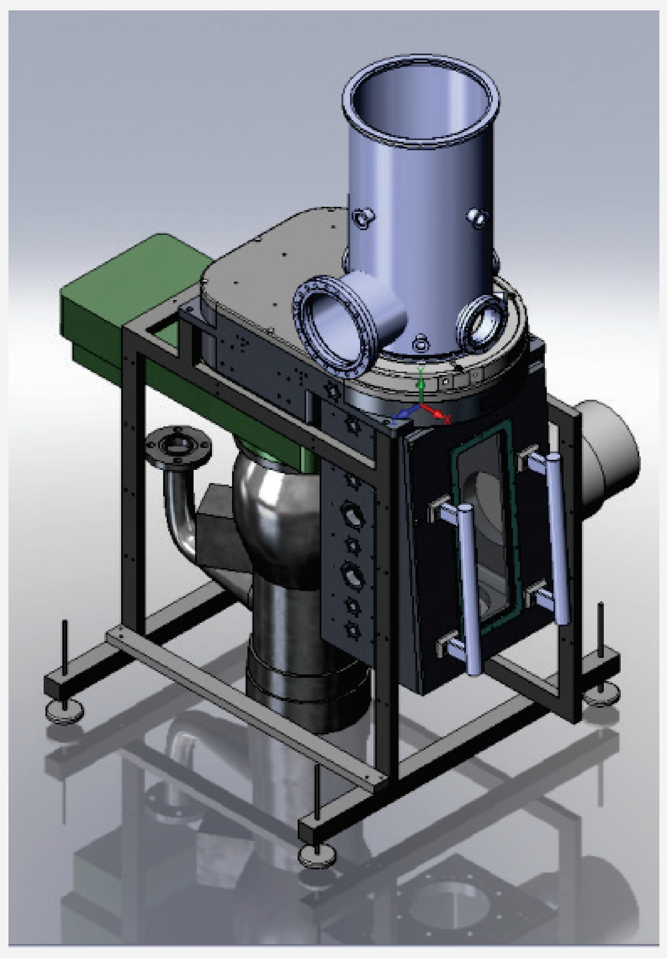
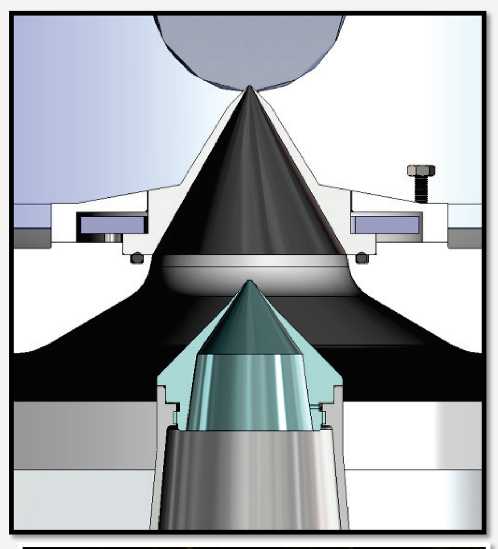
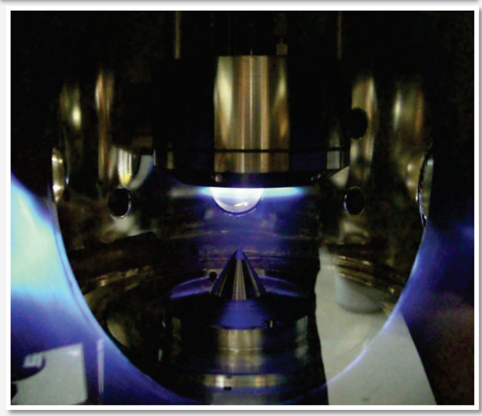
Flame-assisted synthesis is a route for scalable production of nano-sized metal oxide materials with well-defined properties. Iron oxide nanoparticles are used in diverse applications, including optical magnetic recording, catalysis, gas sensors etc. Their properties are dependent on their characteristics, such as size distribution, phase and morphology. Detailed understanding of the mechanism governing the particle formation in flames is a necessary pre-requisite for flame synthesis of NPs with tailored functionalities. Mechanism validation calls for quantitative (and desirably *in-situ*) diagnostics of both gas-phase species (e.g. FeO) and nascent solid iron oxide NPs.

