DICMaPI
Dipartimento
di Ingegneria Chimica,
dei Materiali e della
Produzione Industriale
Università degli Studi
di Napoli Federico II

INDUSTRIAL BIOENGINEERING

SensApp

Consiglio Nazionale
delle Ricerche

Engineering of pyro-electrohydrodynamic effect for microrheological characterization

Dr. Francesca Setaro

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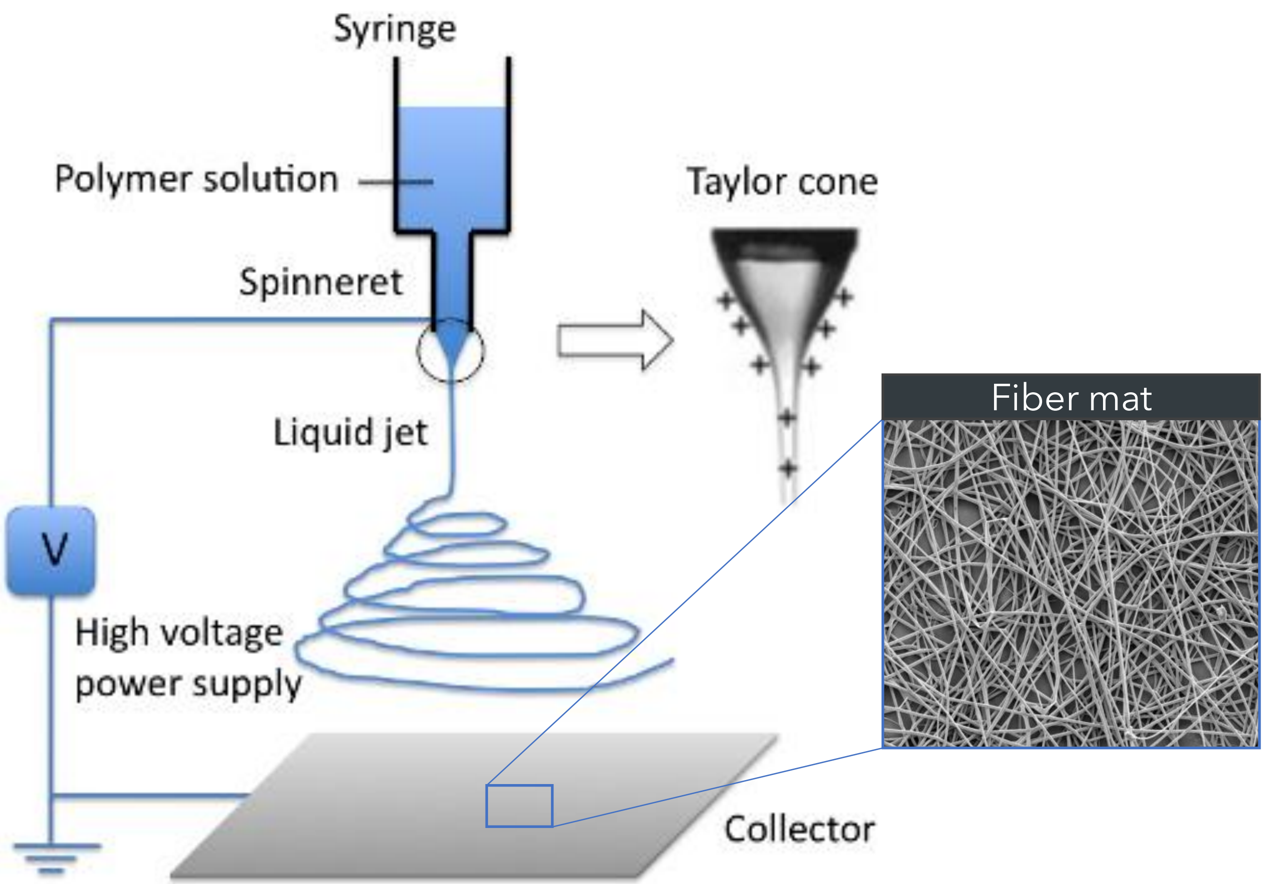
INTRODUCTION

ELECTRIFIED JET – Electrospinning

Electrospinning, a spinning technique, is a unique approach using **electrostatic forces** to produce fine fibers from polymer solutions or melts and the fibers thus produced have a thinner diameter (from nanometer to micrometer) and a larger surface area.



Nandana et al. Electrospinning: A fascinating fiber fabrication technique, Biotechnology Advances, Volume 28, Issue 3, 2010.
R. Rošic, J. et al., The role of rheology of polymer solutions in predicting nanofiber formation by electrospinning, European Polymer Journal, Volume 48, Issue 8, 2012, Pages 1374-1384

STATE OF THE ART – Taylor cone formation

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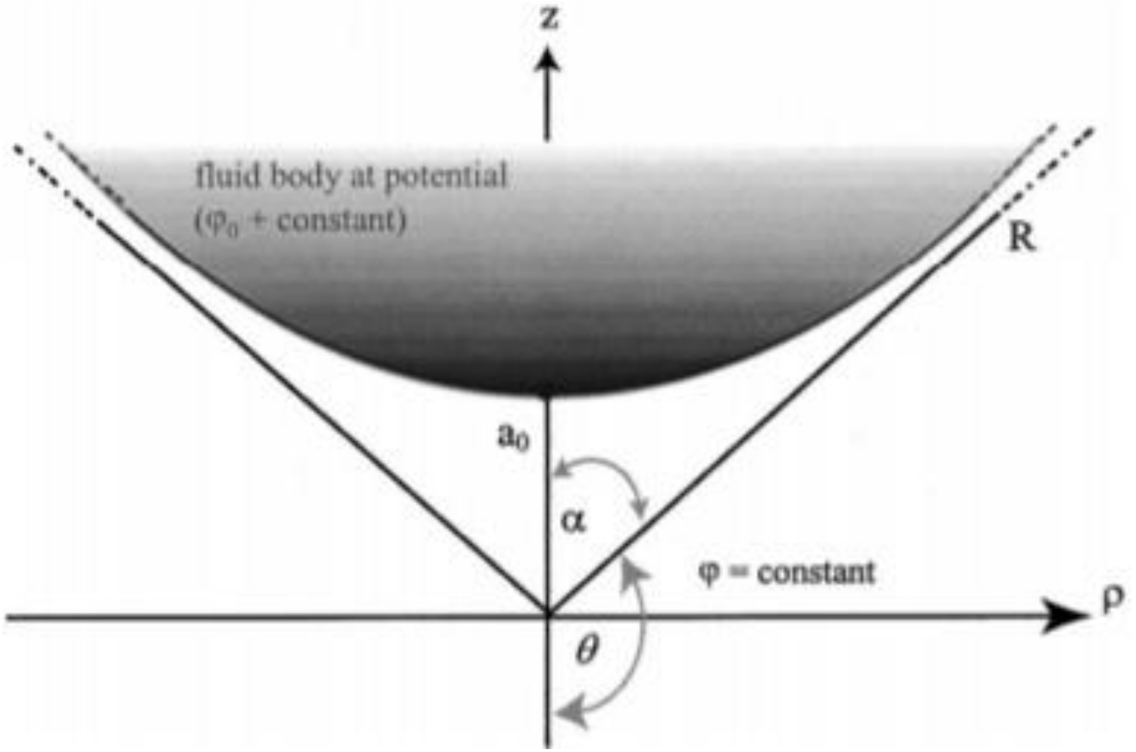
For inviscid fluids, Taylor cone has a semivertical angle 49.3°

$$Bo_e = \frac{F_e}{F_\gamma} = \frac{\varepsilon_0 \Phi^2}{\gamma D_0}$$



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Yarin, A.L.;Koombhongse,S.;Reneker,D.H. J. Appl.Phys. 2001, 90, 4836–4846.
Taylor, G.I. Proc. R.Soc.Lond.AMath.Phys.Sci. 1964, 280 (1382), 383–397.

MAJOR CHALLENGE FOR ELECTROSPINNING

Electrospinning method requires the use of complicated electrodes and high-voltage circuits

The fiber diameter depends on the nozzle diameter

Nandana et al. Electrospinning: A fascinating fiber fabrication technique, Biotechnology Advances, Volume 28, Issue 3, 2010.
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WORK PRINCIPLE OF P-JET SYSTEM

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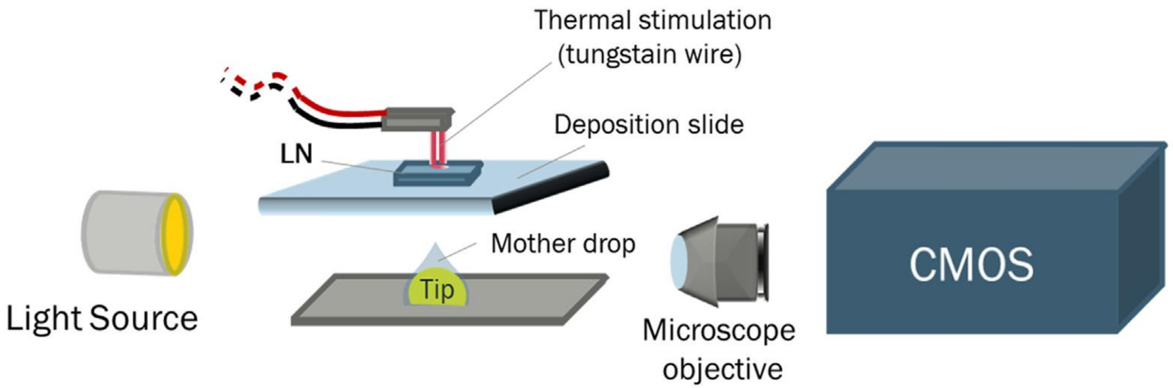
Change ΔT causes a variation ΔP_s

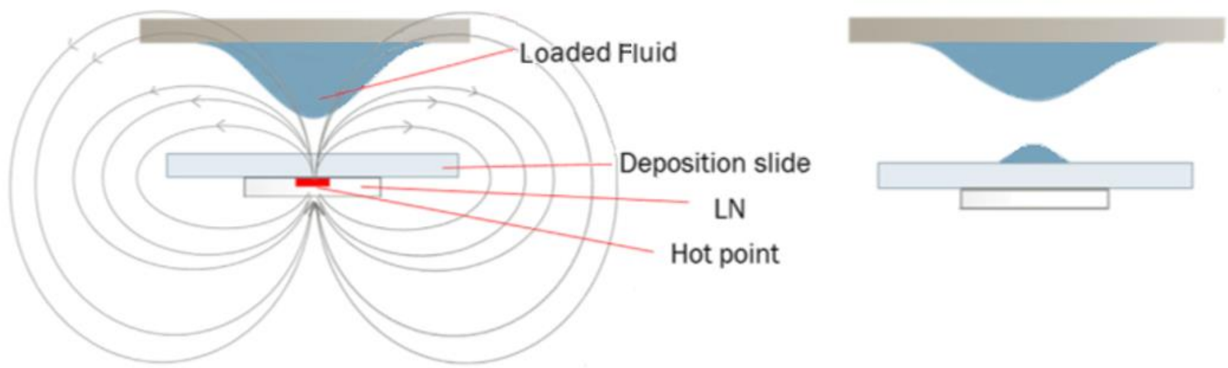
Creation of an electric potential across the crystal surfaces

