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Dynamic Monitoring of Multi-Concentrated Silica Nanoparticles Colloidal Environment with Optical Fiber Sensor

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1. Introduction – Colloidal Suspensions

Dispersed phase **+** **Base fluids**

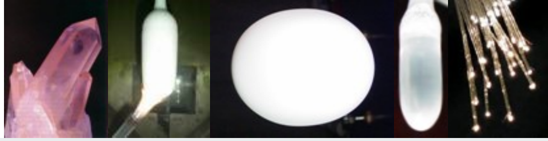
Dispersed phase

- Metal:
  - Cu, Al, Ag, Fe;
- Ceramics and non-metallic solids:
  - Al_2O_3, CuO, SiO_2, Si;
- Biomolecules;
- Polymeric particles.

Base fluids

- Water;
- Engine Oil;
- Organic solvents.

Li *et al.*, 2008

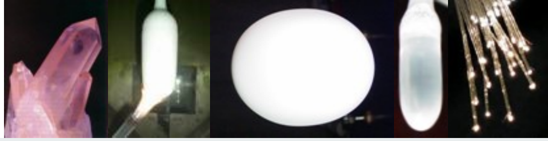


1. Introduction – Traditional Approaches for Colloidal Characterization

TEM Equipment

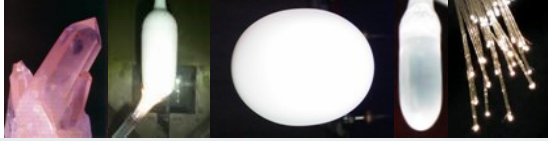
- Evaluation of particles size, morphology and concentration by laboratory techniques, such as:
  - Transmission Electron Microscopy (TEM);
  - Scanning Electronic Microscopy (SEM);
  - Zeta potential measurements.

- Expensive and time consuming;
- Sample preparation;
- Difficulties in calibration.



2. Our Approach – Assessment with Optical Fiber Sensor

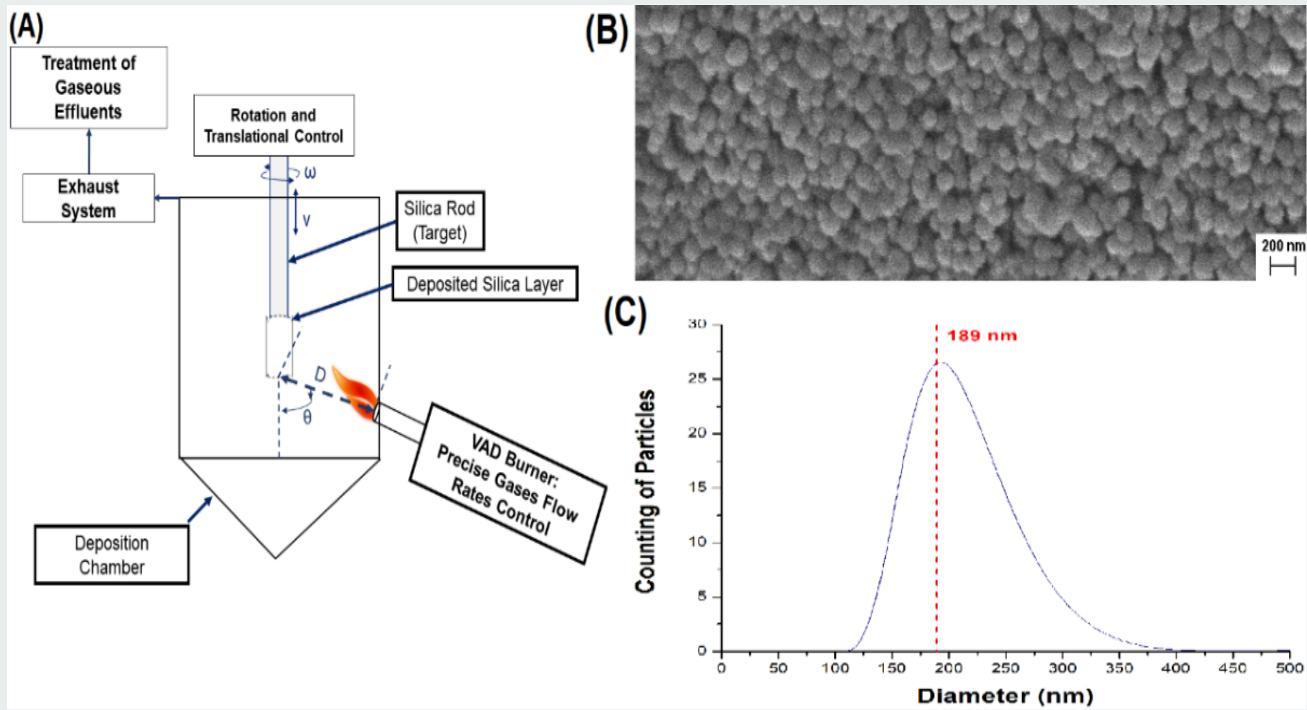
- Compact size;
- Immunity to electromagnetic interference;
- Chemically and biologically inert;
- Easy calibration;
- Fast sample preparation – **dispersion of particles in water, with sonication;**
- Easy data processing.



