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THE CENTRE IN ADVANCED FLUID ENGINEERING
FOR DIGITAL MANUFACTURING

Predicting the droplet size distribution of emulsions produced in a Sonolator

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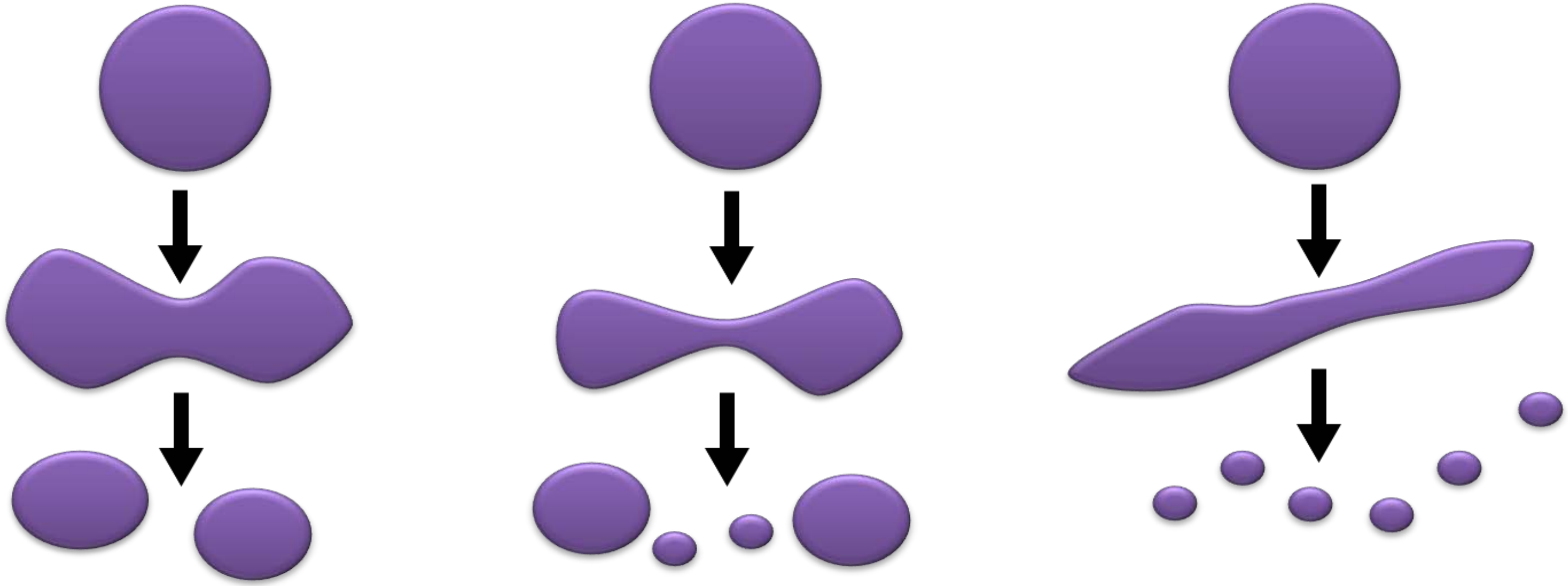
The Sonolator

- High pressure homogeniser used to create formulated products such as emulsions and suspensions etc.
- High speed jet impacts upon sharp blade, creating ultrasonic cavitation which contributes to droplet break-up.
- Very practical as it is a continuous device with few moving parts.
- Also multiple feeds can be used.