Surface charge density immediately appears ($\sigma = P_c \Delta T$)

Deformation of the mother drop into a so-called Taylor cone

Tiny droplets are ejected from the apex of the meniscus





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Marianna Pannico, et al., Applied Surface Science, Volume 531, 2020
"Simple and Rapid Bioink Jet Printing for Multiscale Cell Adhesion Islands." Macromolecular Bioscience. , 2017, Vol.17(3)
Ferraro et al. Dispensing nano-pico droplets and liquid patterning by pyroelectrodynamic shooting. Nature Nanotech 5, 429–435 (2010)

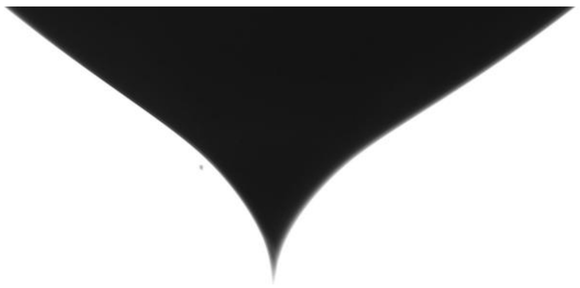
AIM OF WORK

Engineering the P-jet setup in order to use it as a reliable micro-rheometer

FLUID VISCOSITY ⇄ **P-JET EXPERIMENTS**

TAYLOR CONE FORMATION

- Cone angle
- First jet formation time
- Number of jet events



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AIM OF WORK

Engineering the P-jet setup in order to use it as a reliable micro-rheometer

FLUID VISCOSITY ⇄ **P-JET EXPERIMENTS**

- To perform real-time measurements
- To reduce the time analysis typical of a micro-rheometer

MATERIALS AND METHODS

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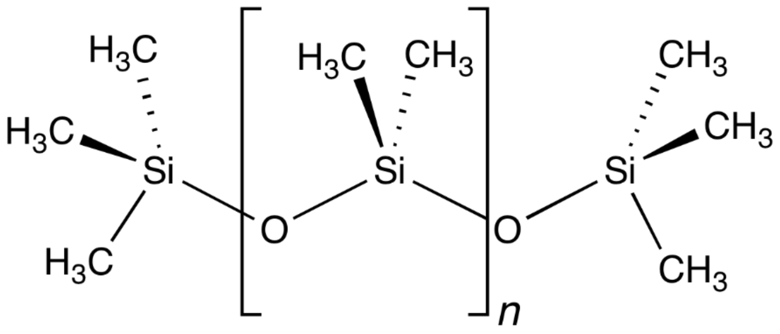
MATERIALS

PDMS



Kinematic viscosity [cSt]
10.000
30.000
60.000
100.000

ORIFICES

PMMA

ARPADIS

PLA

PRUSA i3 MK3S

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SCHEMATICAL VIEW OF THE SETUP

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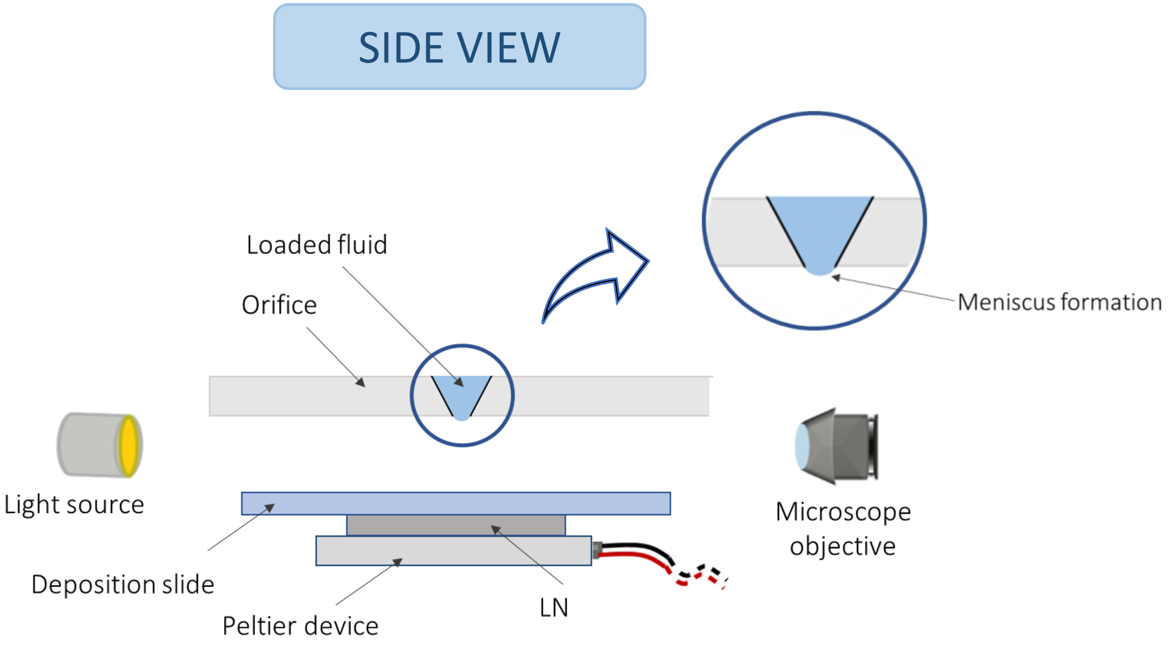

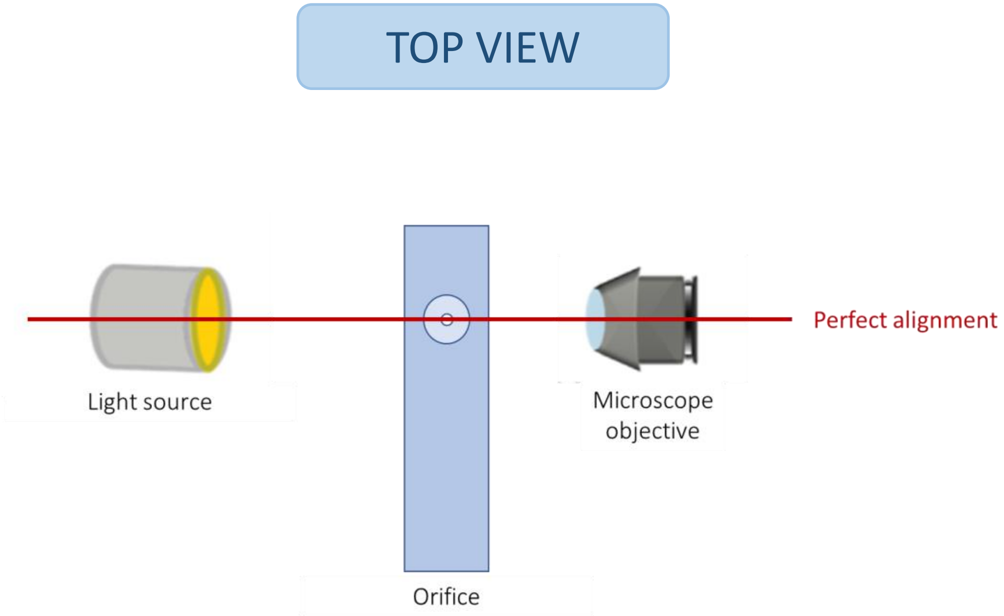

METHODS - EXPERIMENTAL PROCESSING

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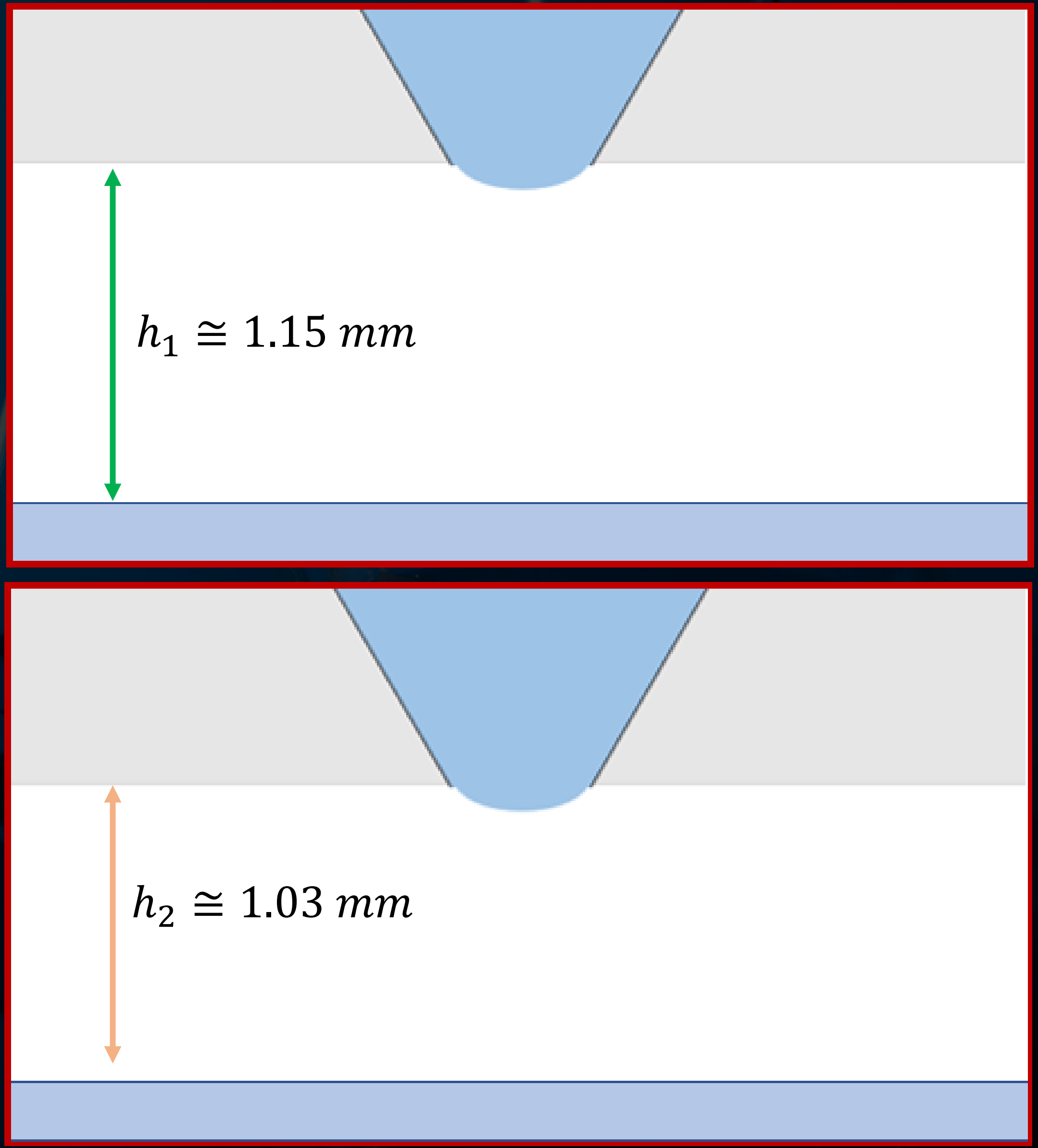
Cleaning orifice with ultrasound (ethanol + water)

