

# PROPAT

INTEGRATED PROCESS CONTROL

## Validation of Online Particle Sizing Sensor for Process Control

T.A. Hazlehurst, F.M. Mahdi, T.N. Hunter, A. Hassanpour, & F.L. Muller  
Institute of Particle Science and Engineering, School of Chemical and Process Engineering,  
University of Leeds, Leeds LS2 9JT, UK  
t.hazlehurst@leeds.ac.uk

### Overview

Validation process overview including partners and their roles as part of ProPAT

Instrument designed and built by IRIS with initial testing of PSD measurement



UNIVERSITY OF LEEDS

Laboratory pilot scale controllable dry and wet particle flow rigs built for validating instrument in particle flow systems for process control

Online implementation of instrument for process control with ProPAT interface with industrial partner systems



End users

### Instrument validation

We have been tasked with calibrating and validating the Image Scatterometer (ISI) from the Institute of Photonic Sciences (ICFO) (Figure 1) at pilot scale in Leeds (i) a granulation process typically used to formulate drug molecules (Figure 6) and (ii) a gas-solid flow loop (Figure 4) with controlled particle concentration. These will be validated in three ways.

1

Off-line sensor validation

To compare different glass bead samples of known geometry and size distribution to produce a baseline for the accuracy of the ISI system. The comparison will be based on the size distribution, including the D10, D50, D90 and the span of each sample.

2

Direct on-line sensor validation

Continuous operation will be monitored in situ, using at least two comparative online instruments (Mettler FBRM and acoustic probes) positioned in-line to measure the particle size simultaneously

3

Indirect on-line sensor validation

Samples obtained from the continuous system operating and subject to additional comparison tests (off-line, Malvern Mastersizer), to confirm the observations obtained from the direct measurements.

### Dry air flow rig

A flow rig to demonstrate the sensors ability to be used in process control is designed and is under construction (Figure 4). A vacuum pump will be used to control the flow rate of dry particles and the split of the particle size distribution (PSD). The split can be determined by

$$d_h = \sqrt{\frac{r \mu_g}{V \pi (\rho_s - \rho_g)}}$$

(Figure 3) where  $d_h$  is the diameter at where 50% of the particles are removed,  $r$ ,  $\mu_g$ ,  $V$ ,  $\rho_s$  and  $\rho_g$  are the inlet radius of the cyclone, air viscosity, air velocity, and solid and air density respectively. The ISI scatterometer can be placed within the system to measure both the coarse or the fine particle streams leaving the cyclone separator. Each of particle streams will be collected to allow off-line measurement of the collected samples.