Droplet Break-up



$$d_{max} \propto {\mu_d}^{0}$$

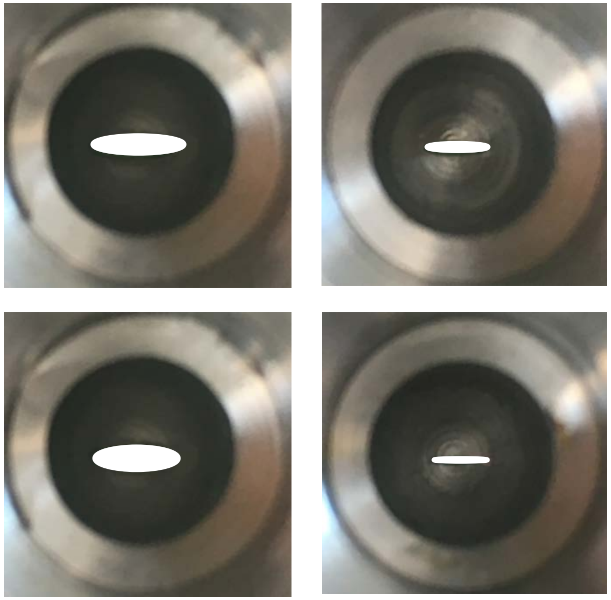
$$d_{max} \propto \epsilon^{-0.4}$$

Increasing μ_d

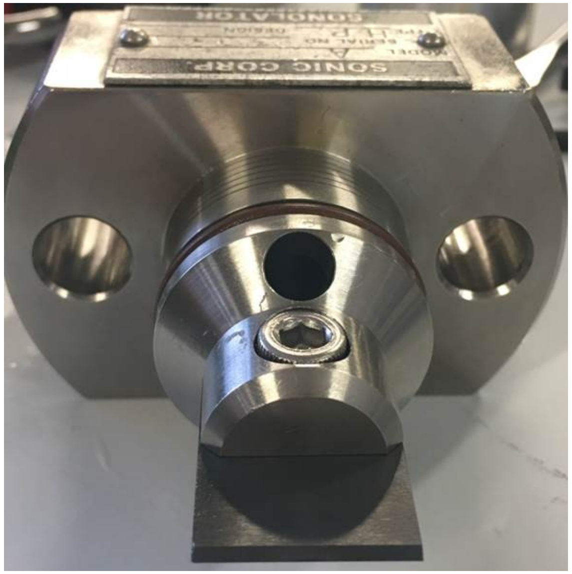
$$d_{max} \propto {\mu_d}^{0.75}$$

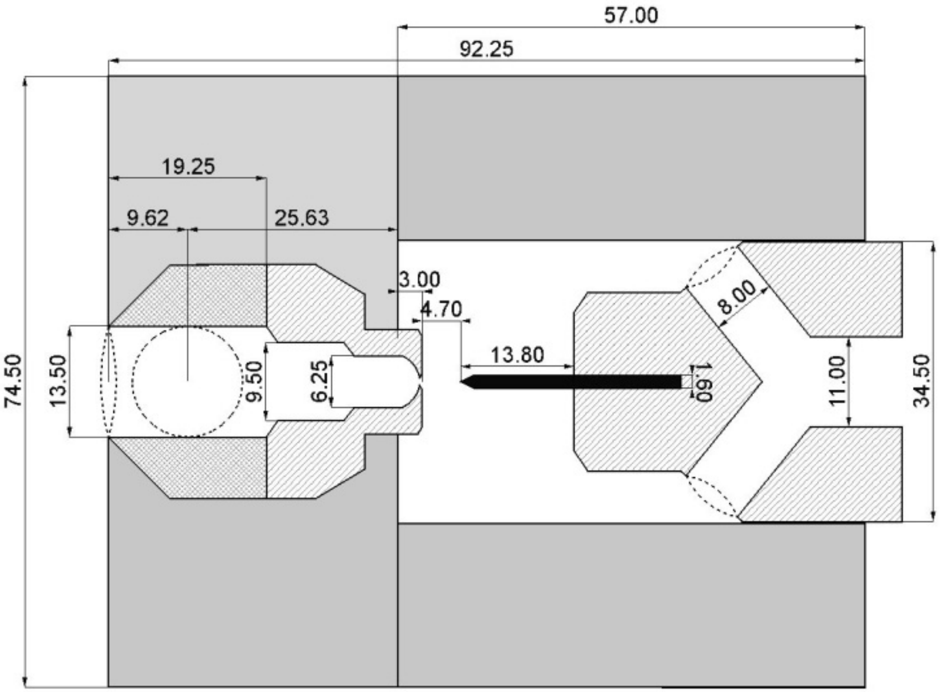
$$d_{max} \propto \epsilon^{-0.25}$$

Materials and Methods



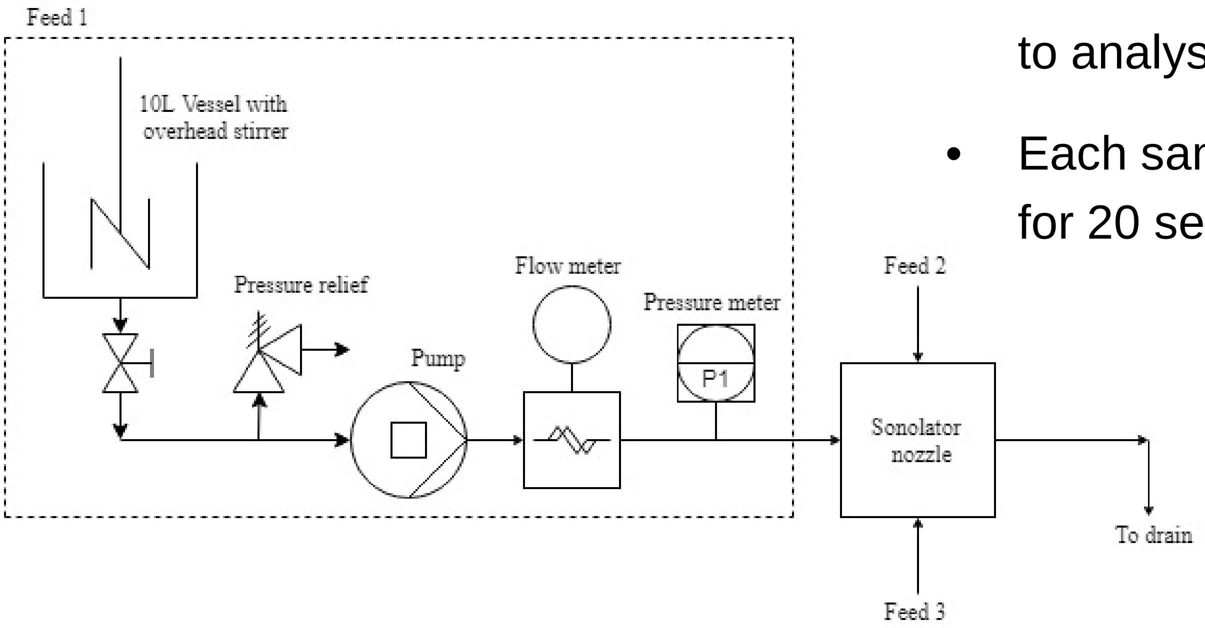