Assembly and positioning (two fixed distances: h_1 and h_2)



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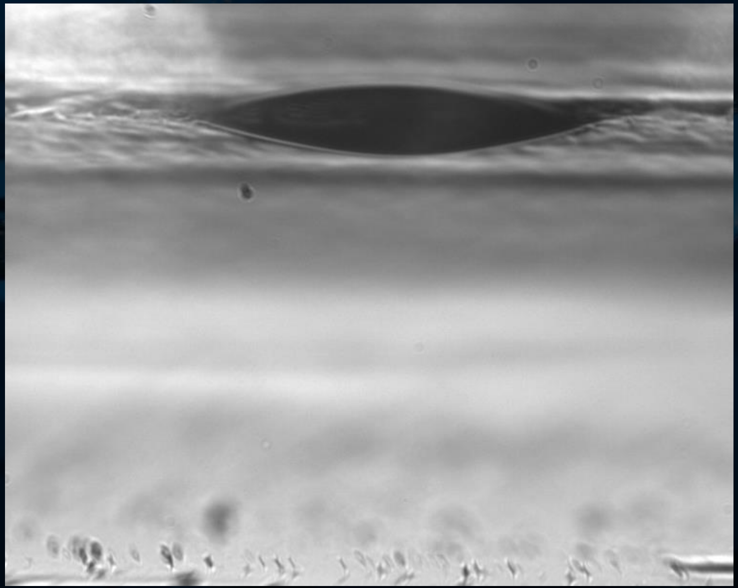
EXPERIMENTAL PROCESSING

Cleaning orifice with ultrasound
(ethanol + water)

Assembly and positioning
(two fixed distances: h_1 and h_2)

Snapshots of meniscus acquired one just after the loading and the other after 1 minute from loading



EXPERIMENTAL PROCESSING

SensApp

Cleaning orifice with ultrasound
(ethanol + water)

Assembly and positioning
(two fixed distances: h_1 and h_2)

Snapshots of meniscus acquired one
just after the loading and the other after 1 minute from loading

Start video and current at the same time
(current value =1.2 A and voltage value =3.2 V)

Waiting 1400 frames acquired
($\sim$ 97 s, time to reach 87 °C of lithium niobate crystal)

Stop current flow

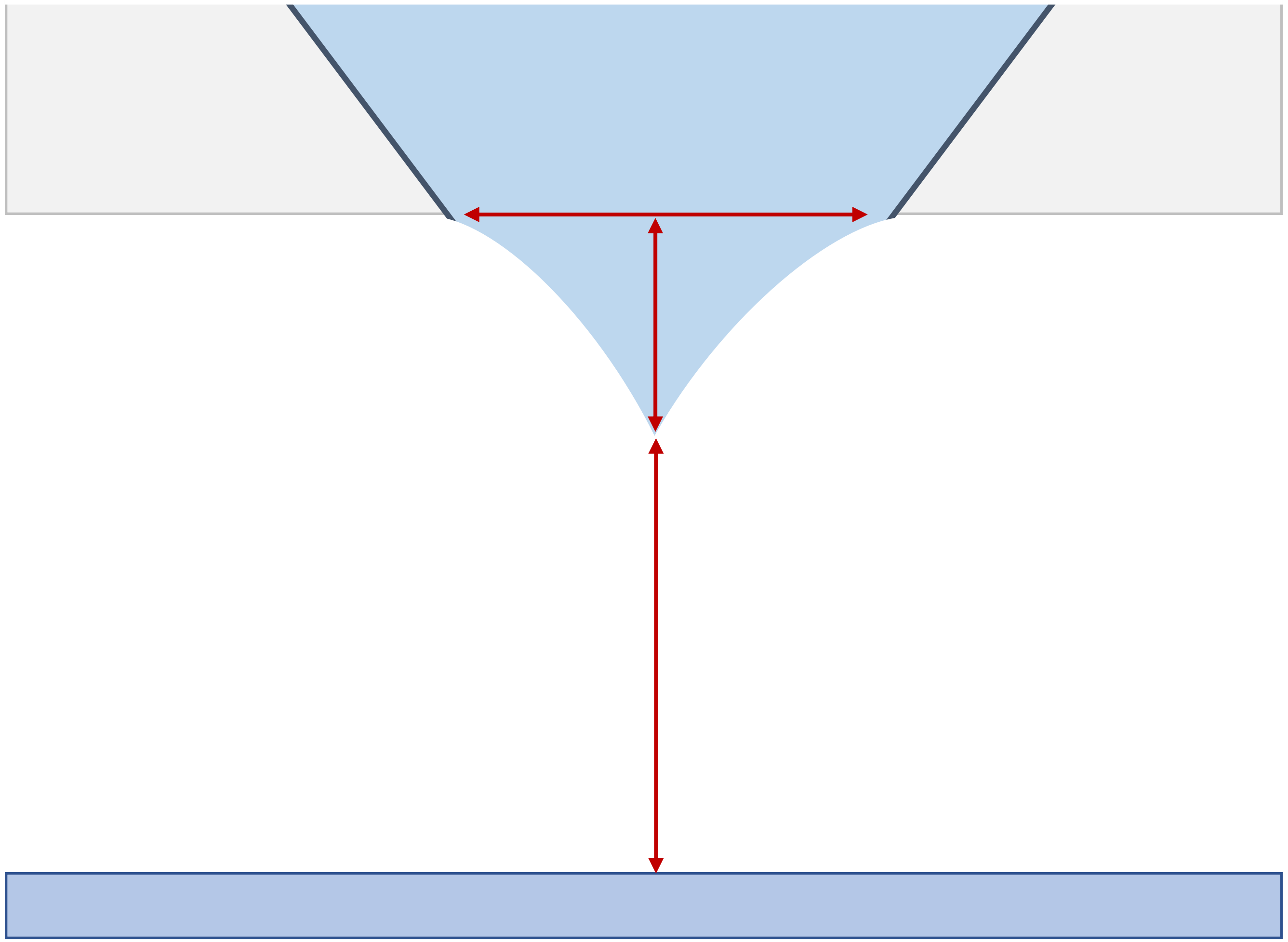
Observation until 2500 frames acquired and finally stop video

IMAGE ANALYSIS: First jet formation

CONE HEIGHT AND CONE DIAMETER

DISTANCE TIP-GLASS

$$544\ pixels = 0.908\ mm$$



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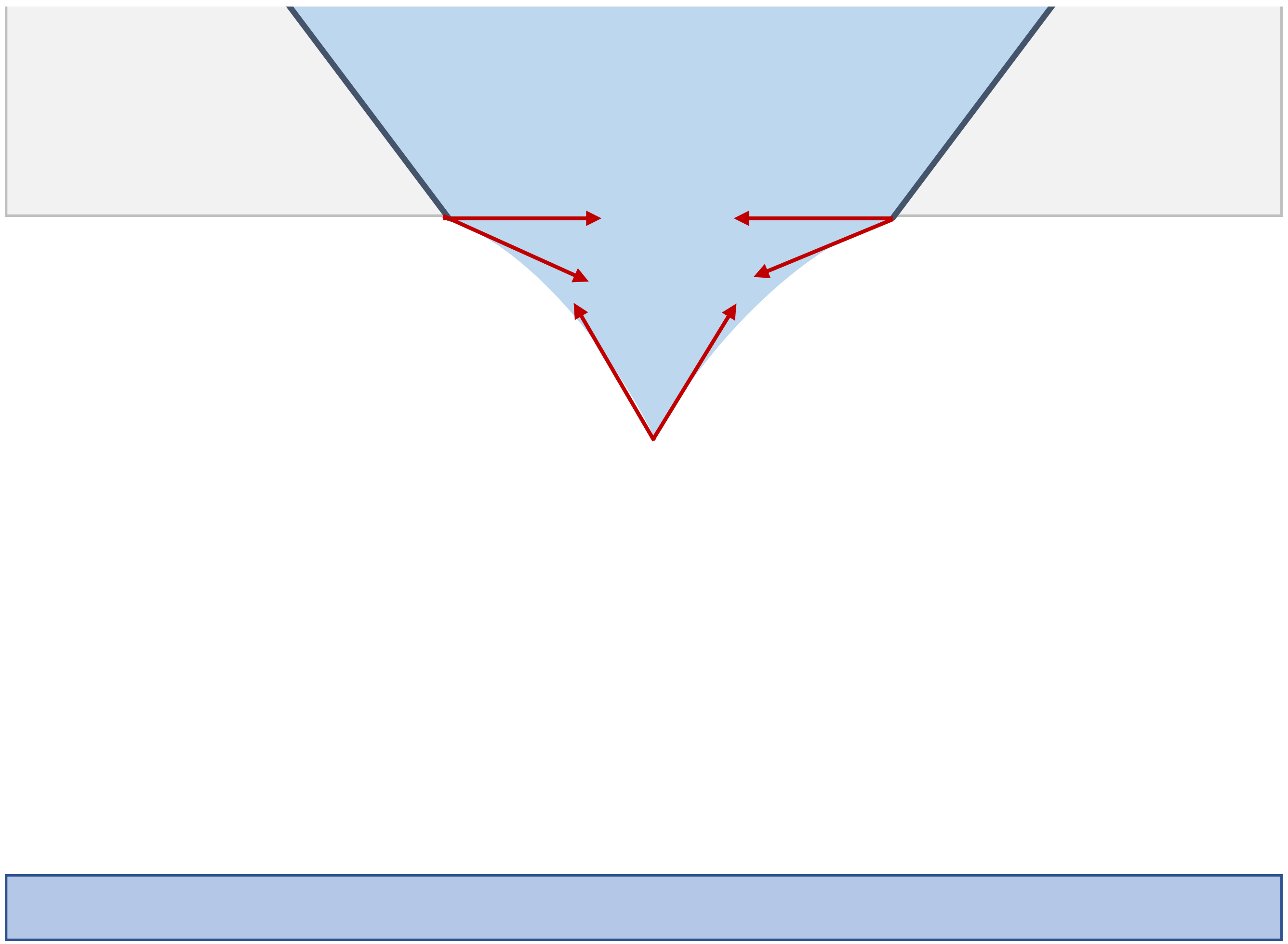
ImageJ

