CORDOUAN Technologies

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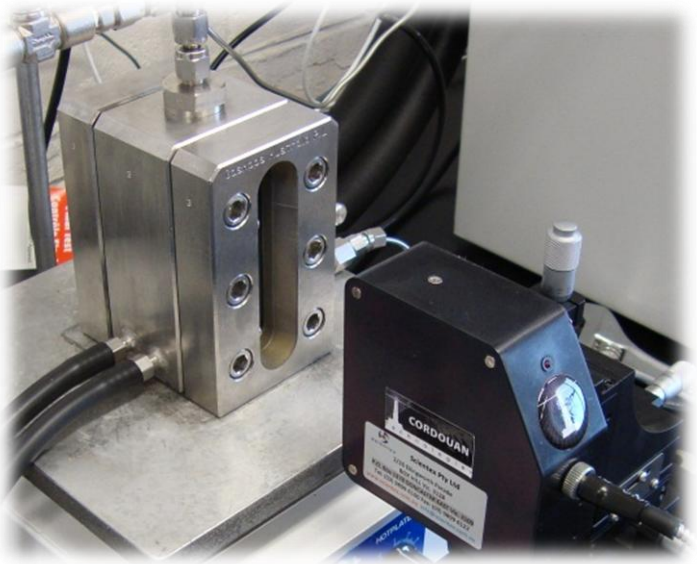
GENERAL MICROTECHNOLOGY & PHOTONICS

VASCO Flex ™ Remote Particle size Analyzer

Nano-particle Size measurements brought into your process!



VASCO Flex™ Analyzer: « in situ » Nano Particle size measurement for process monitoring!

Key Words: *Dynamic Light Scattering (DLS), Nano-Particle sizer, fiber remote probe, In situ measurements, process monitoring, glass capillary.*

Abstract

This note presents our unique fiber remote DLS probe for in situ nano particle size measurements named VASCO Flex™. Developed by CORDOUAN Technologies, VASCO Flex™ is a fully versatile particle size analyzer that makes use of advanced single mode optical fiber to bring DLS measurement to your process. Thanks to its innovative optical fiber remote head which principle is described hereafter, we show how VASCO Flex can be implemented very simply to existing experimental setups like synthesis reactors, or SAXS systems, giving users maximum measurement agility to monitor and improve its process.

Introduction

Since few years now the use of nano particles in the industry is expanding rapidly because of the unique properties brought by nano materials into final products. Thus, as nano-material science progresses, the fields of application are getting broader, ranging from health and medicine, energy, food & agronomy, environment, electronics, advanced material science, aeronautics, cars, paints/ inks,…and it is just the beginning of a new era!


Chemical Synthesis reactor

Among the important properties of nano-particles, size is probably the most critical one because it is intimately related to their physical and chemical properties like specific area/porosity, bio-availability, stability, ability to be functionalized, etc. So, as the number of installations of nano-particle reactors deployed in the field, either in advanced research labs or in fabrication factories, keeps growing fast, the need for reliable and accurate size monitoring tools is getting crucial. In this context and anticipating that demand from the market, CORDOUAN technologies has developed its unique VASCO Flex system.

DLS Measurement principle

Today, many recognized methods exist to measure the size and size distribution of nano-particles [1]. Among these methods, Dynamic Light Scattering (DLS) also known as Photon Correlation Spectroscopy (PCS) is certainly one of the prevalent technique of choice in colloidal sciences. Indeed, DLS is a mature and very powerful technique based on the analysis of scattered light fluctuations caused by the Brownian motion of particles [2, 3]; its principle consists in analyzing the fluctuation of a coherent laser light scattered by particles suspended in a liquid as a function of time and at a given angle. It provides accurate particle size measurements from the nanometer up to a few microns in a minute. Today, different measurement configurations are proposed in commercial DLS set up; in the most common ones, the sample is placed in a rectangular disposal glass or quartz cuvette and the scattered light is usually detected at 90° from incident light direction (see figure 1). In some more recent DLS systems like the VASCO sizer from CORDOUAN Technologies, the measurement cell is integrated directly into the instrument and the light is detected backward direction (135°). In all case, such configuration requires batch sampling; this is acceptable in many laboratory applications, but it is not adapted for in situ and real time process monitoring (time consuming operation, sample stability issues, reproducibility issue, etc). This implies a change of paradigm: **if you cannot bring your sample to the measurement, you have to bring the measurement to your process**; this is basically the philosophy of the VASCO flex.