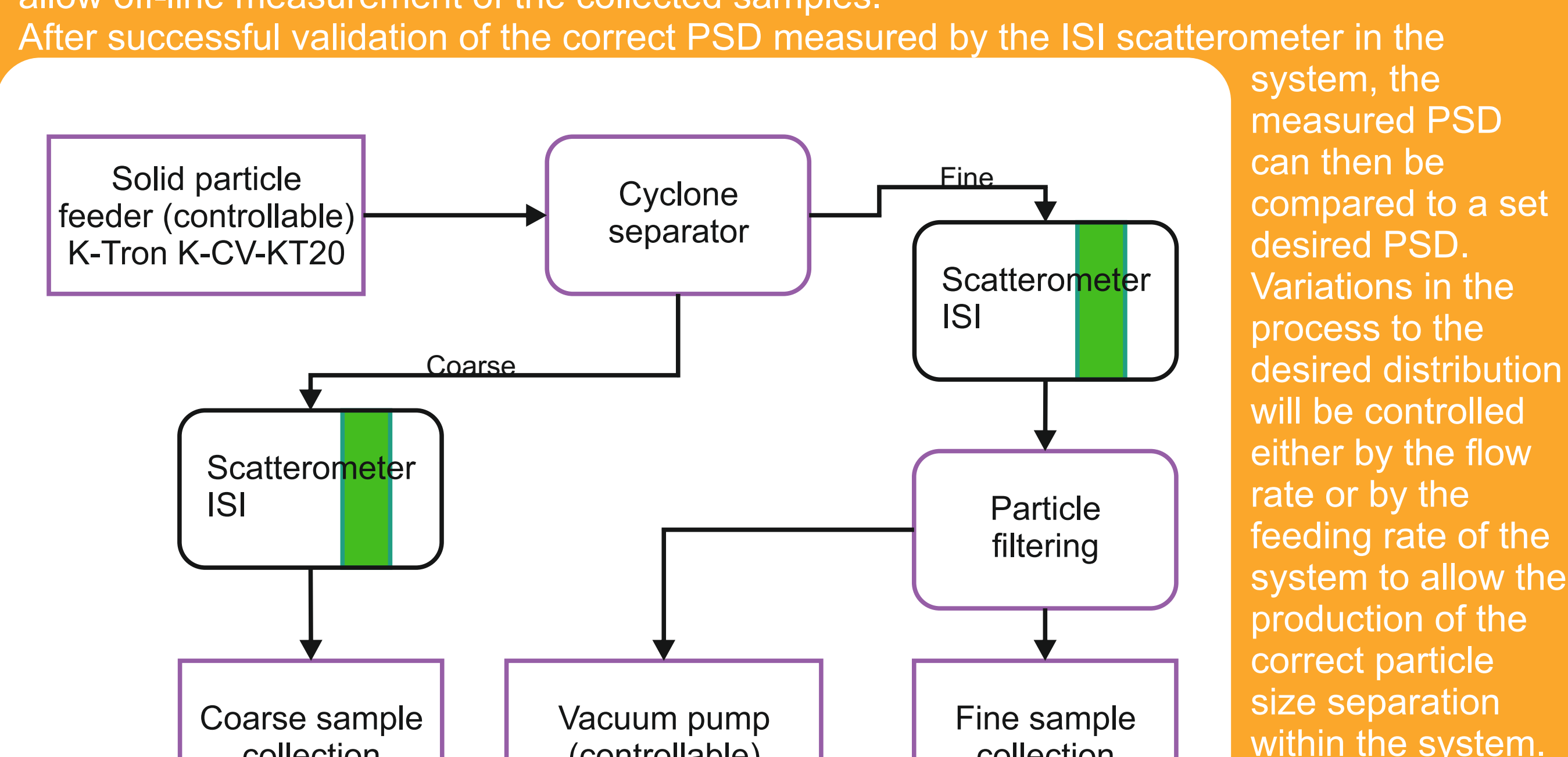