IMAGE ANALYSIS: First jet formation

SensApp

- CONE HEIGHT AND CONE DIAMETER
- DISTANCE TIP-GLASS
- CONE ANGLE
- CONTACT ANGLE (RH AND LH)
- FIRST JET FORMATION TIME



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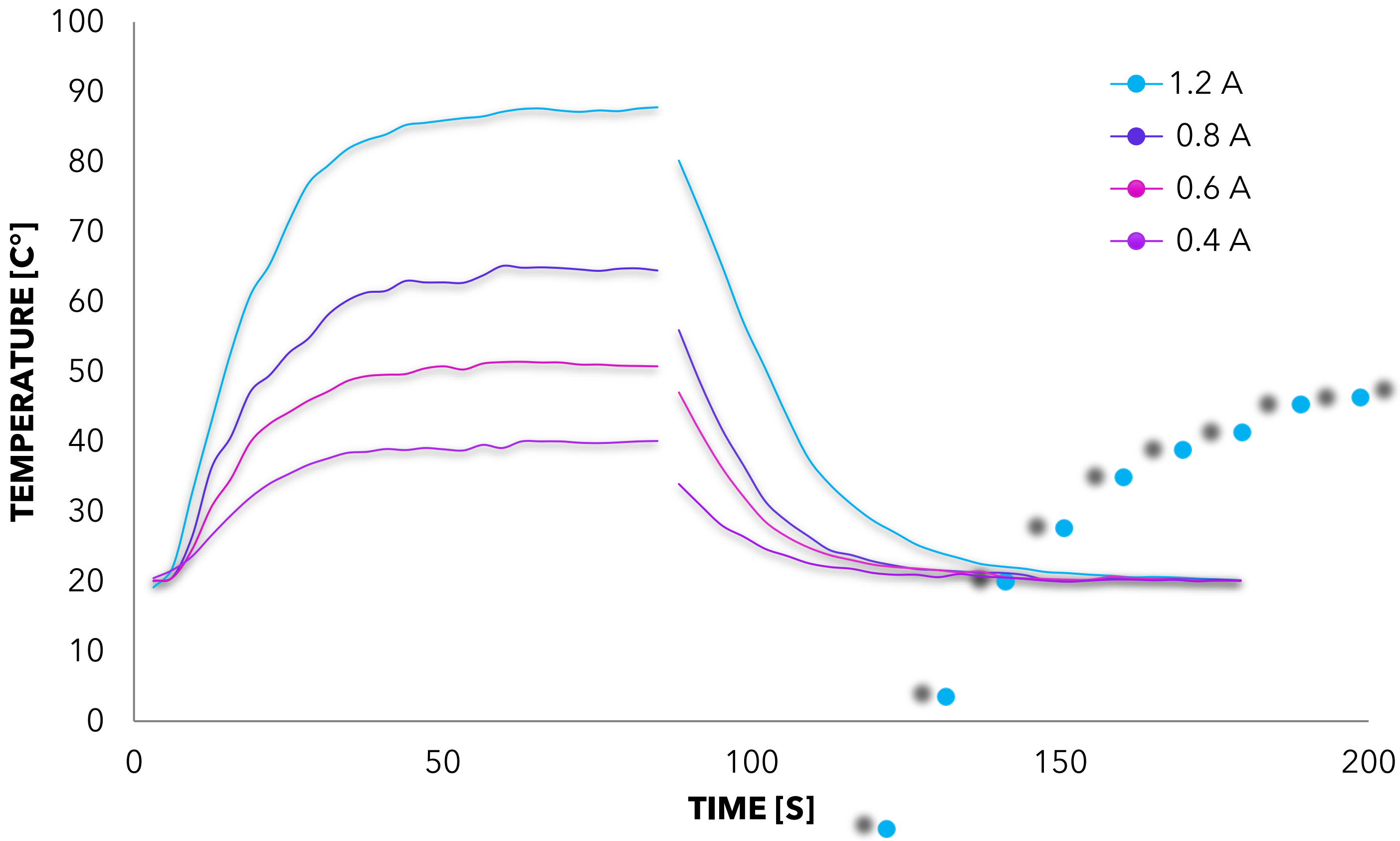
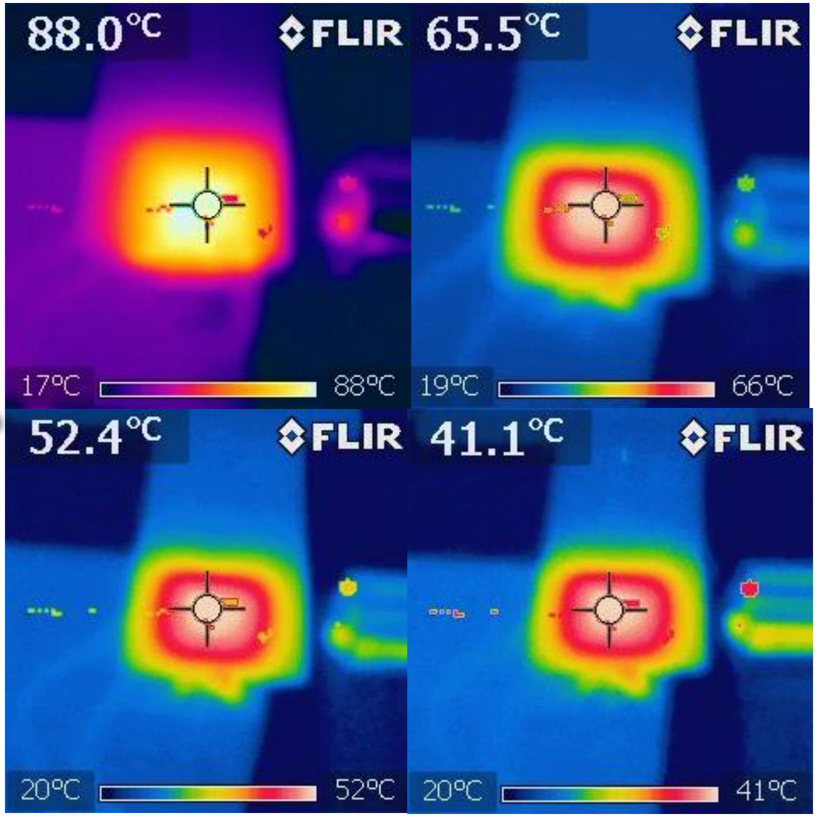
ImageJ