2. Preparation of Silica

- **Method of Vapor-phase axial deposition (VAD);**
- High deposition rate;
- Particle size control.

$$SiCl_4 + 2H_2O \rightarrow SiO_2 + 4HCl$$
$$SiCl_4 + O_2 \rightarrow SiO_2 + 2Cl_2$$



(A) Schematics of the VAD system; (B) SEM image; (C) Size distribution of particles (average diameter of 189 nm).

Soares *et al.*, Mat. Res., 2018

2. Optical Fiber Sensor

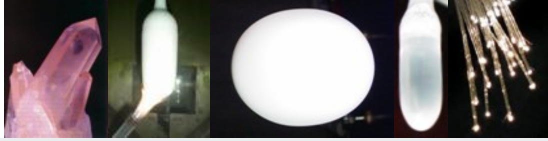
- Based on Quasi-Elastic Light Scattering (QELS);
- QELS is produced when particles are hit by light with wavelength comparable to their dimensions;
- From the I_R signal it is possible to calculate the **autocorrelation of the intensity, which is related to the concentration and dimensions** by Siegert relation:

$$G_2(\tau) = A + Be^{-2\Gamma_m \tau}$$

Where A is the baseline, B is the coherence factor, and Γ_m is the average decay rate. According to the **Stokes-Einstein** equation:

$$\Gamma_m = Dq^2$$

In which D is the translational diffusion coefficient and q is the magnitude of light scattering vector.



2. Assessment of Colloidal Concentration

- Colloidal suspensions produced by dispersing the silica nanoparticles in DI water, followed by mixing and sonication during 180 min: **1% (m/m) silica suspension**;

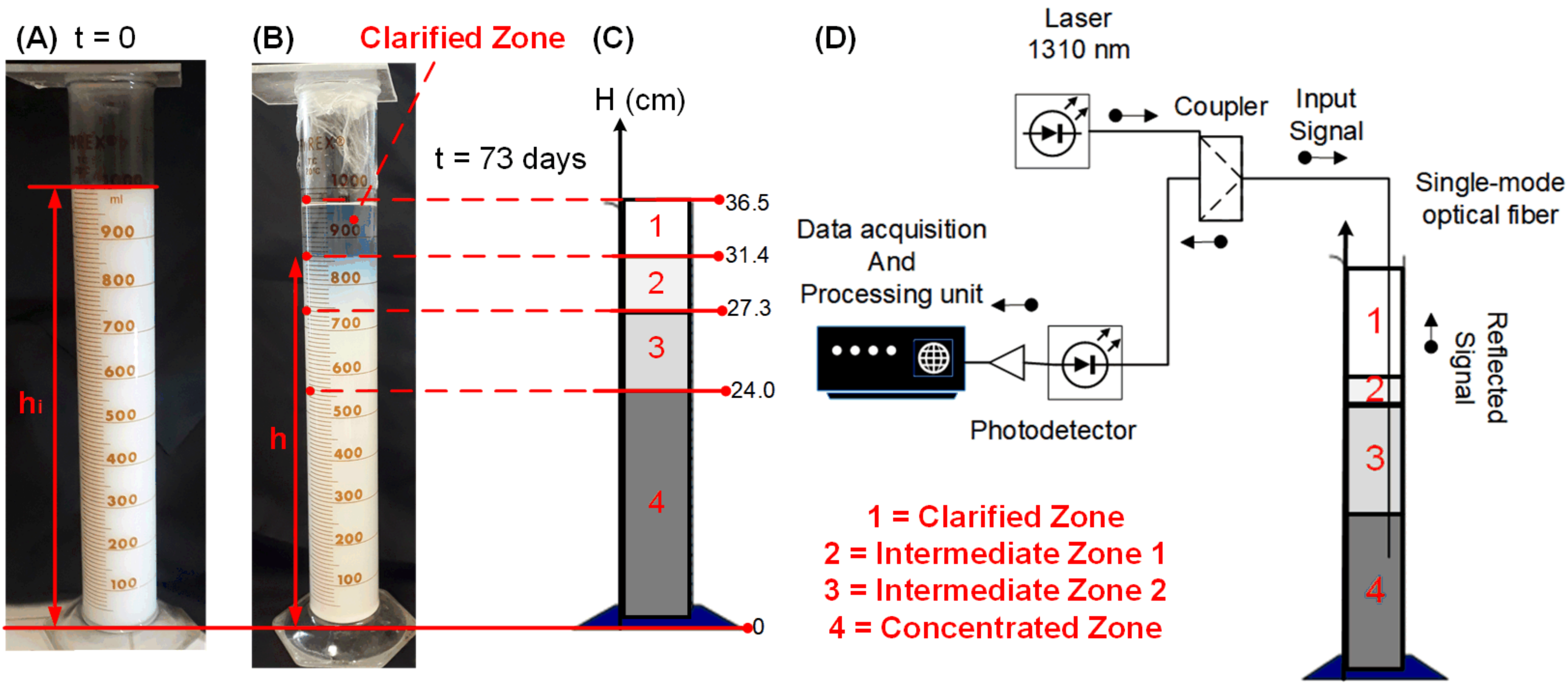
- Two (1000 mL)-test tubes were filled with the suspensions and left in rest so the particles could slowly sediment, creating **a clarified zone on the top of the flask, a concentrated zone on the bottom, and zones with intermediate concentration;**

