Liquid Jet Characterization and Optimization

SLAC NATIONAL ACCELERATOR LABORATORY

Chuying Huang[1], Tim van Driel[2]

[1] Department of Chemistry, Northwestern University, Evanston, IL. Contact: chuyinghuang2027@u.northwesten.edu
[2] Linac Coherent Light Source, SLAC National Accelerator Laboratory, Menlo Park, CA. Contact: timbvd@slac.stanford.edu

Background

Linac Coherent Light Source (LCLS), as a Free-electron Laser (FEL), generates intense pulses of X-rays, which can be used to study the fundamental structures and dynamics of materials at the atomic level. Since the X-ray pulses are bright enough to destroy the probed sample, it is essential to develop sample delivery methods that avoid probing damaged samples. Liquid Jets provide a way to create a thin and stable stream of sample material, which guarantees sample replenishment after each X-ray pulse. Both cylindrical jets and sheet jets can be used in X-ray experiments and it's important to characterize and optimize the flow conditions in order to obtain high-quality and reproducible data. This study investigated different parameters that affect the liquid jet performance. Together with the newly developed liquid jet test setup at ASC, improved documentation, and a user "quick guide", which pave the way for reliable user support and operation.

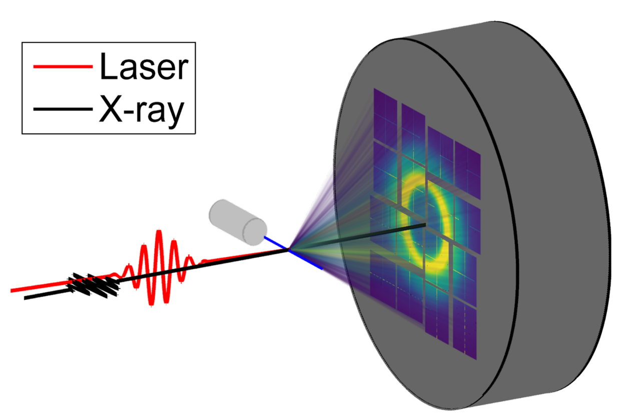


Fig 1. Experimental schematic for time-resolved X-ray scattering typically deployed at LCLS.

Cylindrical Liquid Jet

Introduction: A cylindrical jet is created by a continuous stream of liquid flowing through a nozzle with the aperture defined by a fused-silica capillary. The jet size is thereby determined by the inner diameter of the capillary. The produced jet has a stable (laminar) region and a turbulent region in the flowing stream. It is important to determine and optimize the stable region for different solvents to improve stability and fidelity of the recorded data.

Experiments:

- 10 Solvents: ACN, Chloroform, Methanol, Toluene, DMF, Water, Cyclohexane, Ethanol, DMSO, IPA
- 3 Capillaries (1.2 cm) : 30μm, 50μm, 100μm
- Constraints: 50 psi < pressure < 1500 psi

Results:

- Higher-viscosity fluids tend to have more stable jets; longer stable lengths can be obtained before they break down.
- Higher flow rates give longer stable lengths within a limit; there is a linear relationship between flow rates and length.
- Higher pressures are needed for high-viscosity fluids to flow.

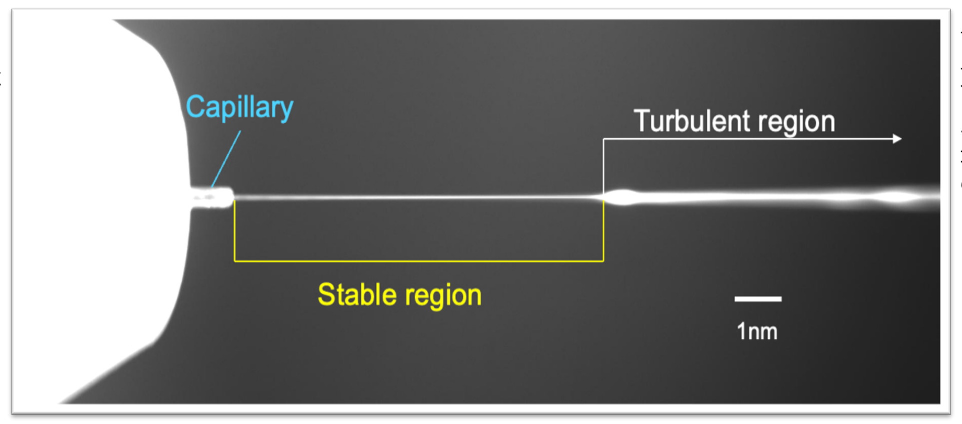


Fig 3. Cylindrical jet with water as the sample, running at a flow rate of 1.8 mL/min. The capillary size is 50 μm and the stable length is 8.7 nm.