RESULTS AND DISCUSSION

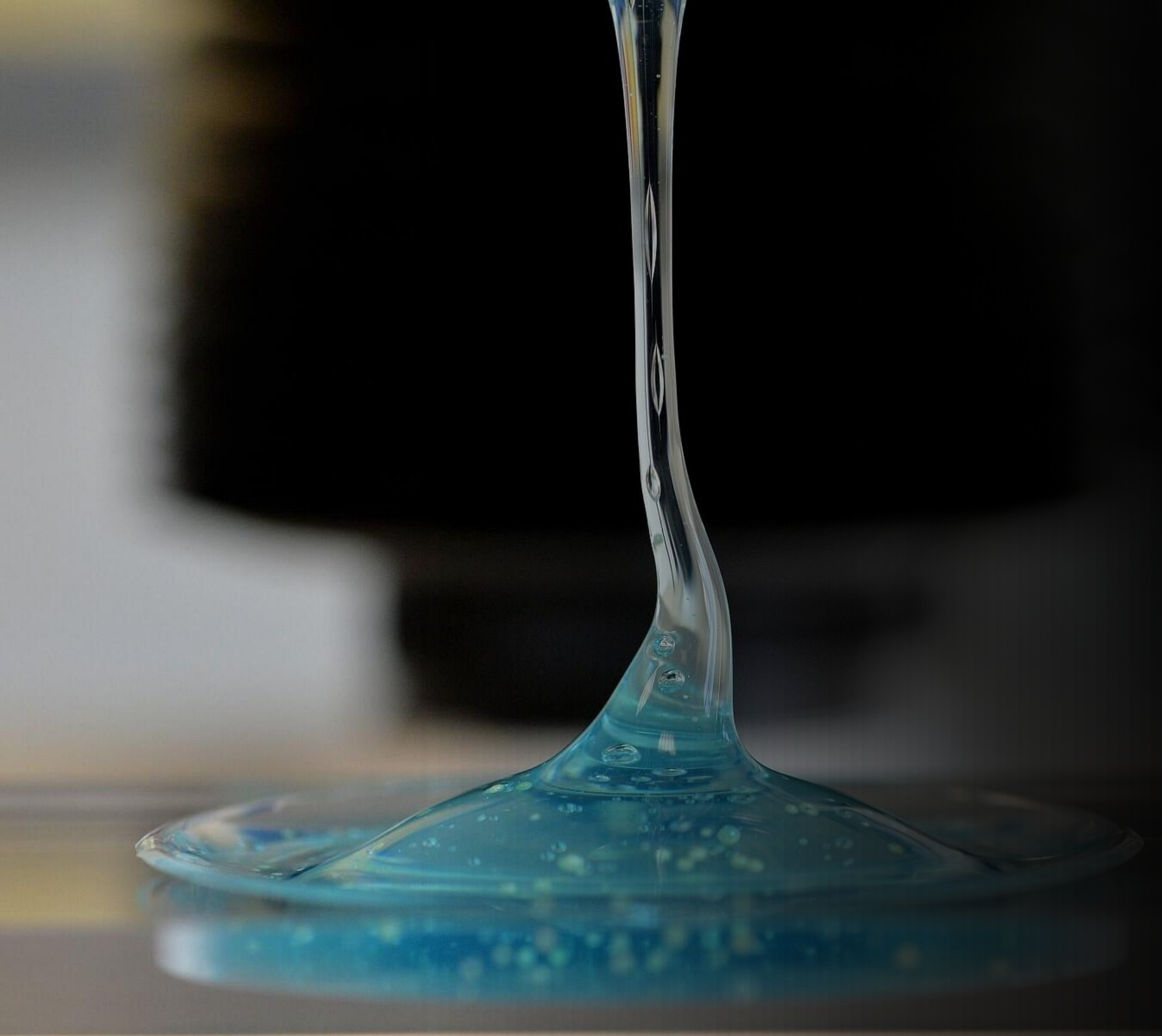
CRYSTAL TEMPERATURE VARIATION



1.2 A
0.8 A



0.6 A
0.4 A

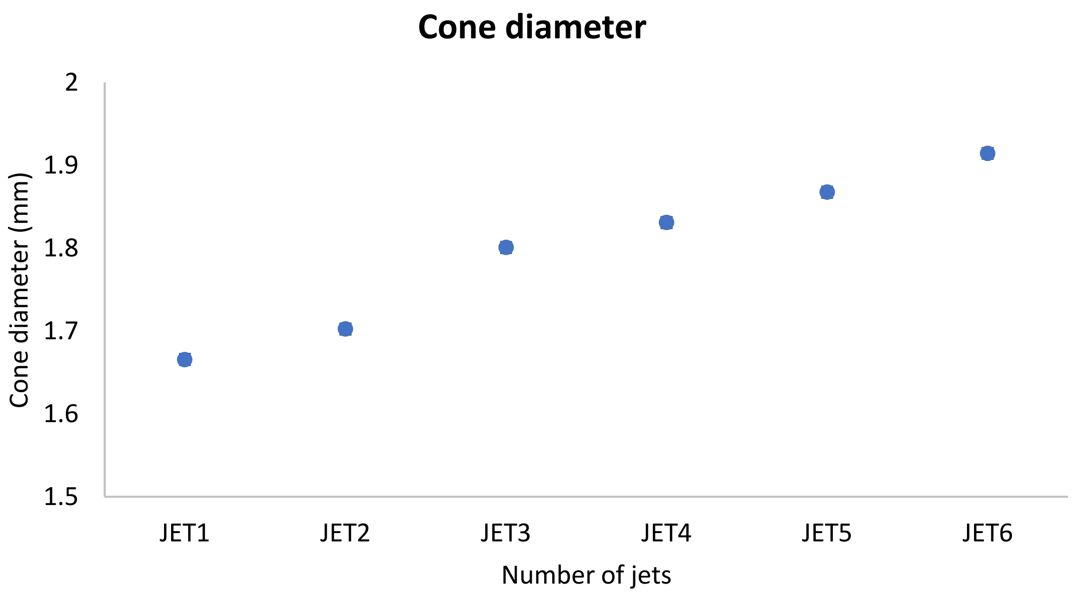


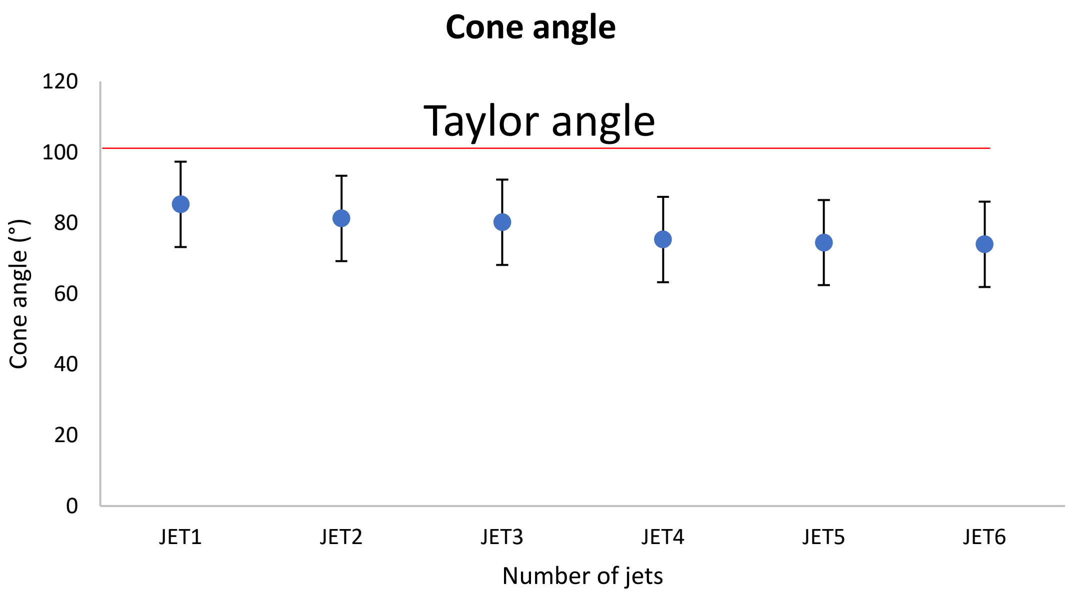
ONE MODEL SYSTEM – Jet frame analysis

- Single experiment variability
- Multiple experiments variability

Single experiment variability

Analysis of evolution of the parameters during the **<u>jet events of a single experiment</u>** of PDMS - 30.000 cSt:





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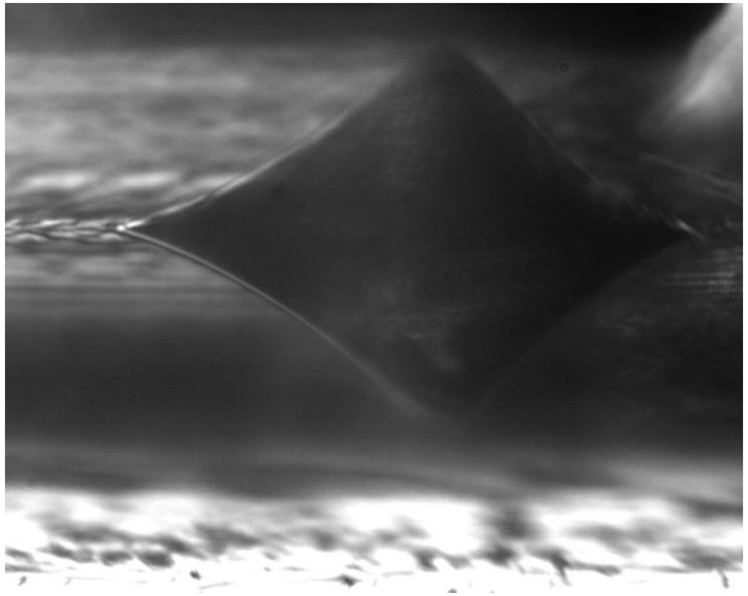
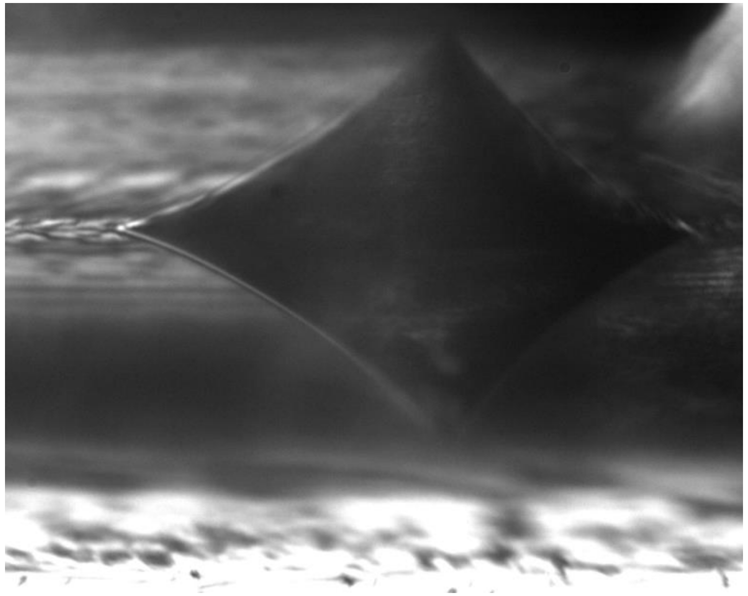
1 MODEL SYSTEM – Frame choice analysis

