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Highly sensitive quantification of aquatic nanoparticles by LIBD

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Introduction

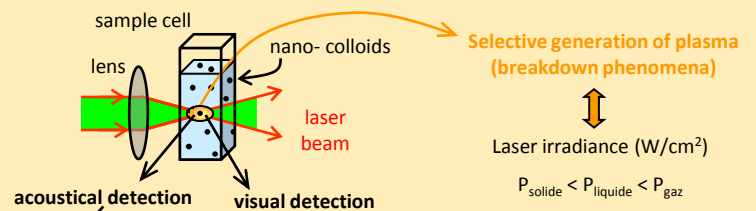
All natural aquatic systems contain nanoparticles which can be inorganic, organic or even include microorganisms. The quantification of these nanoparticles presents considerable difficulties as they are often present at very low concentrations and with a predominance of the size distribution over small diameters (< 100 nm). The methods traditionally used for their characterization are based on separation and/or fractionation (Ultrafiltration, Ultracentrifugation, Size-Exclusion Chromatography, Flow Field Fractionation...) but also on Static and Dynamic Light Scattering methods.

However, these techniques have important limitations: separation and fractionation interfere with the colloidal system and easily lead to changes in the colloidal population (coagulation, sorption and precipitation) while Light Scattering has relatively high detection limits for particles with an average diameter of less than about 100 nm.

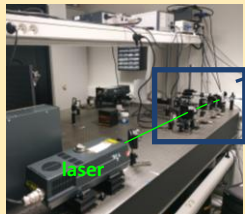
Alongside these techniques, LIBD (originally invented to monitor ultrapure water in the semiconductor industry) was rapidly used and improved for the detection and characterization of synthetic or natural aquatic nano-colloids.

Principle

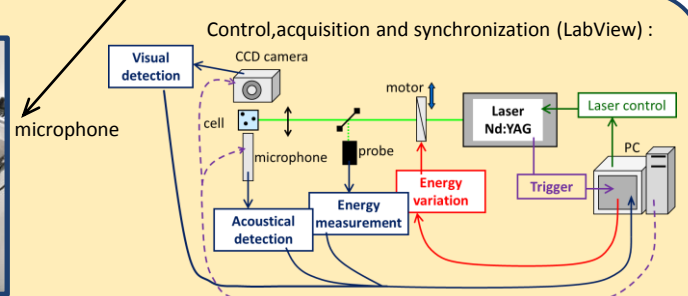
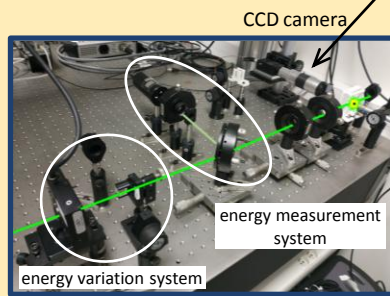
This technique uses a high-energy pulsed laser beam that is focused in a measuring cell to selectively generate a plasma when solid matter (the nano-colloid) interacts with high-intensity light (the laser beam). Absorption of photons by the nanoparticles during the laser pulse leads to the heating and expansion of the plasma, which can then be detected acoustically or optically via, respectively, the propagation of the shock wave in the medium or the emitted light.



Experimental set-up

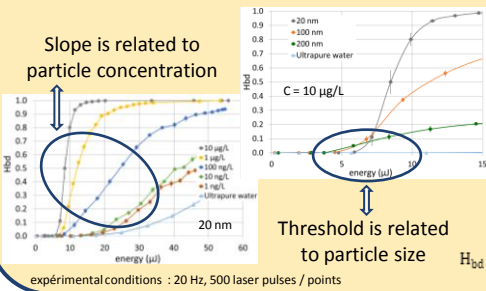


Nd:YAG laser technical characteristic
 $\lambda = 532 \text{ nm}$, $E_{\text{max}} = 10 \text{ mJ}$, $\tau = 10 \text{ ns}$, $f = 1 \text{ to } 20 \text{ Hz}$.



Calibration

Calibration of the system is made from standard solutions constituted by nano-sphere of polystyrene which sizes vary between 20nm and 1µm and for concentration going up to the ng/L.

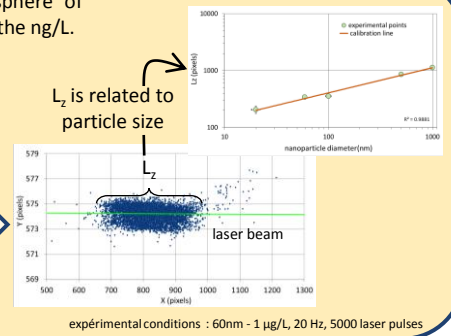


Acoustical Detection

"S" curves are obtained by plotting Breakdown probability (H_{bd}) versus laser's energy

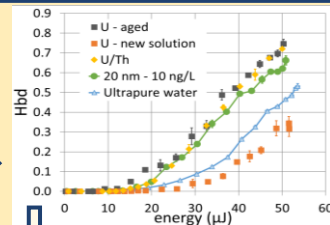
Visual detection

"Butterfly" curves are obtained from the mapping of each plasma generated by a nanoparticle



Results

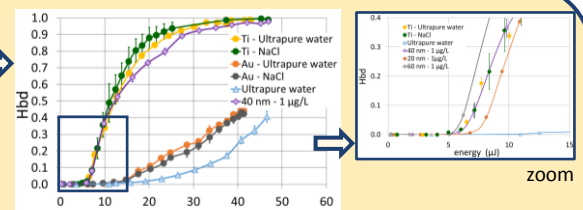
LIBD is used here to detect the presence (or absence) of colloids in U (VI) solutions (after acid reduction and/or aging) and U/Th solutions (after centrifugation)



- ⇒ Size below 20 nm for U (new) solutions, the influence of solution aging is visible in the subsequent appearance of colloids
- ⇒ Low concentration and size of colloids for U/Th (approx. 20nm - 10ng/L)

For more informations about this study, you can go to see the poster: PA2-4 SOLUBILITY EVALUATION OF THE COPRECIPITATE URANIUM AND PLUTONIUM OXIDE UNDER HYPERALKALINE AND REDUCING CONDITIONS IN THE CONTEXT OF DEEP DISPOSAL OF ILW RADIOACTIVE WASTES, M. Le Meur and al.

LIBD is used here to detect the presence (or absence) of colloids in standard Ti and Au solutions for SP-ICP-MS experiments



- ⇒ Presence of Ti colloids (whereas it's normally in dissolved form), with an estimated diameter of 40 nm
- ⇒ Absence of Au colloids

Conclusion

Despite certain drawbacks (partial destruction of nano-colloids, limitation to solids, etc.), LIBD has proved to be a reliable and powerful tool for the detection and characterization of aquatic nanoparticles under "extreme" conditions : it enables highly sensitive detection and characterization of nanoparticles with small diameters (from approx. 5 nm, up to 1000 nm) and very low concentrations (from approx. 1 ng/L, up to mg/L).

In addition to sizing and concentration determination, it can also be used for a variety of other purposes, such as kinetics studies, breakthrough curves, colloidal tracings, etc. opening up a vast field of investigation to users.