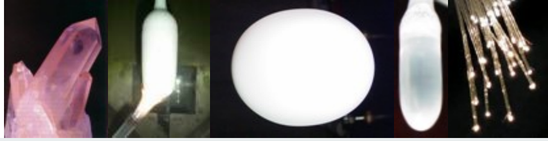
- The sensor response for the probe immersed in the regions of different concentrations was evaluated (~25°C);

- The initial value of the height h(t) from the bottom of the tube to the beginning of the clarified zone is h_i and the initial concentration of particles is C_i (mass/volume). If the concentration in the clarified zone is negligible, **the estimated concentration C(t) on the bottom zone is**:

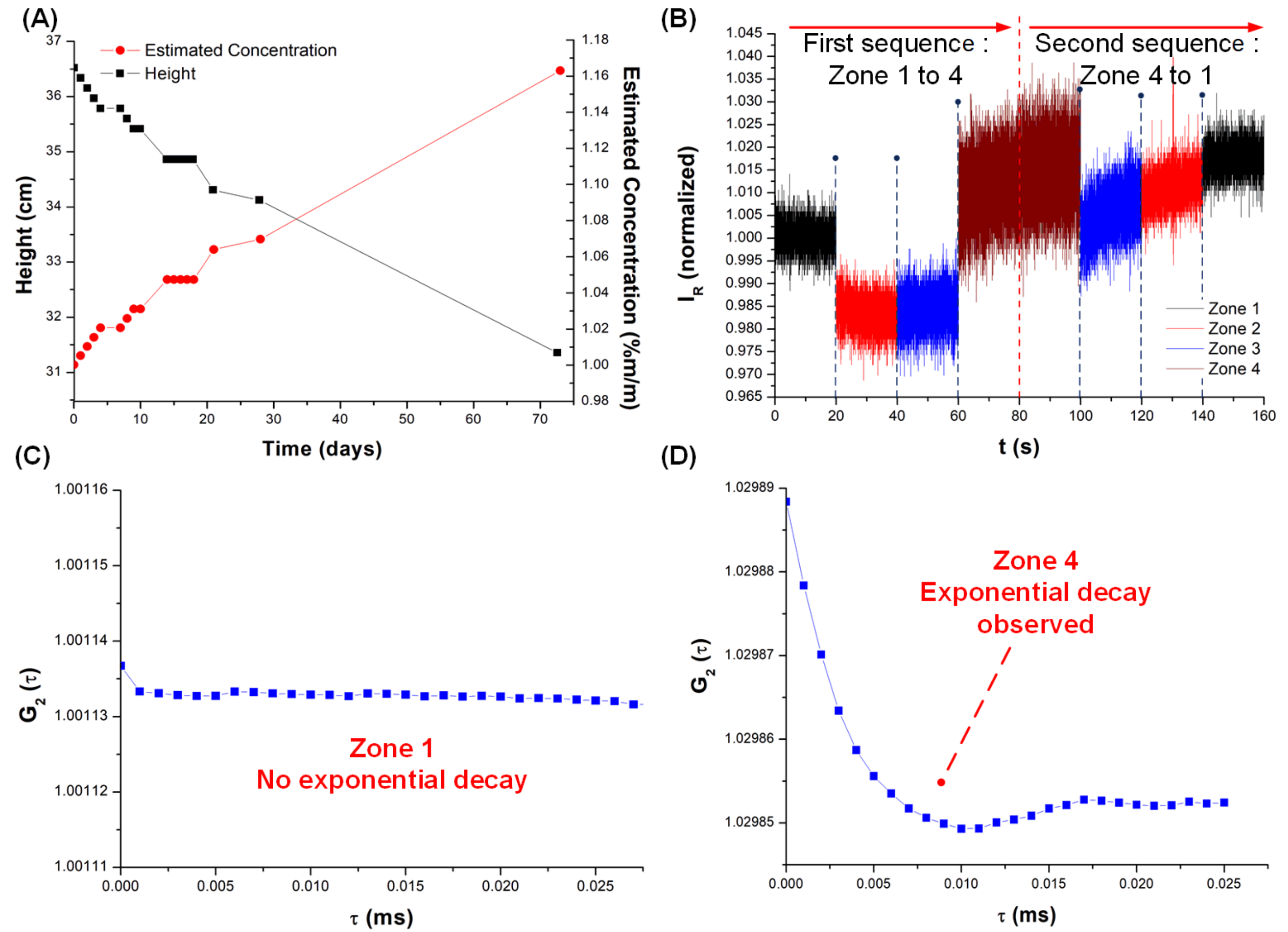