THE EXPERIMENTAL SETUP



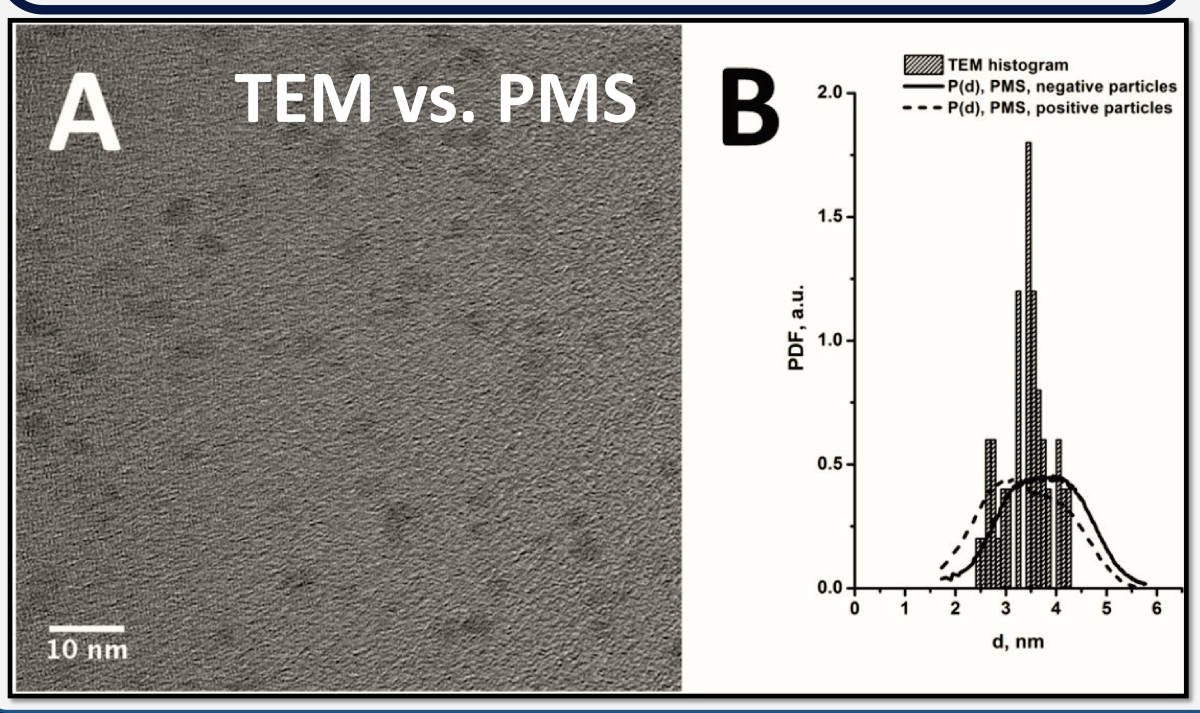




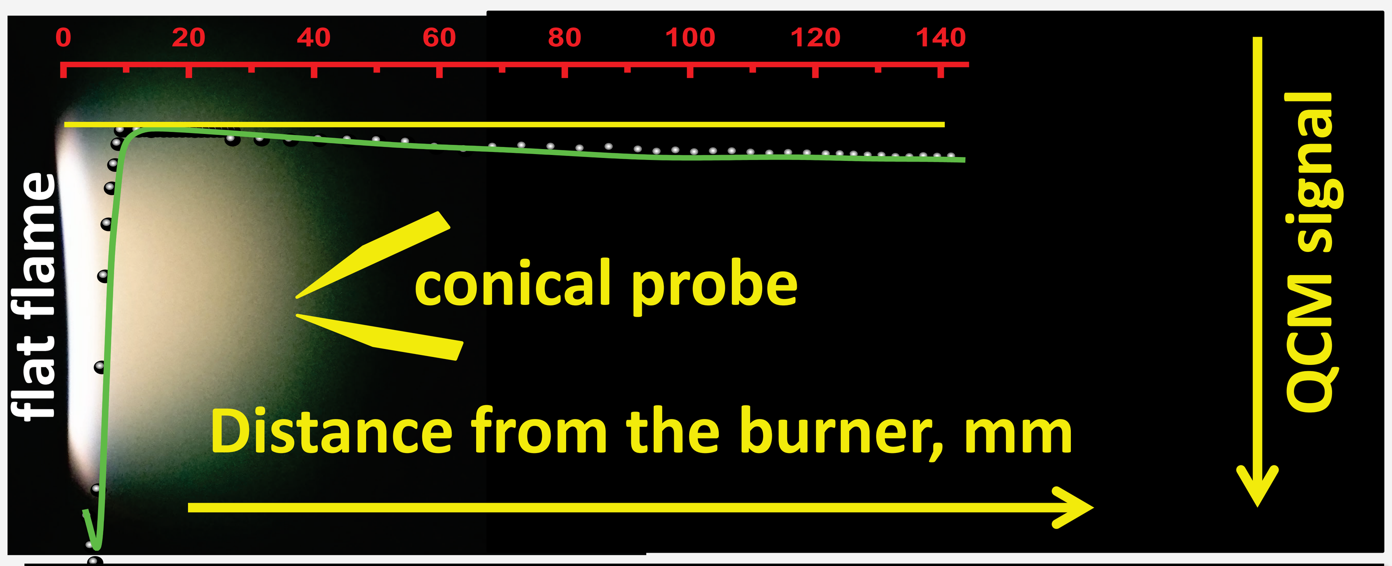


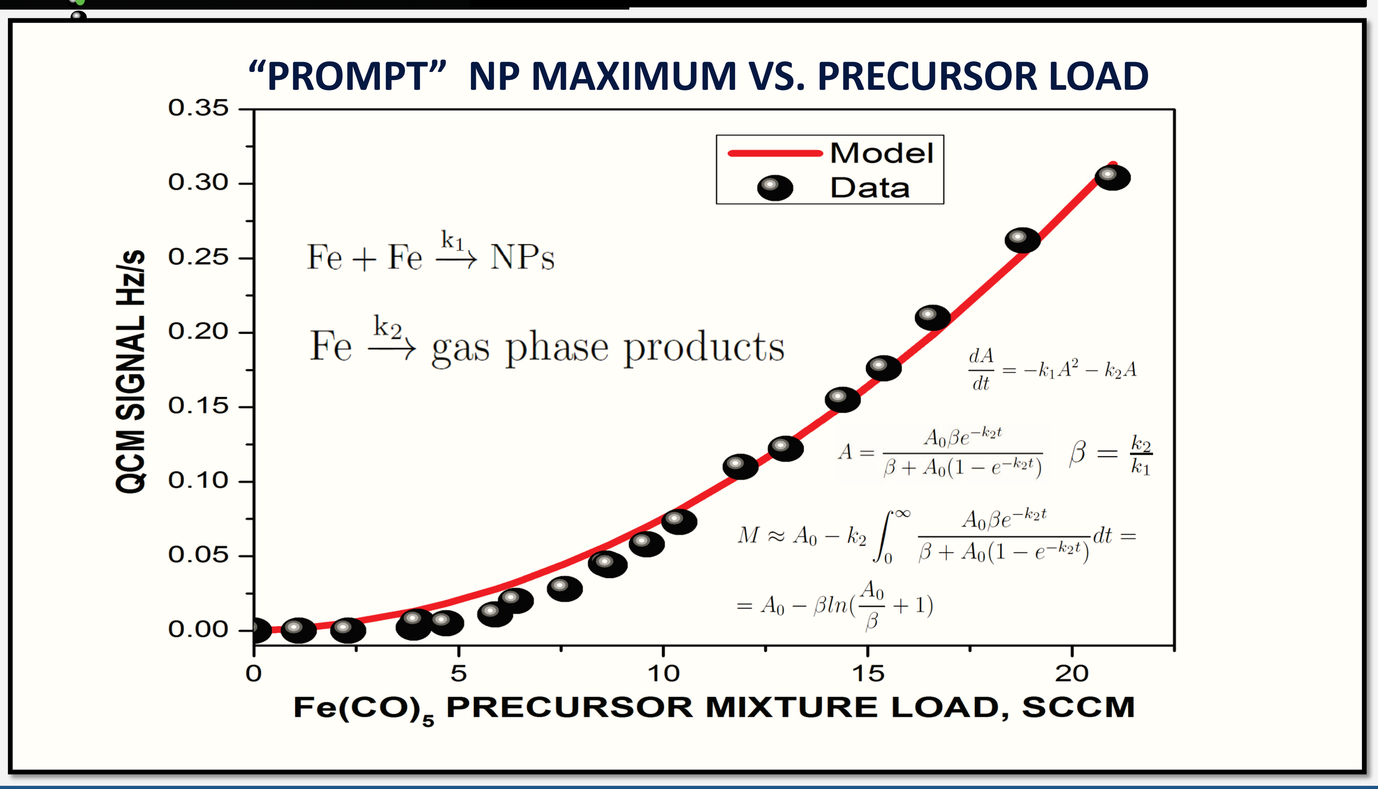
NANOPARTICLE FORMATION AND CONSUMPTION

The measurement is based on a combined Particle-Mass-Spectrometry-Quartz Crystal Microbalance (PMS-QCM) analysis technique. The analysis protocol incorporates molecular beam sampling of the flame aerosol, sorting the charged particles according to mass/charge ratio (*m/z*) via electrostatic deflection and detection of neutral particles by monitoring the oscillation frequency of the quartz crystal exposed to the particle-laden molecular beam. This yields the information regarding total mass concentration, probability density distribution of *m/z* and particle number density.