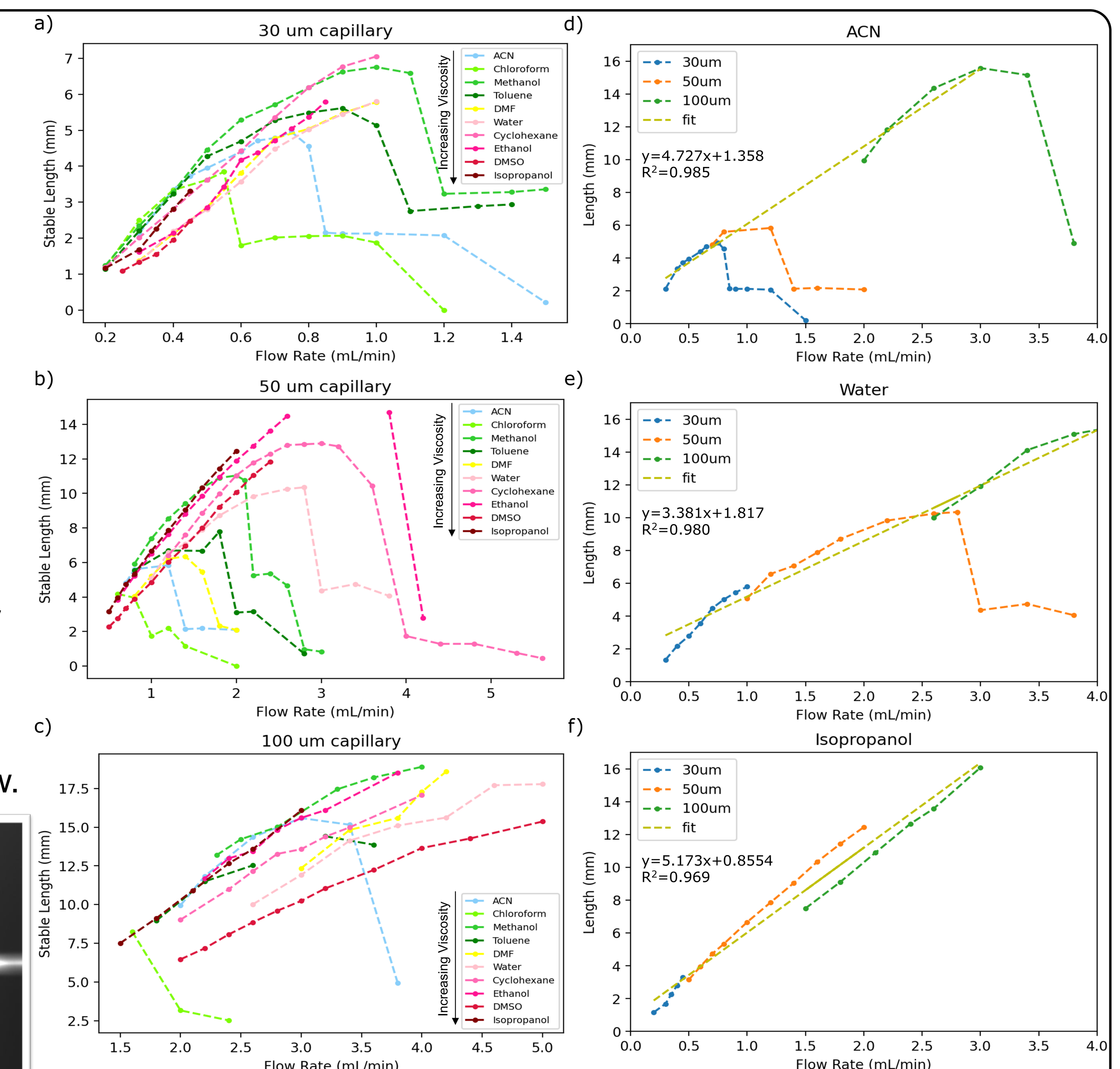


Fig 4. Stable cylindrical jet length vs. Flow rate for different solvents with different capillary diameters. a) 30 μm b) 50 μm c) 100 μm d) Acetonitrile e) Water f) Isopropanol.