- Nozzle sizes for the Sonolator are arbitrary sizes (supposedly orifice area in square inches).

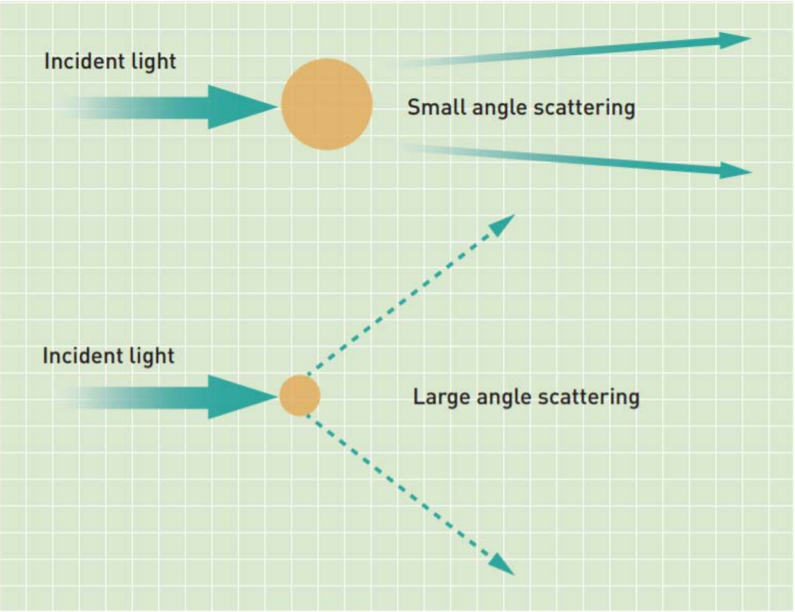




Materials and Methods

- Single feed configuration was used due to the low amounts of SiOil used.
- System was firstly calibrated for each Nozzle.





- Malvern Mastersizer 3000 was used to analyse the DSD of each sample.
- Each sample was measured 5 times for 20 seconds.