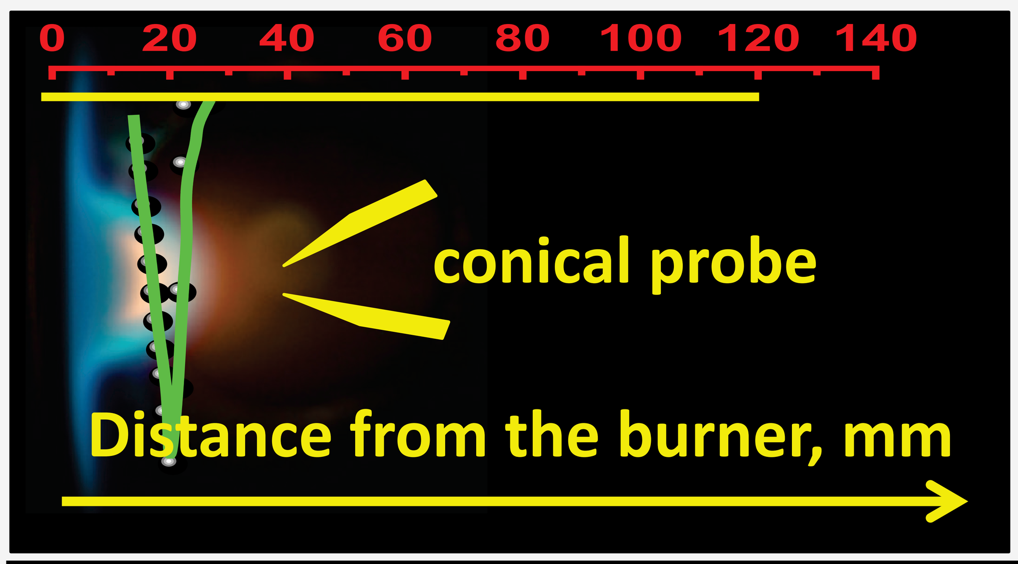


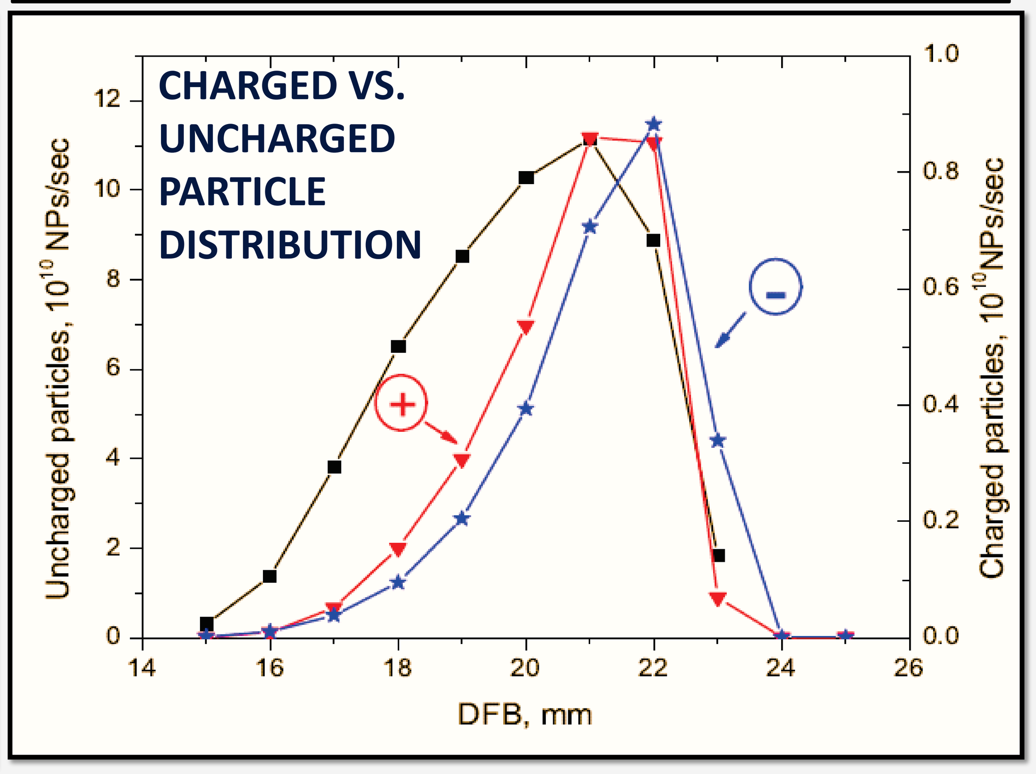
LOW PRESSURE FLAT FLAME: FACILITATES BOTH MODELING AND MEASUREMENTS



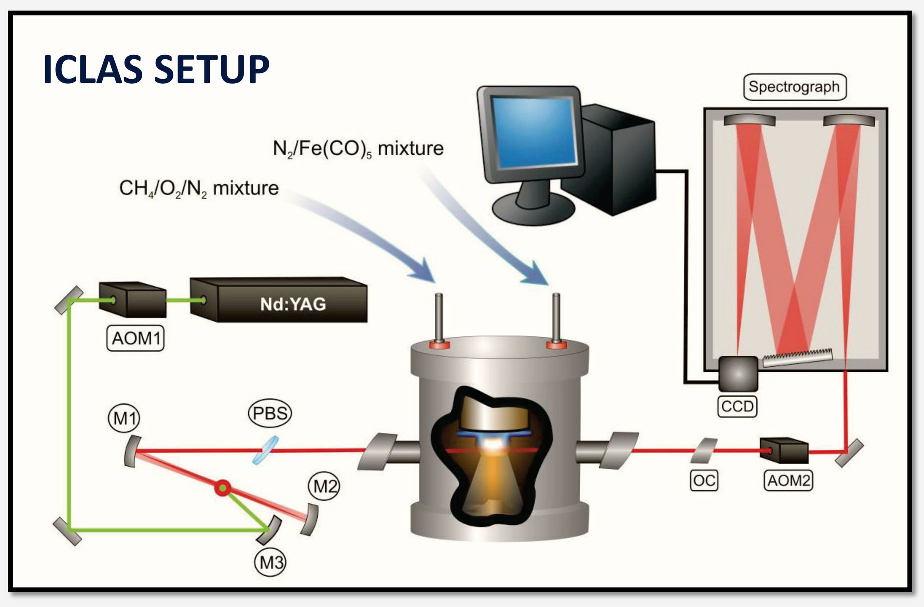


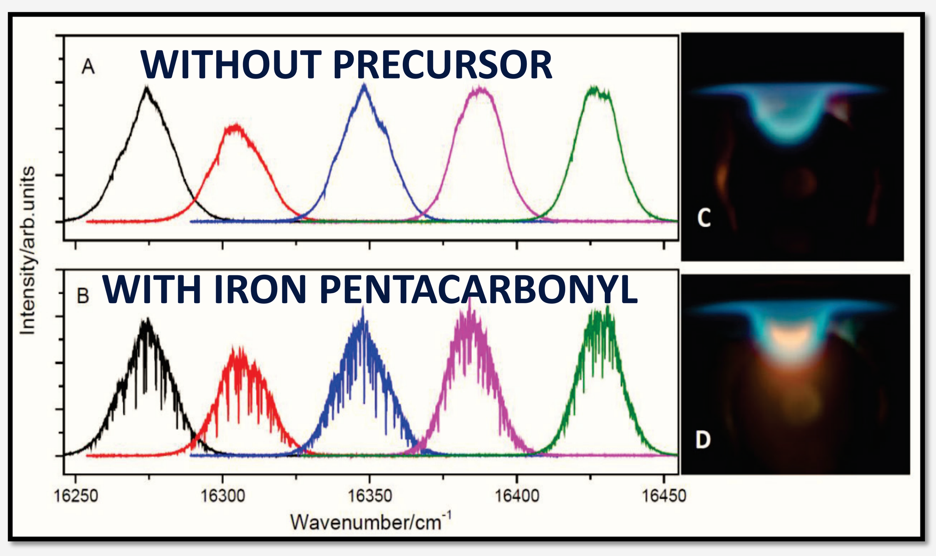
HYBRID FLAME: WIDE RANGE OF CONDITIONS AND EASY ACCESS TO THE FLAME FRONT





GAS-PHASE SPECIES

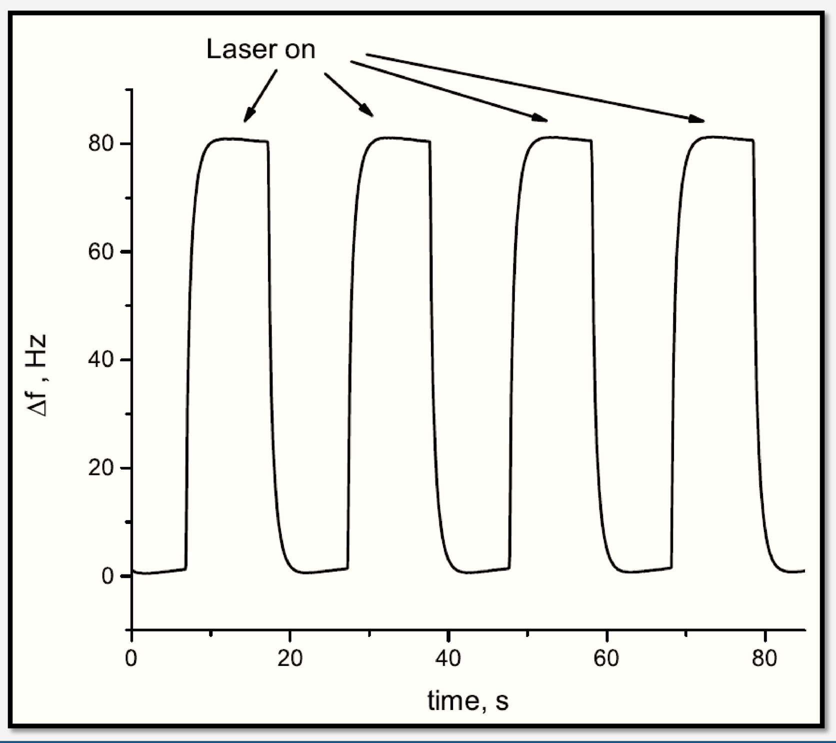


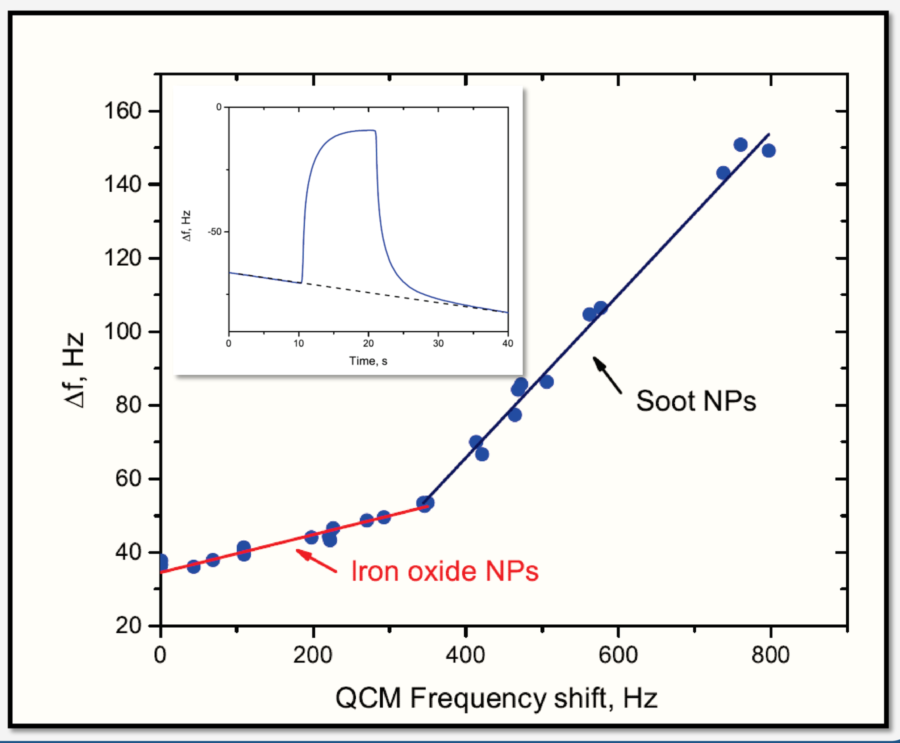