Experiments at XCS

Time-resolved X-ray scattering:

- Laser pump: 800nm
- X-ray probe: 8.9keV
- Time resolution: 100fs

$$Q = \frac{4\pi \sin(\theta)}{\lambda}$$

2θ: scattering angle
λ: X-ray wavelength

Results:

Recent LCLS experiment studying the impulsive Raman excitation of liquid water

- Isotropic signal → Heating
- Anisotropic signal → Vibrational response

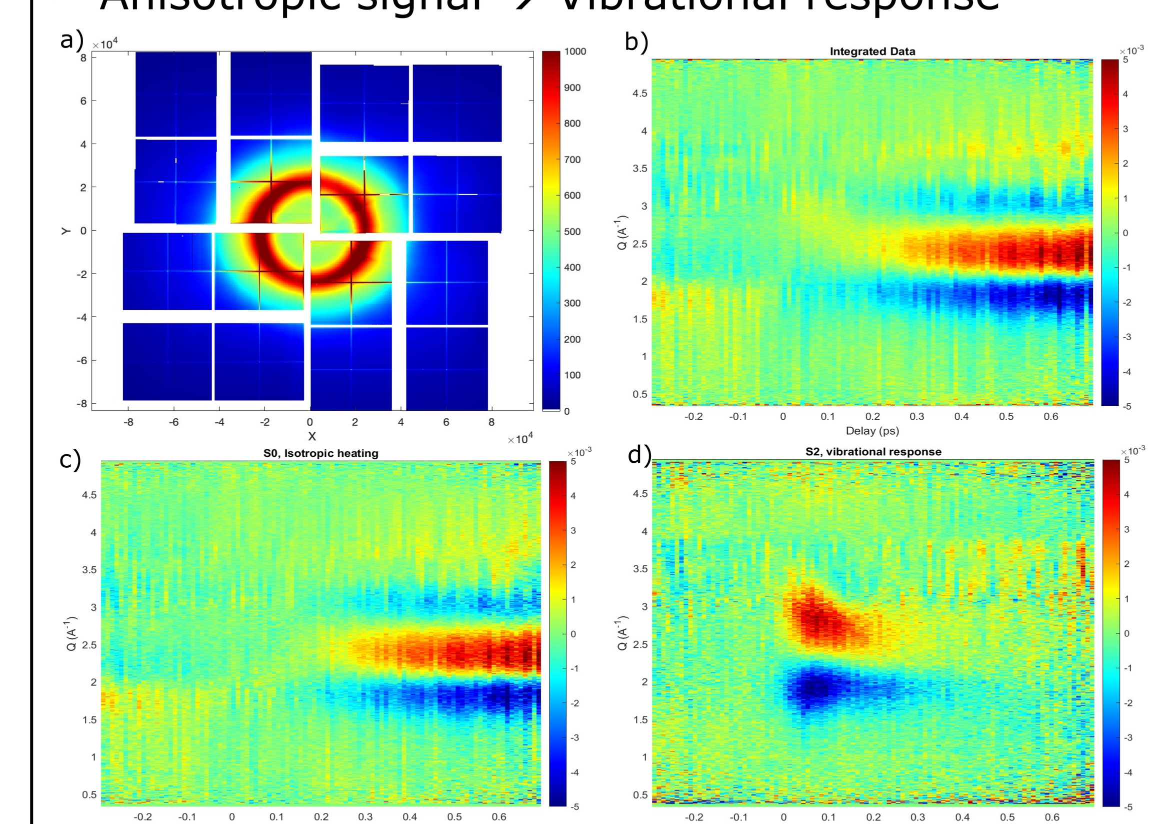
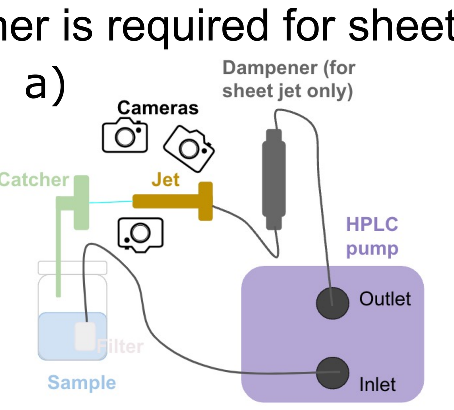


Fig 8. X-ray Scattering of water. a) Average signal of the detector. b) ΔS Integrated signal of X-ray scattering. c) S_0 component of the signal, which represents the isotropic heating. d) S_2 component of the signal, which represents the anisotropic vibrational motions.

