NURF – optimisation of multiple in situ simultaneous, autonomous characterisation techniques with small-angle neutron scattering

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Aims

- Multiple in situ characterization techniques
- High throughput
- Automated flow cell cleaning
- Sample temperature control
- Sample volume minimization

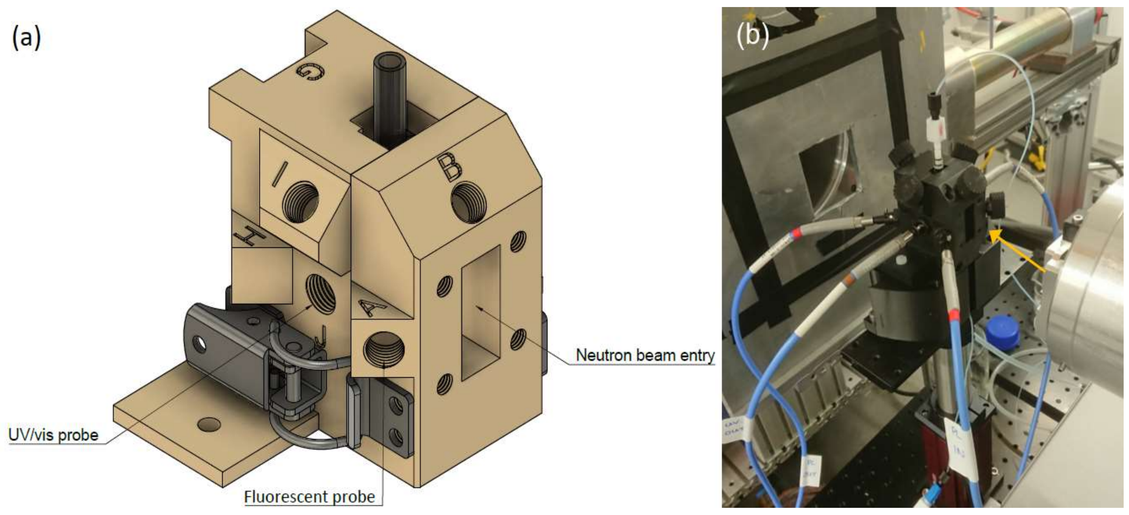

Fig 1. Two part quartz flow cell mount. 10 input ports enables multiple characterization techniques parallel to SANS measurements. Possible probes: UV/Vis, fluorescence, Raman, temperature readings and camera inspection etc.

Hardware setup

Sample delivery

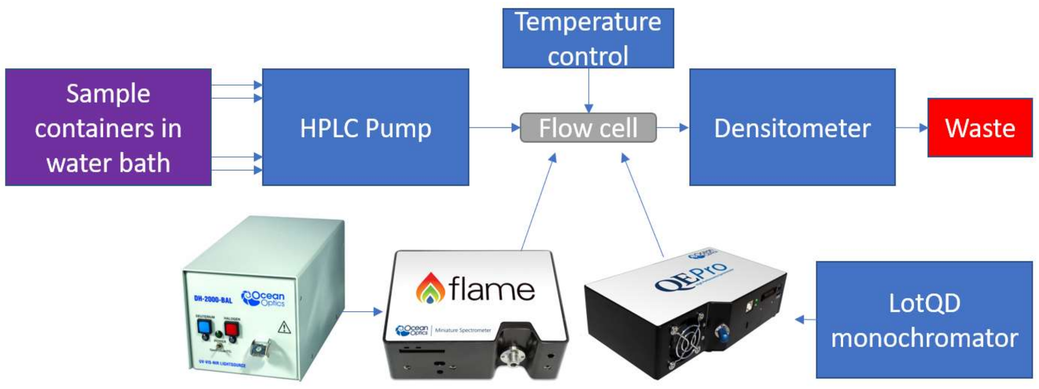

Fig 2. Schematic hardware setup. Sample delivered by HPLC pump to the flow cell, where SANS, UV and FL measurements are done. In-line densitometer provides additional data before sample is pumped to waste. Temperature control system is presented in Fig 3.

Temperature control

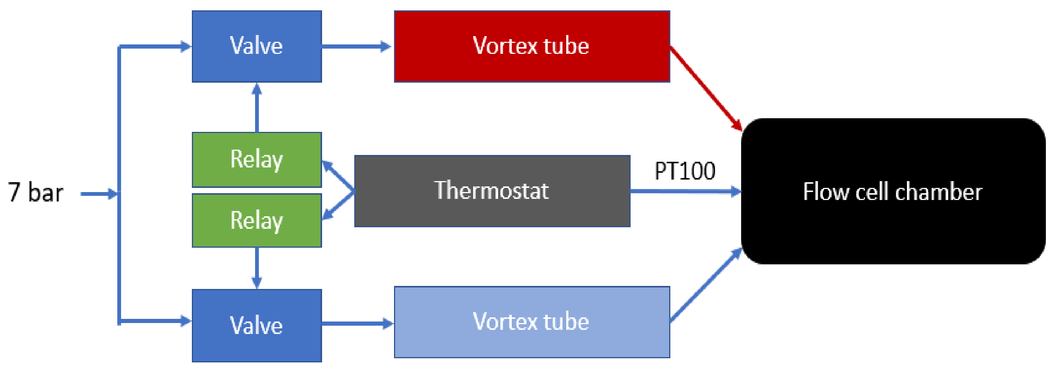

Fig 3. Schematic temperature control setup. Pressurized air flow is used to generate a hot and a cold air stream with the help of vortex tubes. Flow through the vortex tubes are controlled by solenoid valves. The air is lead into to flow cell chamber. Since pressurized air has a low moisture content, condensation on the flow cell is minimized.