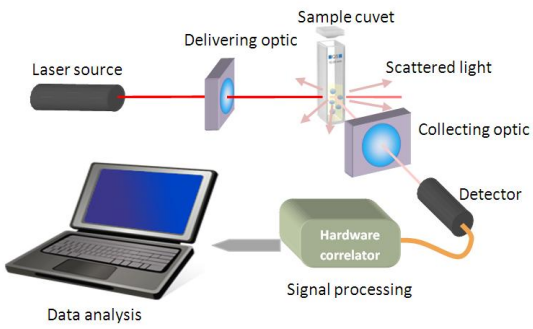


Figure 1: "classical" DLS measurement configuration

The VASCO flex innovative approach

VASCO Flex™ is a unique and fully agile nano-particle size analyzer based on DLS measurement. At the heart of the VASCO Flex system is the Optical Fiber Remote Head (OFRH). The OFRH integrates into the same case housing both the sample illumination optical system and the scattered light collector. The illumination and the collector systems are made of two optimized optical collimators, one used as condenser and the other as a focuser (see figure 2) The OFRH is linked to a central unit with an optical fiber patch cord which integrates two single mode fibers: one Polarization Maintaining fiber used for the laser beam and one standard single mode fiber used to collect the scattered light from the sample to be characterized. The central unit includes a single photon detector (APD or PM), a powerful data processing linear correlator, an external temperature sensor that can be attached to the OFRH, and a highly reliable fiber pigtailed laser diode source with its electrical driver. A PWM temperature controller for Pelletier elements can also be integrated into the central unit for an accurate sample temperature control.

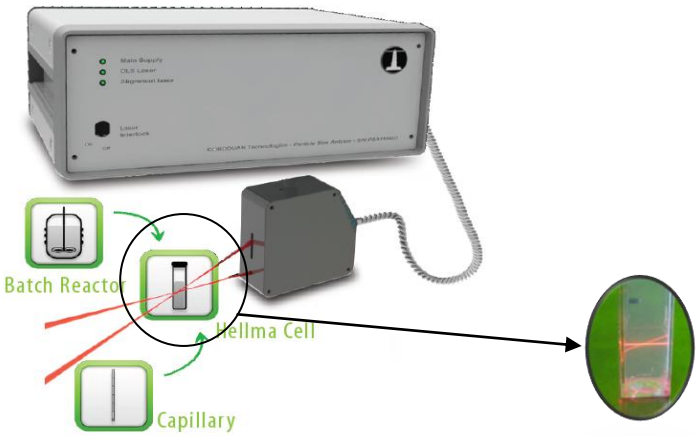


Figure 2: the VASCO Flex and its Optical Fibre Remote Head

The central unit also integrates an alignment laser diode and an optical switch which allows to couple the alignment laser automatically into the signal fiber in order to image the coherence area into the sample cell or reactor and to facilitate installation (see insert of fig 2).