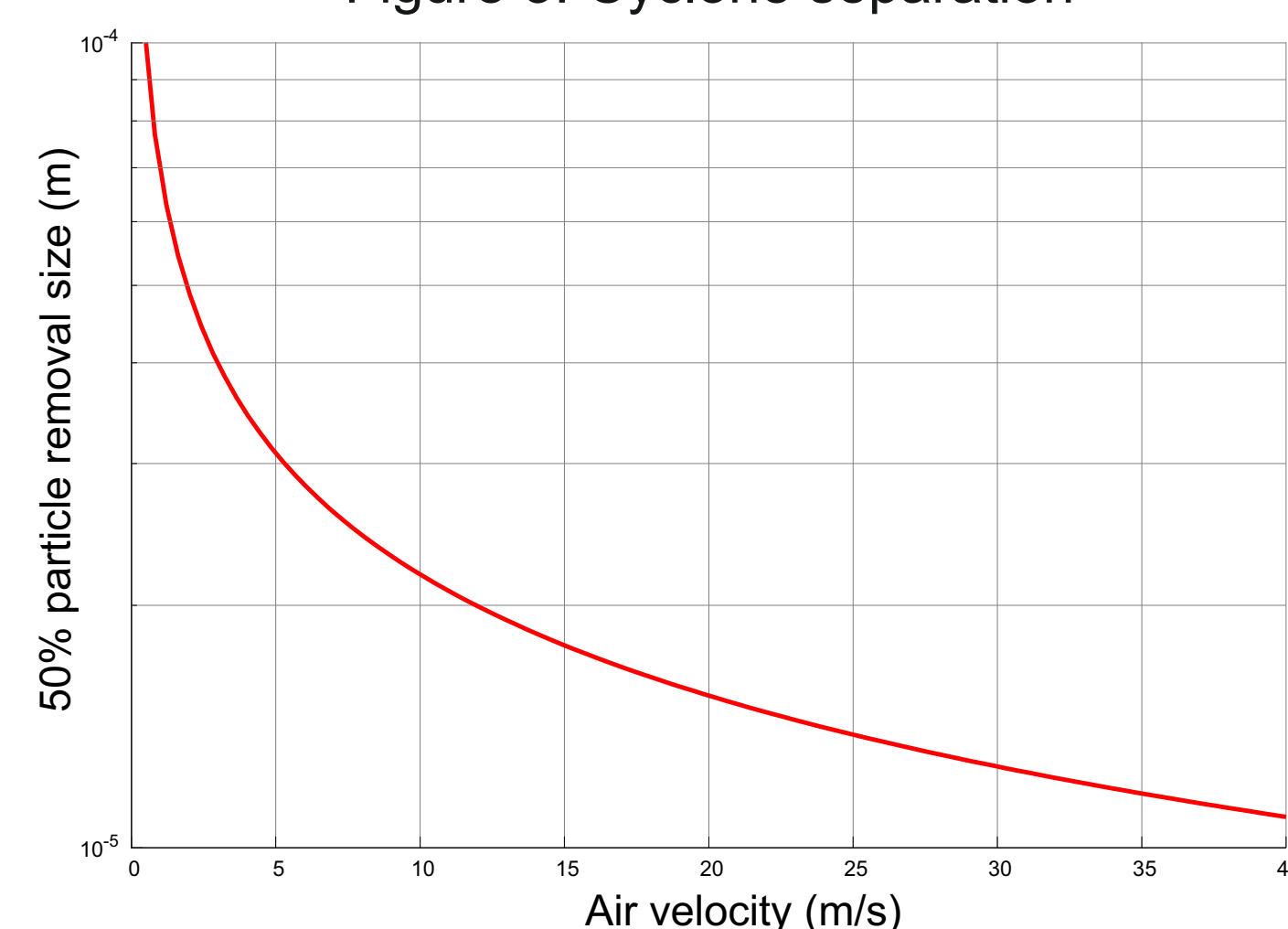