$$C = \frac{h_i}{h} C_i$$

2. Assessment of Colloidal Concentration





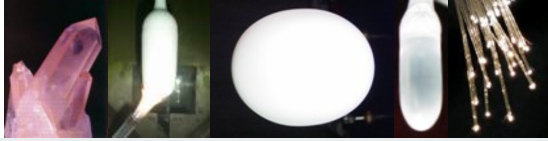
4. Results and Discussion



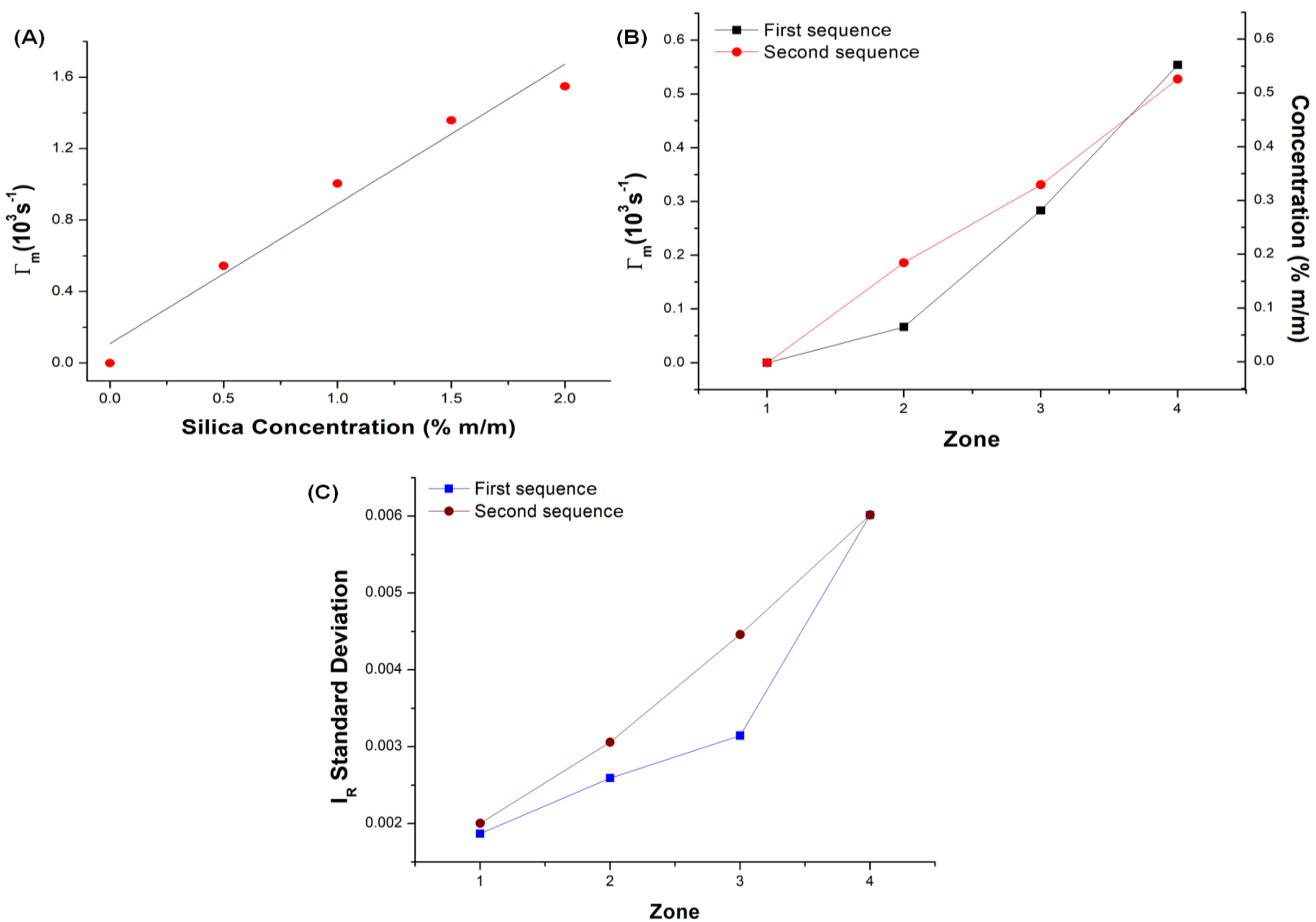
(A) Average values of the heights verified for the two tubes and concentrations estimated from Equation 3 (secondary vertical axis); (B) sequential step disturbances on the concentration of the OFS probe; (C) $G_2(\tau)$ obtained for Zone 1; (D) $G_2(\tau)$ obtained for Zone 4, with an exponential decay.