Experimental Setup

Liquid jet setup mainly consist of a sample, a HPLC pump, and a nozzle. A dampener is required for sheet jets to minimize pulsation. Cameras are used to monitor the length and stability of the liquid jet and a laser is used to determine the breakup point of the cylindrical jet.



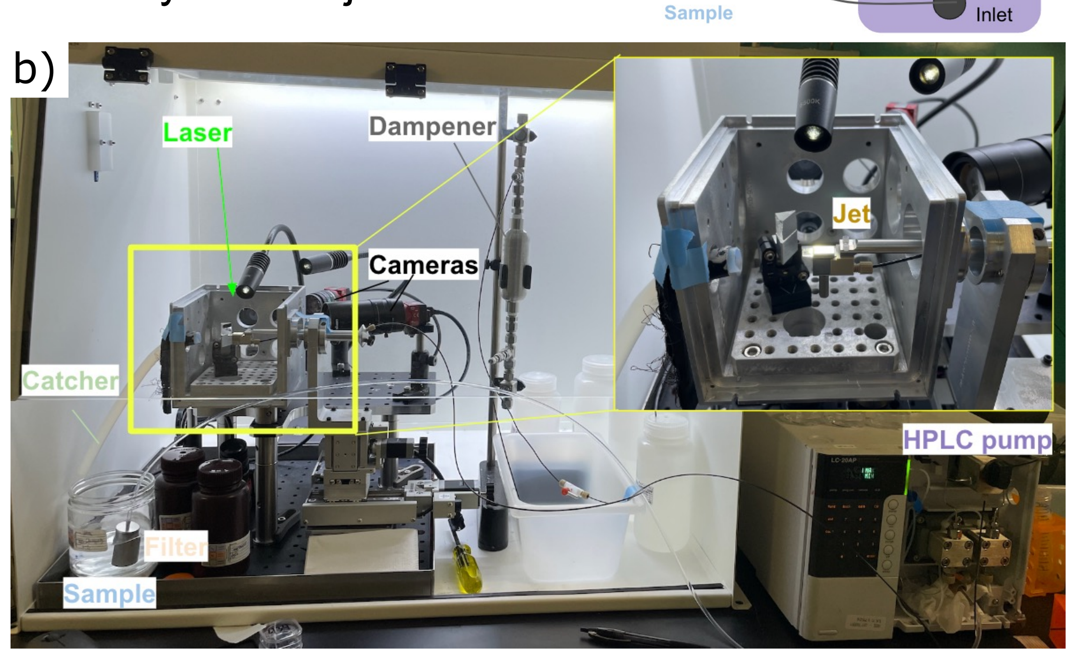


Fig 2. Liquid jet setup. a) Simplified scheme of the setup. b) Actual setup in the lab under the fume hood for testing.

Sheet Liquid Jet

Introduction: Sheet jets provide a larger surface area and a thinner thickness than cylindrical jets. With intense XFEL pulses, cylindrical jets blow up due to high laser fluence. With larger sample areas in sheet jets, the incident beam can be defocused to decrease the fluence to avoid sample damage. The thinner sheet thickness also helps X-rays transmit through the samples and can improve time resolution. Generally, nozzle geometries and flow rates determine the sheet thickness and quality.

Experiments: Water is tested in 4 converging chips (300 μm, 434 μm, 623 μm, 800 μm)

Results:

- Higher flow rates lead to longer sheet lengths.
- Chips with smaller outlet widths can produce longer sheets.
- The widths of the sheets are dependent on the converging angles.