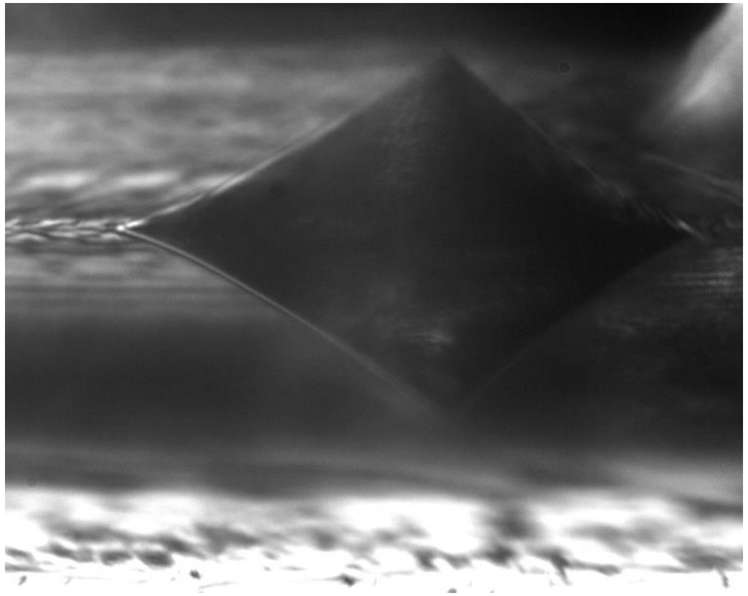
SensApp

The error bars were evaluated based on a frame choice analysis

Jet frame before ← 0.14 s → Jet frame ← 0.14 s → Jet frame after

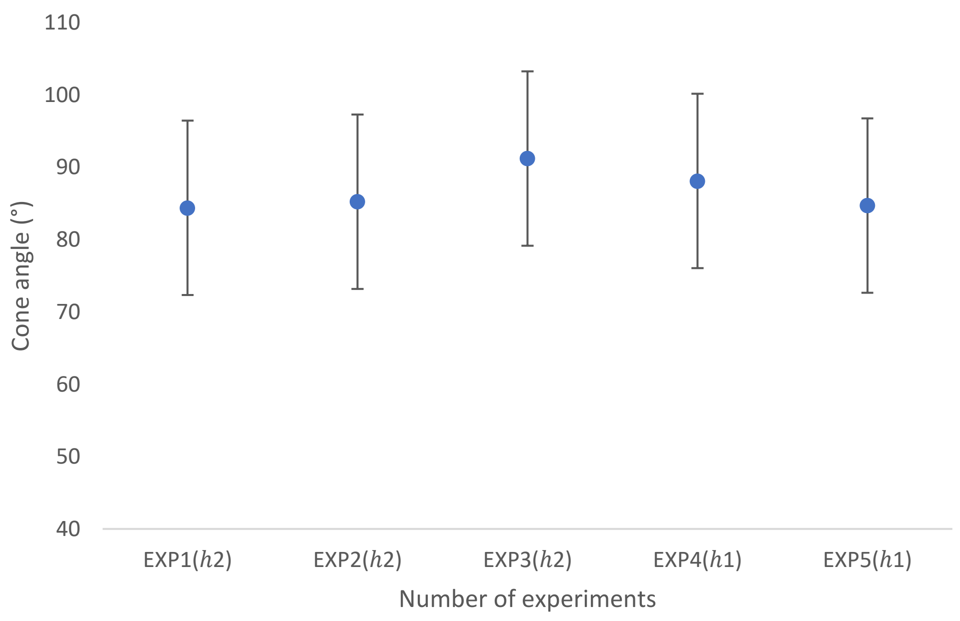


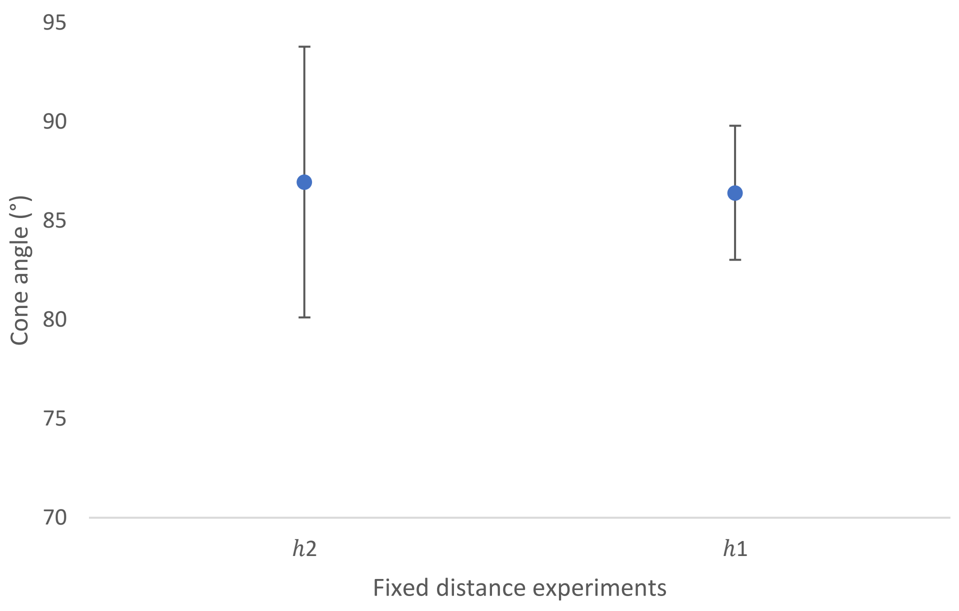




Multiple experiments variability – Cone angle

Analysis of evolution of the cone angle during the **first jet event of multiple experiments** of PDMS – 30.000cSt:





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3 MODEL SYSTEMS

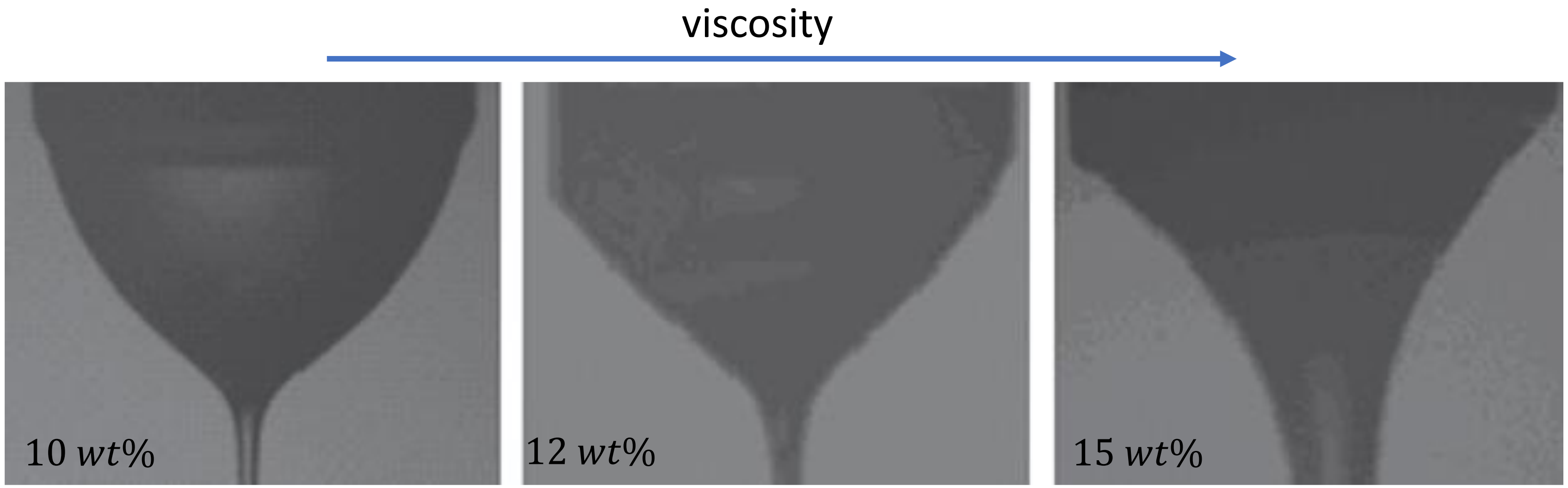
P-jet Measurements - Viscosity Relationships

- Cone angle evolution
- First jet formation time

LITERATURE

Yarin demonstrates, using PMMA (M_W= 540 kDa) solutions with different concentrations, demonstrated that the electrified meniscus can assume a wide variety of shapes that are different from that of the Taylor cone

Gañán-Calvo et al. Electrohydrodynamic Reynolds number: $\delta_\mu = [\frac{\gamma^2 \rho \varepsilon_0}{\mu^3 K}]^{1/3}$



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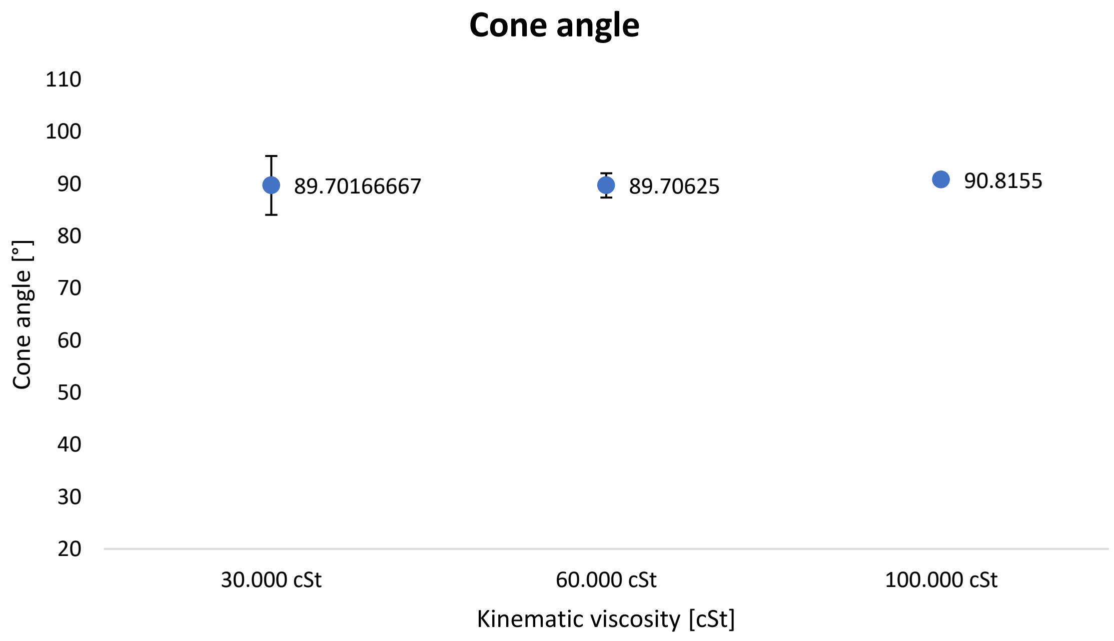
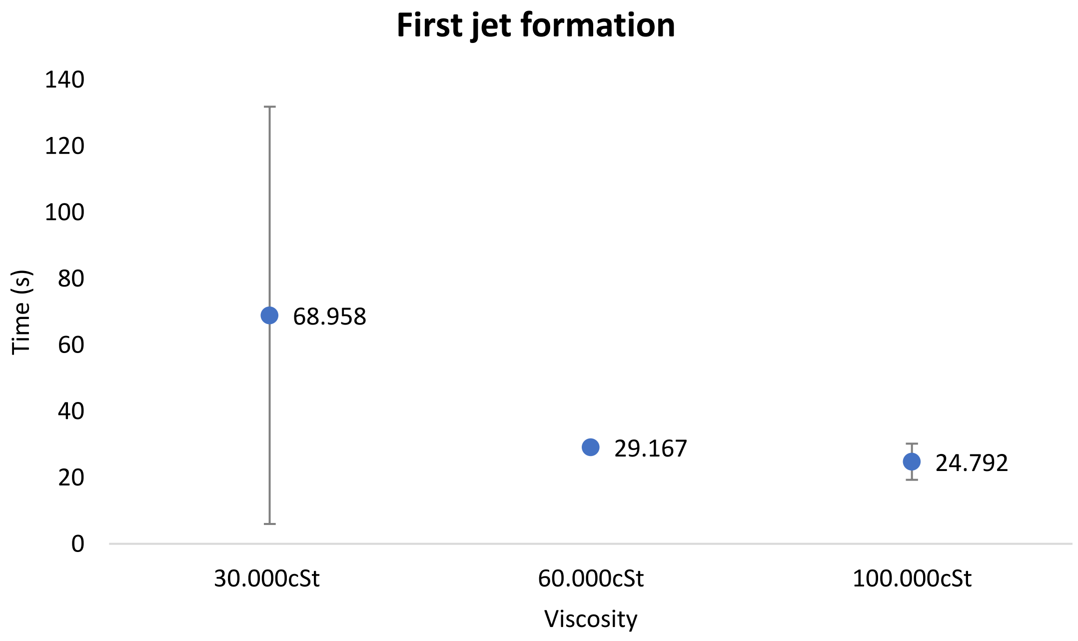
Yarin, A.L.;Koombhongse,S.;Reneker,D.H. J. Appl.Phys. 2001, 90, 4836–4846.
Taylor, G.I. Proc. R.Soc.Lond.AMath.Phys.Sci. 1964, 280 (1382), 383–397.