Advantages and Benefits of VASCO Flex

Compact and robust: The VASCO Flex has been designed to be easily integrated into various environments, accounting for user's constraints. Using optical fibers and telecom based technologies, VASCO Flex is a cost effective, robust and compact solution for process monitoring (maintenance free); The use of single mode optical fiber naturally acts as a spatial filter that saves space and cost of additional parts like those needed in conventional DLS system. Also light propagation in an optical fiber is much more robust and immune to stray light and external perturbation than free space optical layout

Agility : Its working distance (distance between the OFRH and the measurement area) can be easily tuned from 4 cm up to 40 cm, allowing non contact and in situ measurements, ie, without tedious batch sampling (time and cost saving). Also, the fiber patch cord length can be specified to any length between 1 to 20 m; this is a real advantage for remote operation in difficult environments: the Optical remote head can easily be placed in a vacuum chamber or inside a glove box.

Easy to implement: With its alignment laser, the OFRH can be implemented and adjusted very easily, even for a non experienced user. OFRH can also be mounted on XYZ translation stages for fine positioning of the probe.

Versatility: thanks to its design the VASCO flex can accommodate various measurement configurations: in situ measurement inside a reactor through a glass window, in a standard disposable square cuvette, in cylindrical glass capillaries with diameter as low as 1 mm, in a sample drop, etc.

Highly reliable Data analysis: the VASCO flex system is powered by the **NanoQ 2.0** software with its unique Pade Laplace inversion algorithm; combined with multiple acquisition capability and measurement replay, this makes VASCO flex an ideal tool for size kinetics monitoring.

Examples of VASCO Flex integration

Below are two examples of VASCO flex integration into existing setup: The first one is the coupling of DLS measurement to a commercial SAXS bench. This system allows simultaneous DLS +SAXS measurement of a sample into the same glass capillary; Such coupling could be very interesting in application like the one described in ref [4] and [5] where simultaneous measurement by complementary techniques allows a better understanding of the nano-particles behavior and structure.

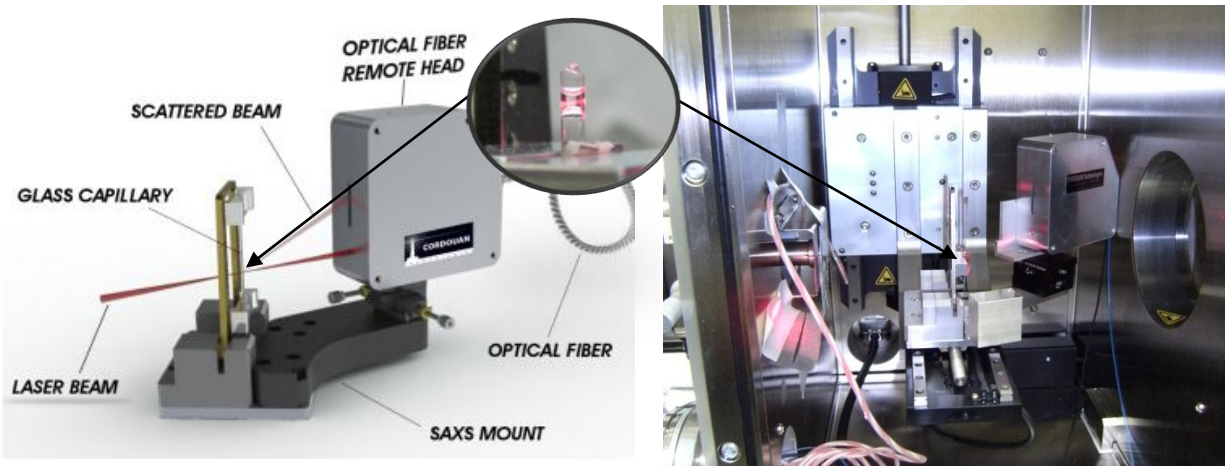


Figure 3: the VASCO Flex and its Optical Fibre Remote Head coupled to a commercial SAXS measurement bench

The second example is the integration of the VASCO Flex to a high pressure (80 bars) chemical reactor system used for the synthesis of polymer nano-particle under supercritical CO_2. The setup is presented in figure 4. In this configuration the reactor has two transparent side walls made of 2 cm thick glass windows. The laser beam is focused in the center of the reactor through the front glass window. The backscattered light is detected through the same window. In the setup, the distance between the reactor and the OFRH is about 15 cm.