Results

SANS

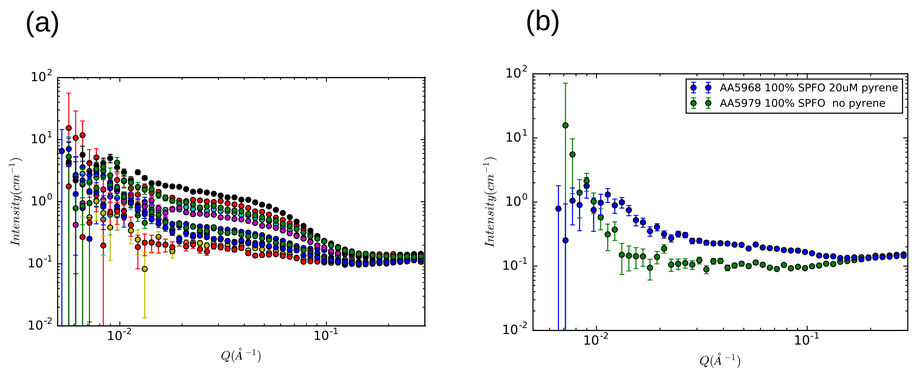

Fig. 4. SANS results measured at Zoom (ISIS facility, UK) for (a) a series of compositions from 100% Brij 35 to 100% sodium perfluorooctanoate (SPFO) at 5 mM in D_2O and (b) comparison of SANS for the SPFO with and without pyrene (20 μM) showing that the fluorescent probe alters the micelles.

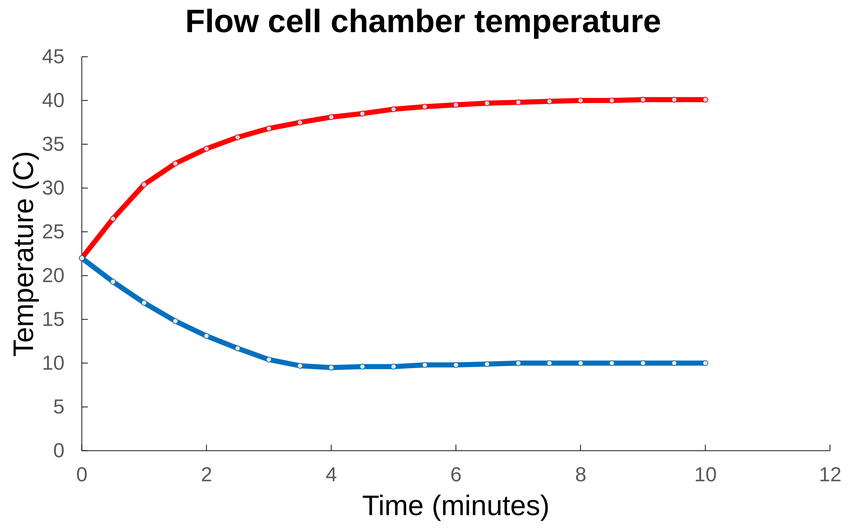

Fig 5. Temperature control capabilities. Set temperature is within 0.2 °C of setpoint after 7 minutes

In situ optical spectra

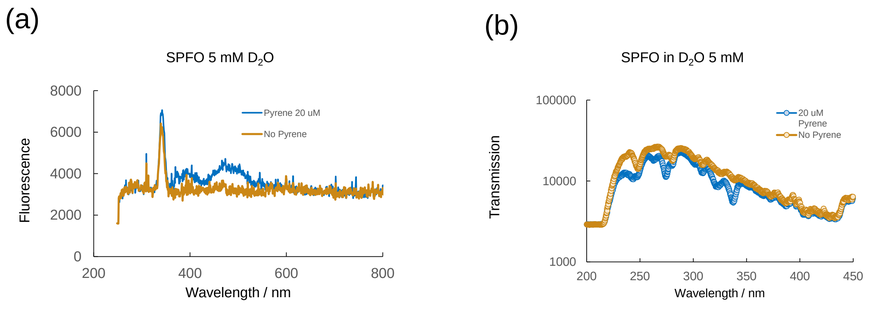

Fig. 6. Optical spectra measured in-situ during SANS data acquisition (a) fluorescence and (b) absorption in the UV/vis region.

Conclusions

- Multiple in-situ characterization techniques are viable and provide useful complementary information
- Vortex tubes provides heating and cooling capacity, eliminating condensation problems

Future developments

- Automated cleaning cycles
- Plug flow to minimize required sample volumes
- Flow cell geometry and connections needs optimization
- Additional probes, pH, IR, Raman, etc.