Figure 4: Dry particle flow rig

Figure 3: Cyclone separation



### What is the instrument, and how does it work?

The instrument is a novel light Image Scatterometer (ISI) designed and built the Institute of Photonic Sciences (ICFO) (Figure 1). Leeds have been tasked with validating the use of the instrument in process control for particle flow systems.

It functions by using a low cost LED light source directed through a cell containing the measurement sample. The light passing through the cell is scattered at different angles by the sample particles according to Mie Theory.

The scattered light passes through a filter (Figure 2). This each of the filters holes only allows scattered light from a certain angle range to pass through. Different sizes of particles generate different levels of scattering. The camera captures the different intensity levels that appear through the filter and which is processes to generate the particle size distribution of the sample. This can then be used to control parameters of the flow system.

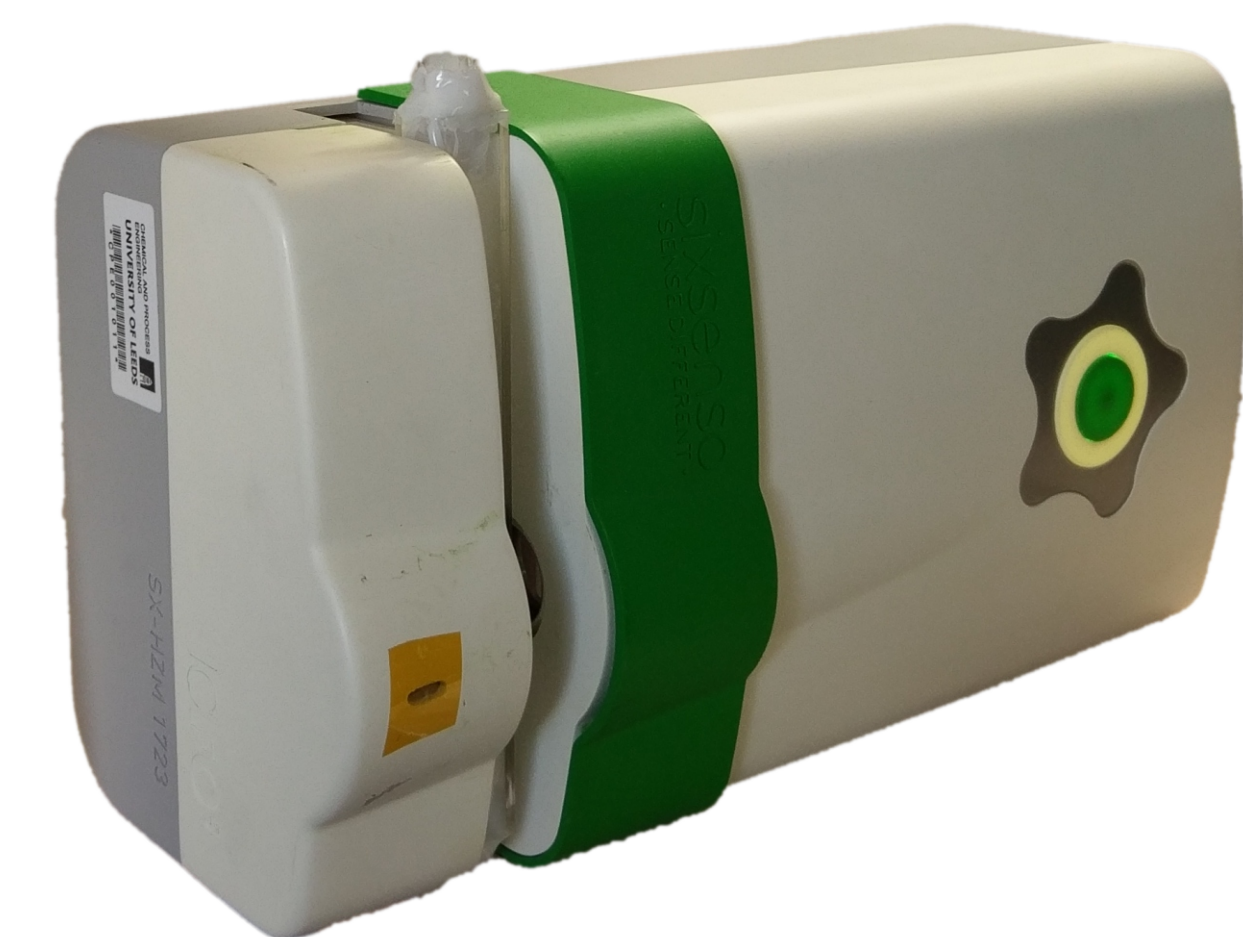


Figure 1: Institute of Photonic Sciences (ICFO), Image Scatterometer (ISI)