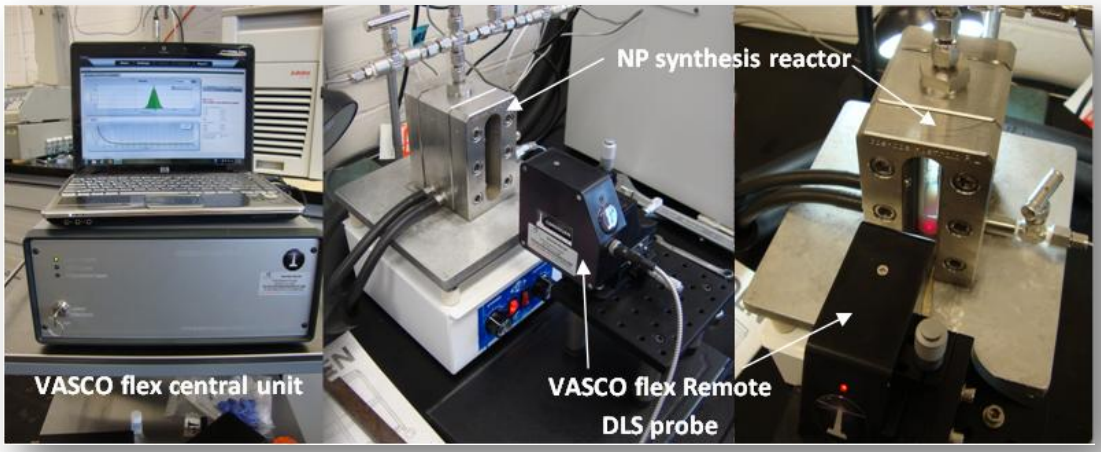


Figure 4: the VASCO Flex and its Optical Fibre Remote Head coupled to a nano-particle polymerization reactor;

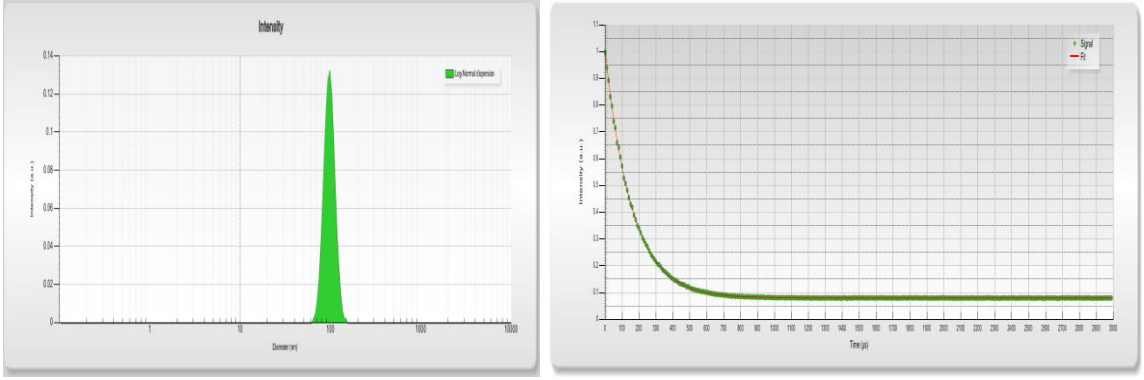


Figure 5: Set up validation for in situ measurement with a 100 nm calibrated latex standard

CONCLUSION

Developed by CORDOUAN Technologies, VASCO Flex™ is a unique fully versatile particle size analyzer that makes full use of advanced single mode optical fiber to bring DLS measurement to your process. Thanks to its innovative optical fiber remote head VASCO Flex can be implemented very simply to existing experimental setups like synthesis reactors, or SAXS systems, giving users maximum measurement agility to monitor and improve its process.

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