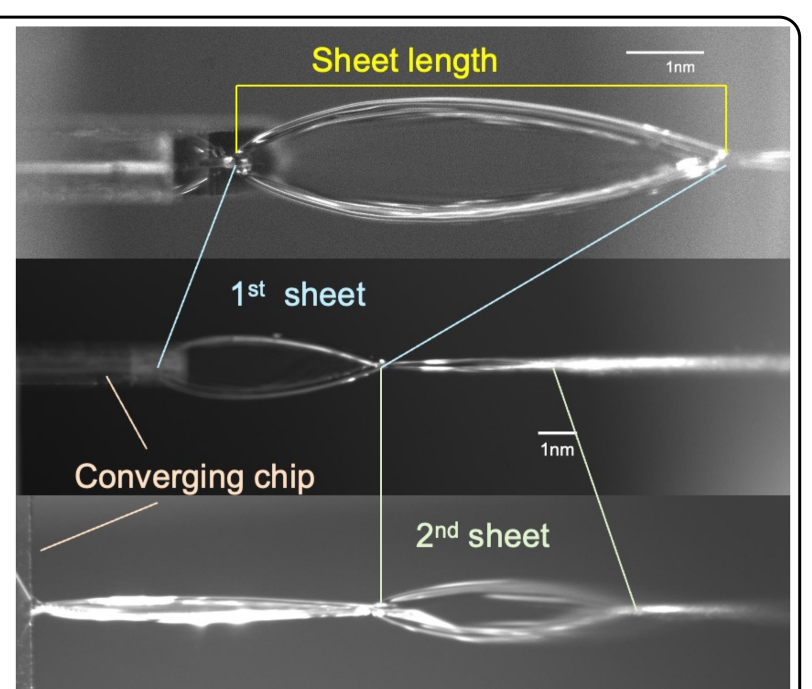


Fig 5. (above) Pictures of sheet jet running water at 20 mL/ min from 3 cameras showing the 1[st] and 2[nd] sheets.

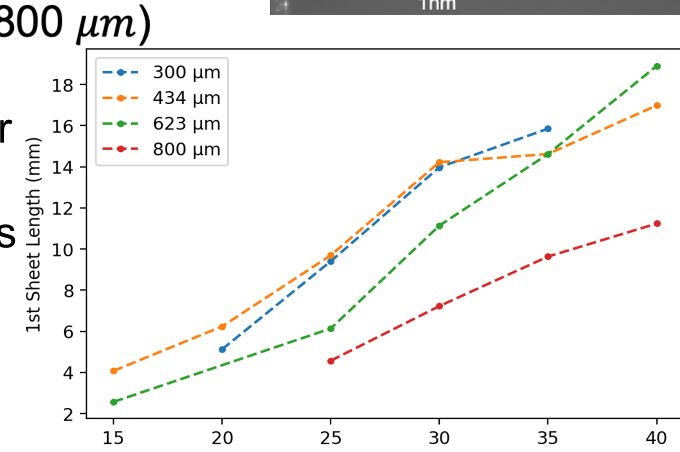


Fig 6. (left) 1[st] sheet length vs. flow rate for chips with different outlet widths.

Prepare for Users

Hutch Box

- Tool kits for liquid jet setup

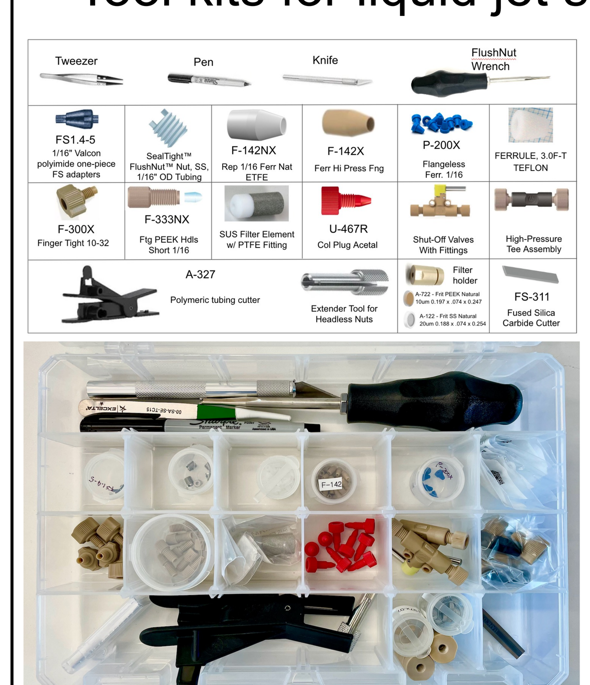


Fig 7. A list of the toolbox and a picture of the toolbox. It includes essential parts in setting up the liquid jet, such as adaptors, ferrules, filters, valves, cutters, wrenches, tweezers, etc.

One-page document

- Procedures of the liquid jet setup and the summary of preferred flow rates

Conclusions

Both cylindrical jets and sheet jets were tested under different conditions. The parameters for optimal conditions were tabulated and a "hutch box" was prepared for future users. This project helps onboard users and save time for optimization prior to experiments with our established testing protocol. I also participated in beamtime at XCS and learned the analysis of X-ray scattering. In the future, general physical models should be developed to better describe the sample performance. Overall, the project has been conducted successfully and it will have an important impact on a large number of user driven solution phase experiments at LCLS.

Acknowledgments

I would like to express my deepest appreciation to my mentor, Tim van Driel for his invaluable guidance, support, and encouragement throughout my internship. I would also love to thank the LCLS SED team for all their support.