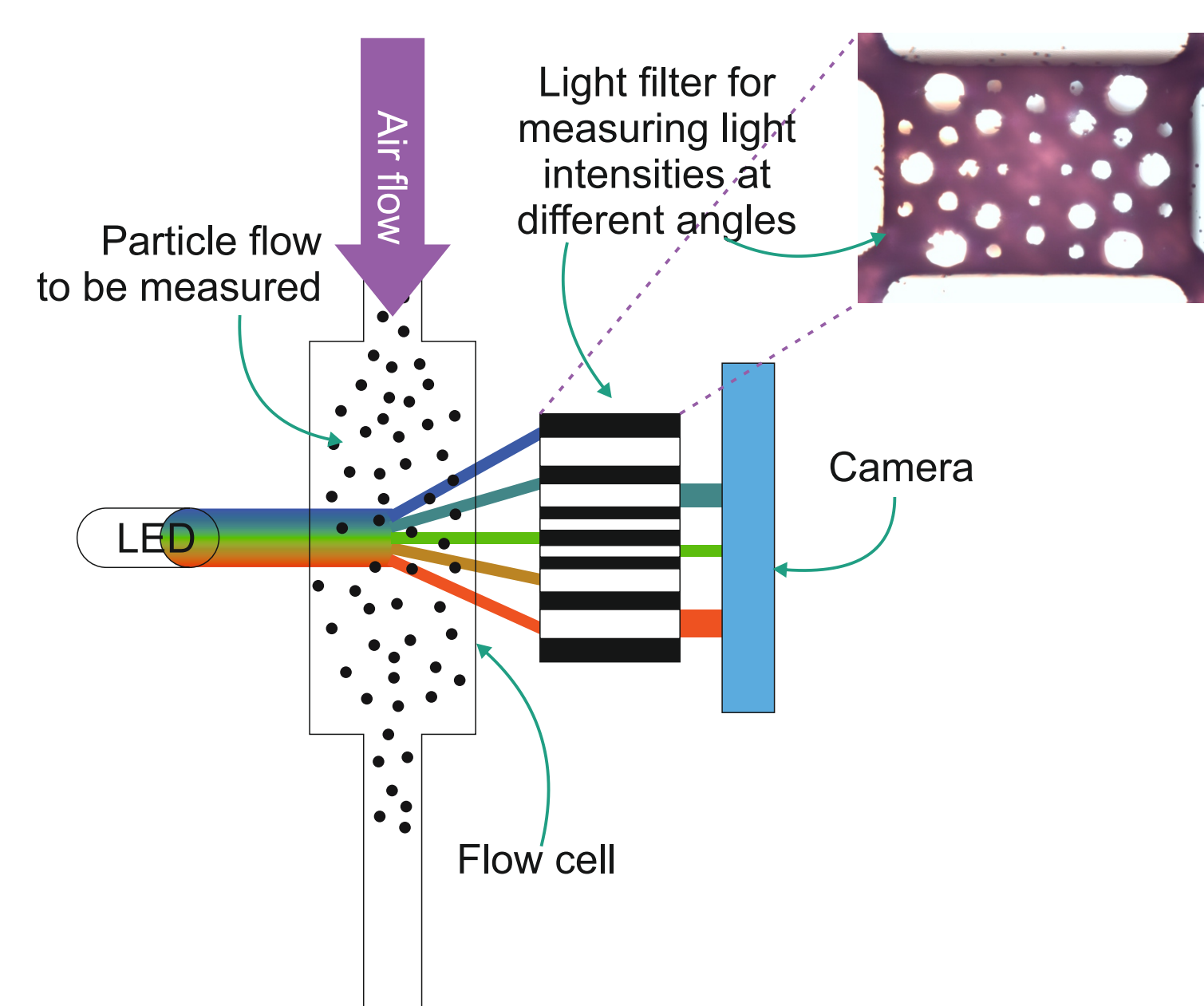


Figure 2: ICFO ISI sensor

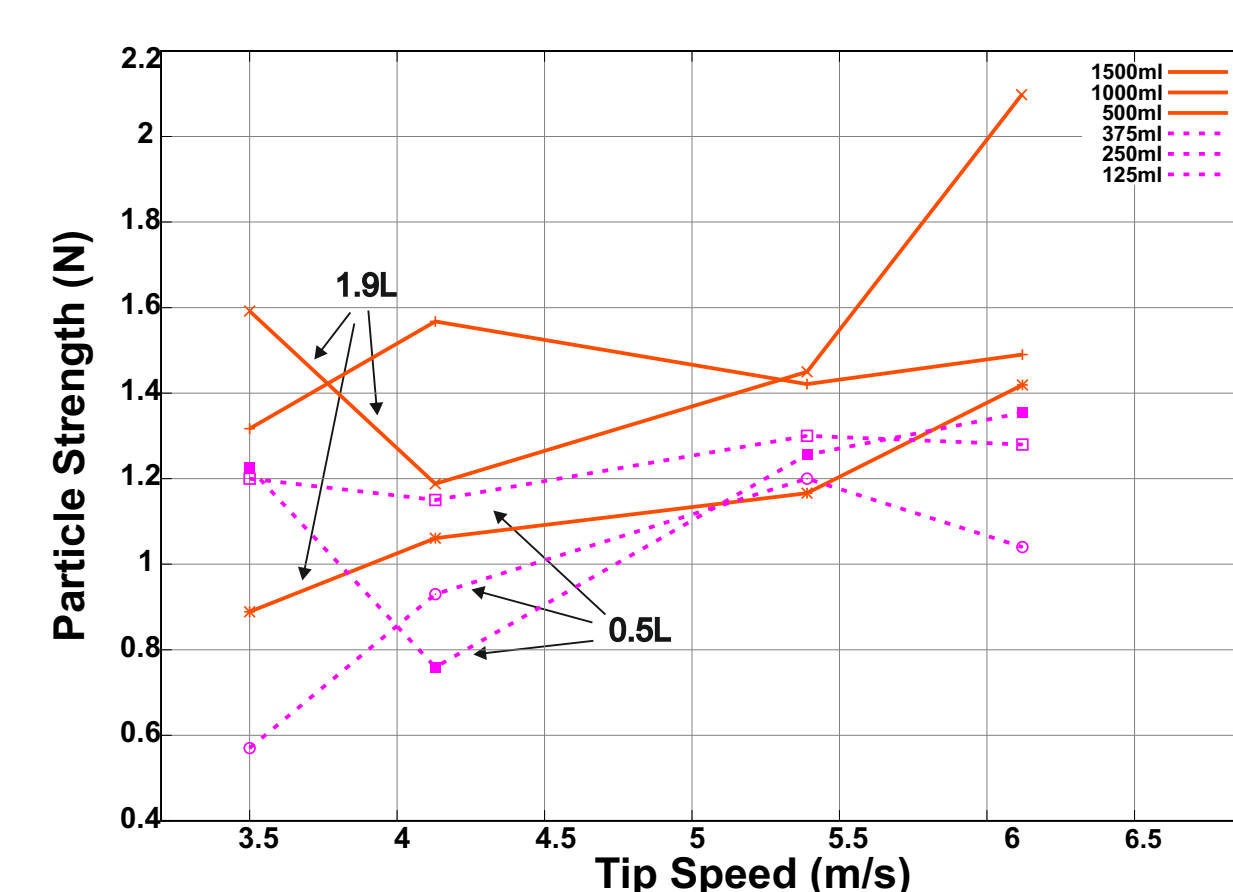


Figure 5: The effect of both scales with various impeller tip speeds and different volumes of material on the granules strength

effect of two different scales (0.5 and 1.9 L) with various impeller tip speed and different volume of material on the granules strength and size have been investigated with the so called seeded granulation process. Figure 5 shows that, at the same scale, increasing the volume of material or speed, shown increase of granules crushing strength. On the other hand the strength increased with increasing the scale while size of granule (D50) reduced. At the same speed and scale, increasing the volume can increase the D50. While at the same volume and scale, increasing speed did not show clear effect on D50. Density of granules did not effecting during the granulation and show similar density for all tests.