3. Results and Discussion

- **In all of the 73 days: no QELS detected on the clarified zone** (the hypothesis that the clarified is approximately free of particles is correct);
- Calibration curve was obtained: Γ_m for suspensions with known concentration of silica ($\Gamma_m = 0.78288C + 0.10898$, C in % m/m, $R^2 = 0.95942$) - **$0.78288 \times 10^3\ s^{-1}$ sensitivity**.
- The sensor probe was sequentially moved from Zone 1 to Zone 4 (concentration of 1.16%, estimated from Equation 3), in a first sequence of step disturbances, and then a second sequence was performed, from Zone 4 to Zone 1.



3. Results and Discussion



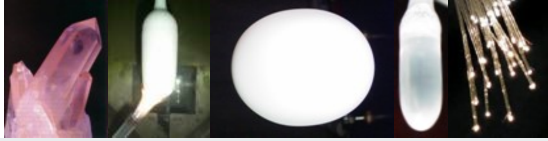
(A) Calibration curve;

(B) Γ_m and concentrations;

(C) standard deviations in each zone.

3. Results and Discussion

- Same tendencies between Γ_m and σ with similar behaviors for both sequences;
- Small modifications of the average signal when the probe is moved from one zone to another, what is probably caused by **fiber macrocurvature losses**;
- **Lowest and highest signal-to-noise ratios (SNRs)** evaluated as μ^2/σ^2, where μ is the average signal: **2.604 x 10^4 (Zone 4, second sequence) and 2.869 x 10^5 (Zone 1, first sequence)**, respectively;
- **The measured concentrations were substantially inferior to the traditional sedimentation model**: the particles are not all concentrated in a same zone, but distributed between them.



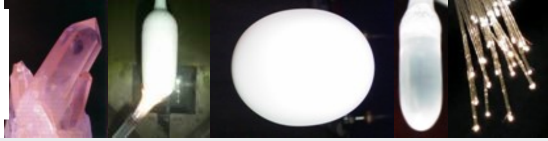
4. Conclusions

- It was shown how a simple optical fiber sensor can be used for the monitoring of a multi-concentrated colloidal system;

- An environment comprised of different sedimentation zones, suitable for the simulation of concentration step disturbances was created;

- The **method has showed itself reliable for the monitoring of such environ**ment, providing **data that can be interpreted both in terms of data dispersion or of the decay rate of the autocorrelation function**, allowing the evaluation of the concentration;

- The information collected with the system show that **the real concentration values are very different from the ones estimated by the simple sedimentation models traditionally applied**.



Thank you for your attention!

Questions?

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