Cone angle evolution- First jet formation

Analysis of cone angle evolution during the **first jet event** of multiple experiments of **3 PDMS of different viscosity**:





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UNCERTANTIES ANALYSIS

Unknown geometry of the orifice

The current variation from 1.1 to 1.2 A could cause a change in crystal temperature

Positioning accuracy : $\pm$1 mm ruler full-scale

Positioning of deposition slide in contact with the crystal

Orifice wettability

Temperature of model fluids

Room temperature and humidity

ORIFICE WETTABILITY

Orifice wettability was considered the key problem related to:

The poor experimental repeatability

The independence of cone shape from the viscosity and therefore in contrast with what reported in the literature

The impossibility to use viscosities < 30.000 cSt

ORIFICE WETTABILITY – Surface metallization

Orifice surface metallization, as first solution, provided lower wettability but it was not sufficient to eliminate the problem entirely

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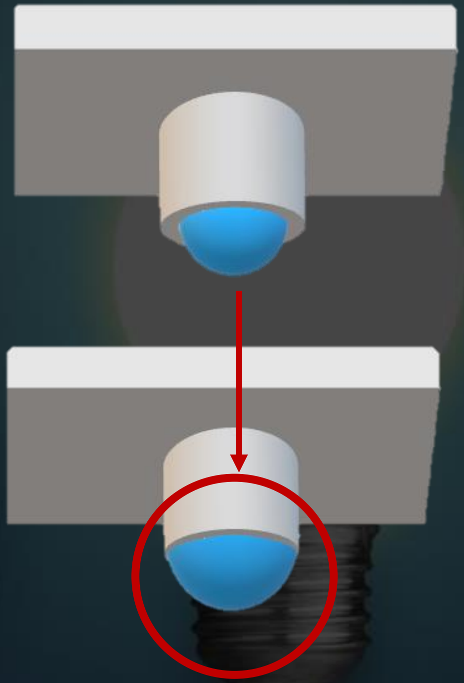
ORIFICE WETTABILITY – Design of new orifice

The cylindrical protuberance, acting as a needle, confines the diameter of the meniscus to this new geometry throughout the experiment



4 MODEL SYSTEM

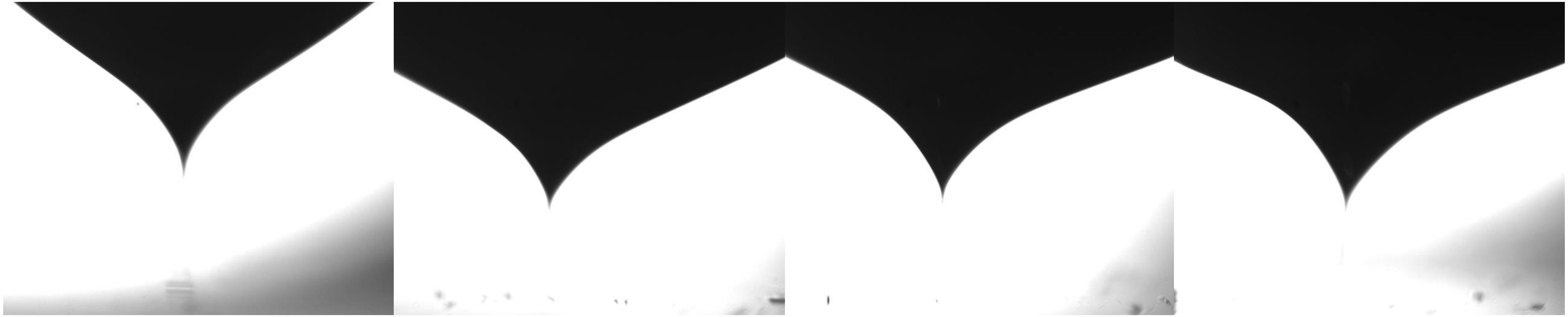
P-jet Measurements - Viscosity Relationships

- Cone angle evolution
- First jet formation time
- Number of jet events

Taylor-cone formation

SensApp

10.000 cSt	30.000 cSt	60.000 cSt	100.000 cSt



Viscosity

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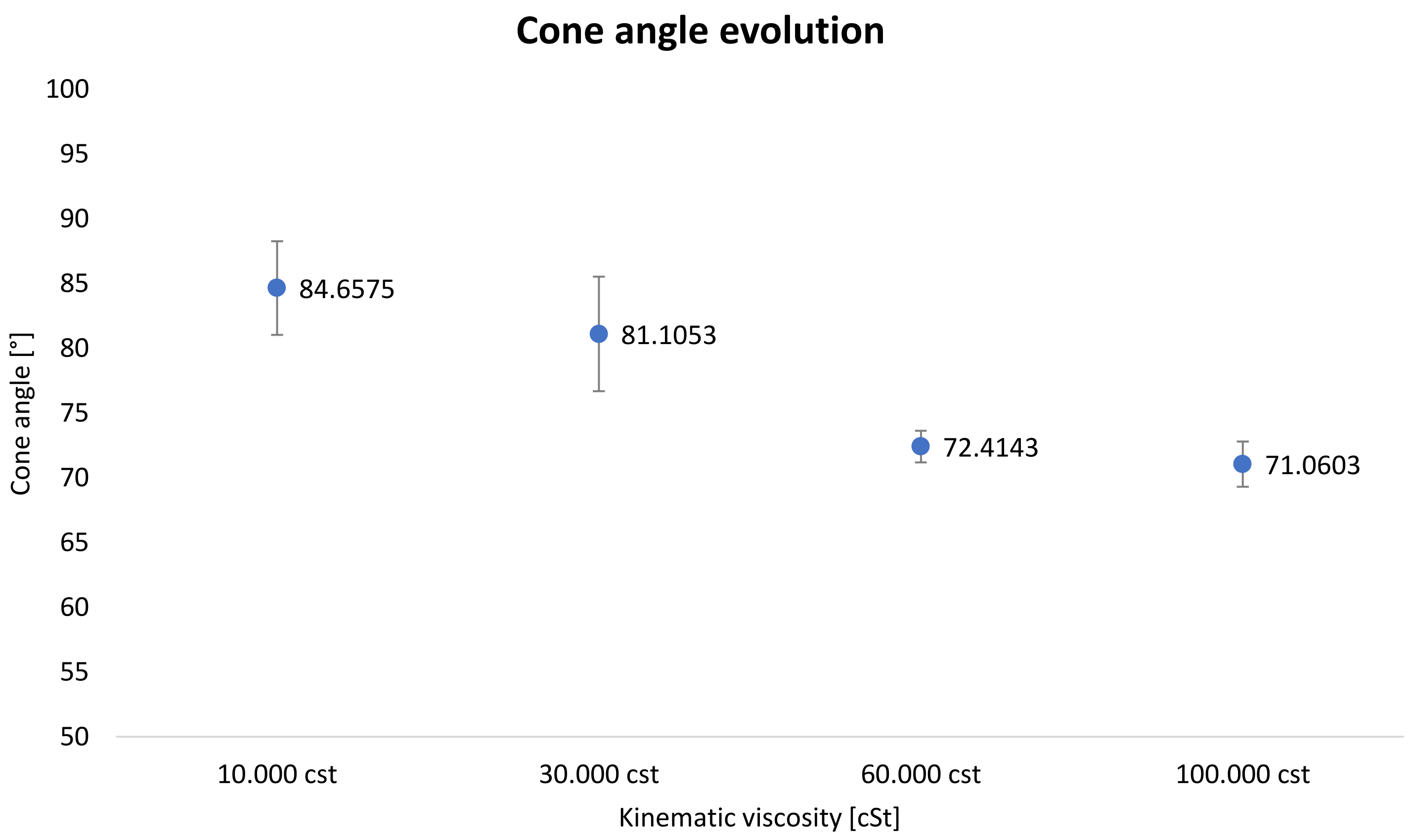
Cone angle shape evolution

Analysis of cone angle evolution during the **first jet event** of multiple experiments of **4 PDMS of different viscosity**:



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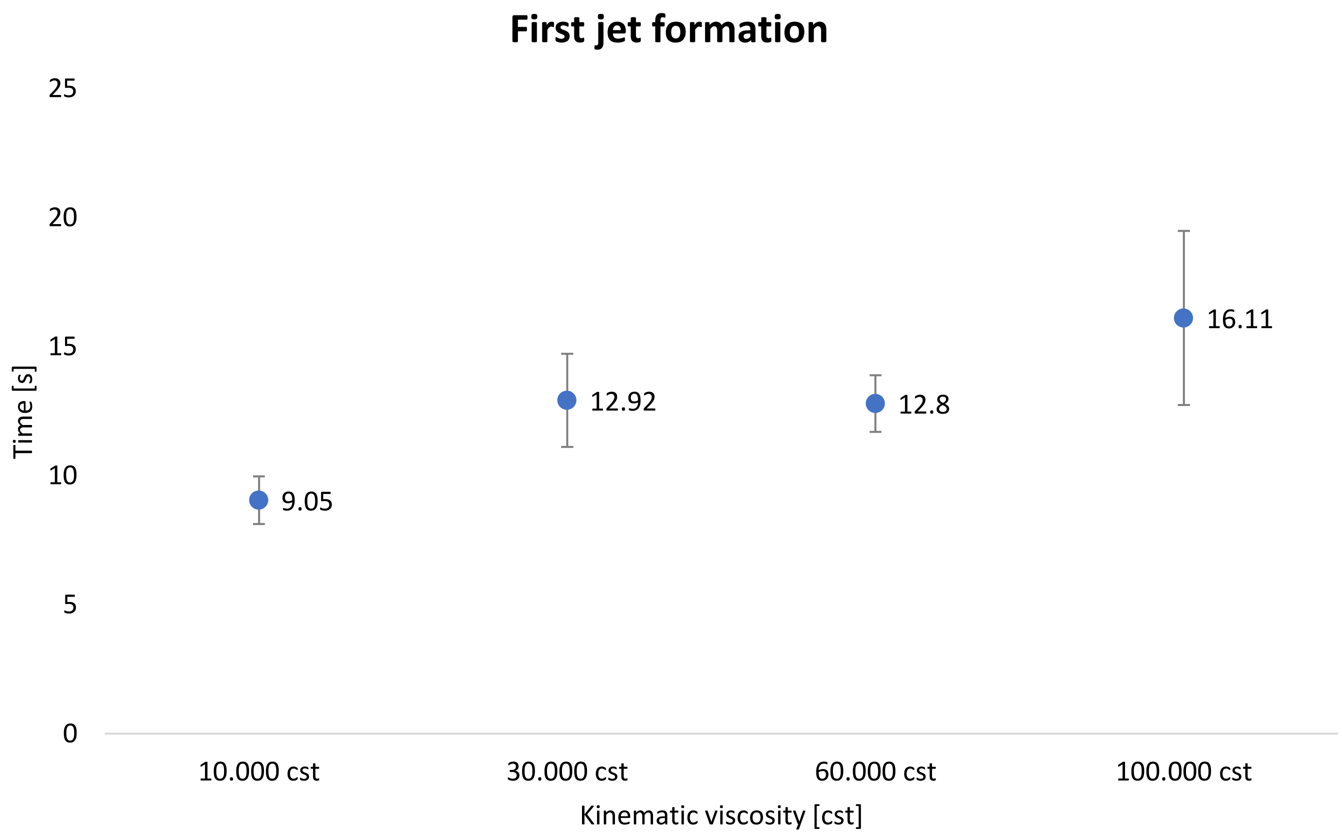
First jet formation time