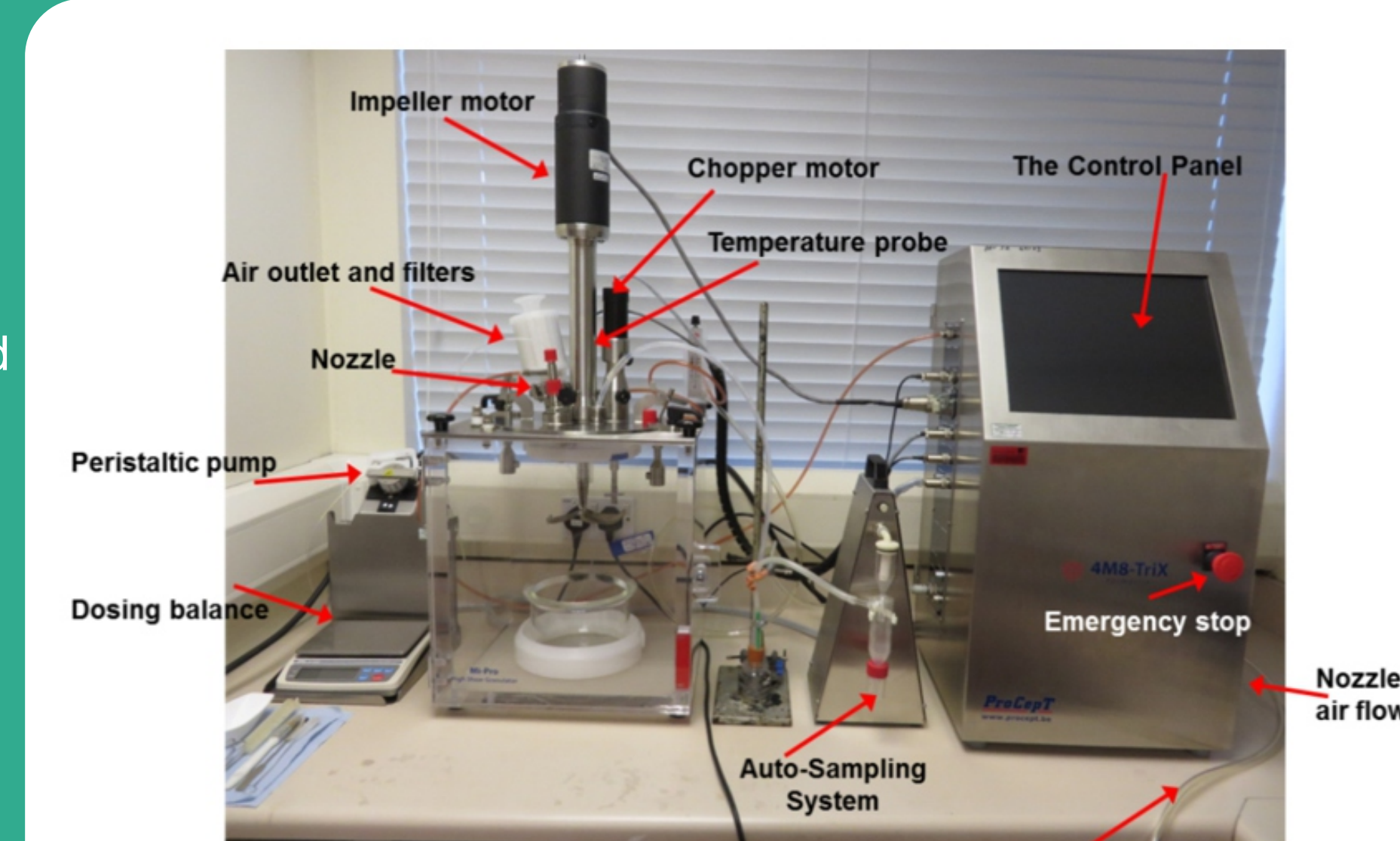


Figure 6: Mi-Pro - High Shear Granulator - ProCepT

### Definition of success criteria

The laboratory scale validation will focus on investigating two main aspects:

The validation of the ISI sensor using a continuous particle flow: The sensor has to be tested for its use in a continuous process. In this case, the main challenge is to check the accuracy in the particle size ranges identified by the sensor.

Process control with the data provided by the ISI sensor: The suitability to perform process control with the data provided by the sensor will be investigated at laboratory scale. For this, a control loop will be implemented, controlling the concentration of particles and flow rate of the system, thus controlling the size fraction obtained by the separator system, using the measurement from the sensor.