The chemistry of iron-containing gas-phase intermediates (e.g. FeO) is also critical in establishing the final characteristics of the synthesized iron oxide nanoparticles. However, their monitoring in particle-laden environment with standard methods, such as laser induced fluorescence, is hampered due to light scattering from solid particulates and luminosity of particle-generating flames. In this work these interferences were circumvented by deploying Intracavity Laser Absorption Spectroscopy (ICLAS). While the sensitivity to narrowband absorption of the gas-phase FeO is largely enhanced, ICLAS is not sensitive to the broadband absorption by the NPs, allowing to monitor FeO in particle-laden environment

NP OPTICAL ABSORPTION

The increase of the resonance frequency of the quartz crystal induced by laser irradiation. was studied. The magnitude of the frequency detuning effect is proportional to the amount of energy absorbed on one of the crystal electrodes. When the crystal is covered by flame-generated nanoparticles (deposited by molecular beam sampling), the amount of the laser energy absorbed, and the consequent frequency detuning are reflecting the magnitude of the absorption coefficient of the NP layer





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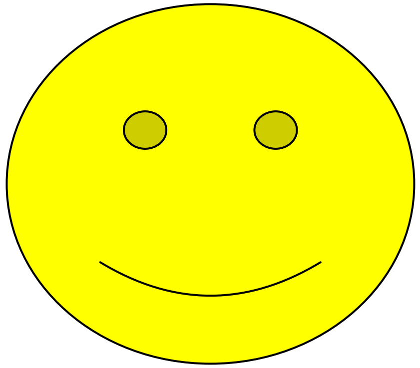

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