Analysis of the **<u>time</u>** for the first jet event of multiple experiments of **<u>4 PDMS of different viscosity</u>**:



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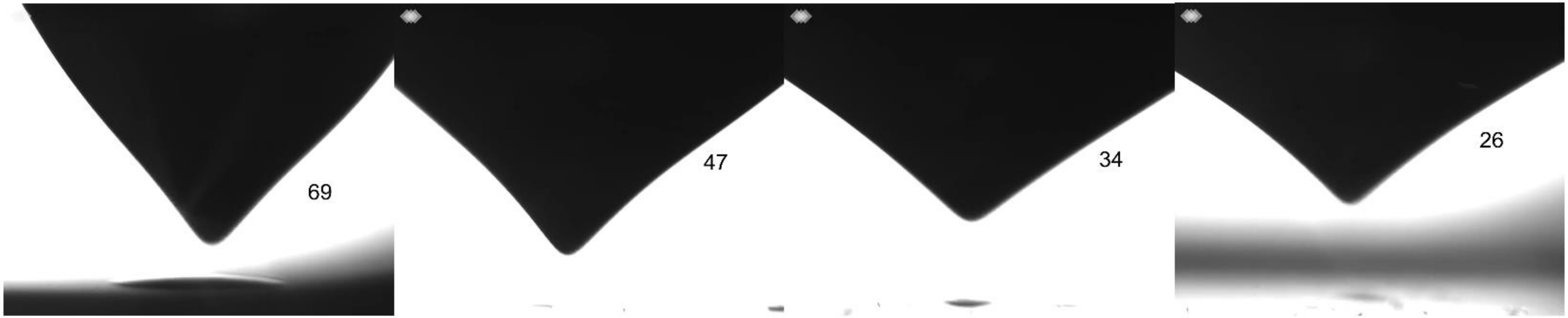
4 MODEL SYSTEMS – Jet frequency

SensApp

10.000 cSt | 30.000 cSt | 60.000 cSt | 100.000 cSt



Viscosity

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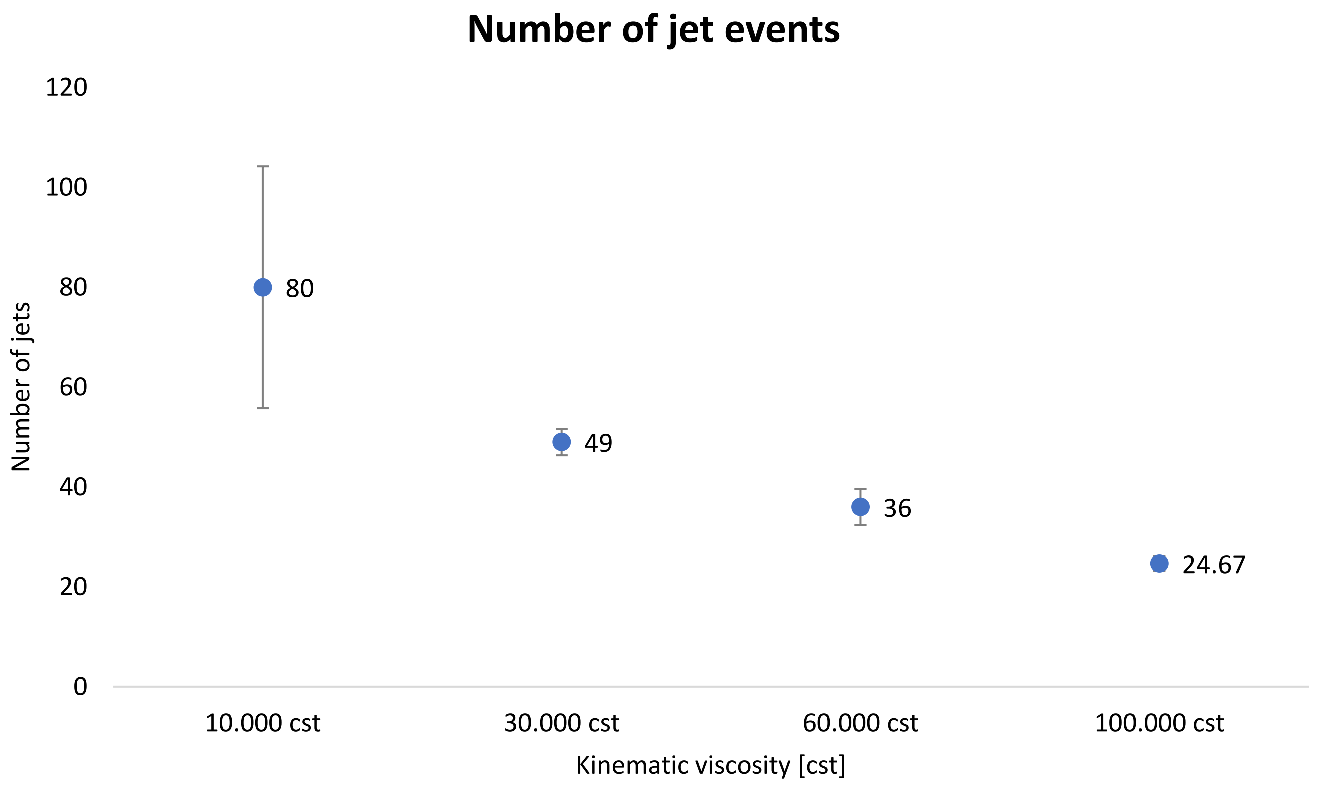
Number of jet events

Analysis of the **number** of the jet events for multiple experiments of **4 PDMS of different viscosity**:



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COMPARISION WITH SIMULATIONS

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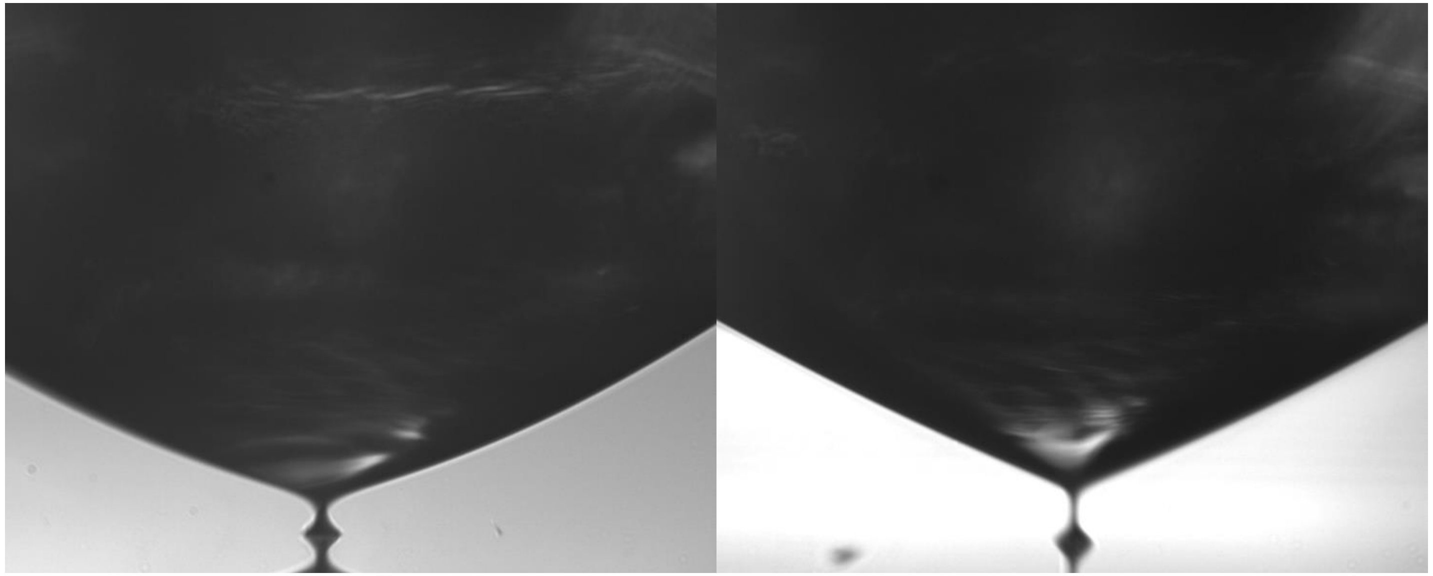
By varying the experimental protocol, we were able to deposit a droplet of macroscopic size

Mesh not extremely fine → Reduce simulation time



10.000 cSt

30.000 cSt

SensApp

POSSIBLE SOLUTIONS FOR IMPROVING ACCURACY

Unknown geometry of orifice	Disposable orifice with laser technology
The current variation from 1.1 to 1.2 A could cause a change in crystal temperature	A more accurate current generator
Positioning accuracy : $\pm$1 mm ruler full-scale	Implementation of step motor
Positioning of deposition slide in contact with the crystal	Implementation of step motor 2
Wettability problem	Design of a new orifice with a specific geometry
Temperature of model fluids	Infrared temperature sensor
Room temperature and humidity	Experiment in glovebox

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THANKS FOR YOUR ATTENTION!