Materials and Methods

- A concentrated pre-emulsion was first made using an overhead stirrer. 1L of pre-emulsion was diluted by a factor of 10 for each run in the Sonolator.

Component	VF %
Water	98.9
SiOil	1.0
SLES	0.1

all noz

μ_d = 10, 50, 350, 1000, 2760 cSt

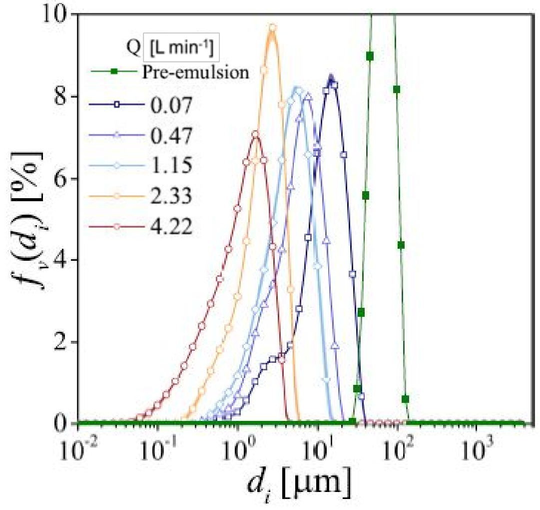
0.001 noz

Component	VF %
Water	88.9
SiOil	10.0
SLES	0.1

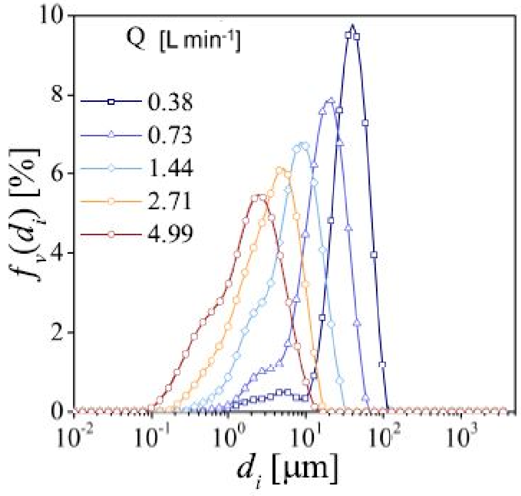
μ_d = 50 cSt

Droplet Size Distributions

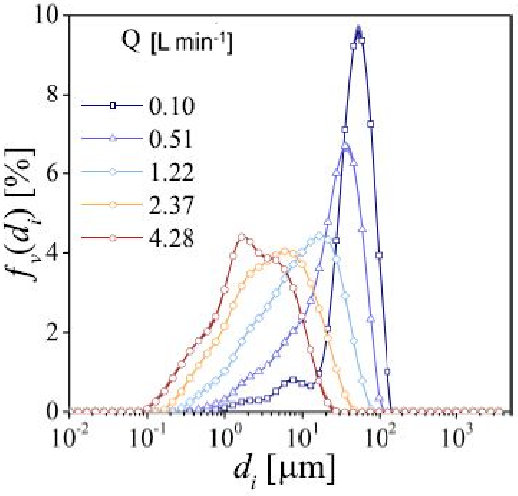
Noz = 0.001



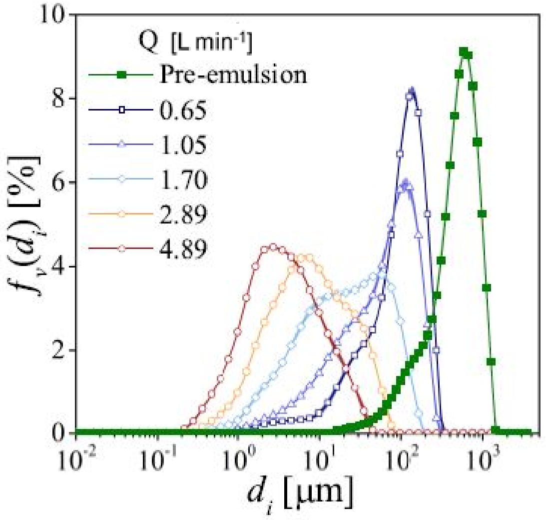
μ_d = 10 cSt



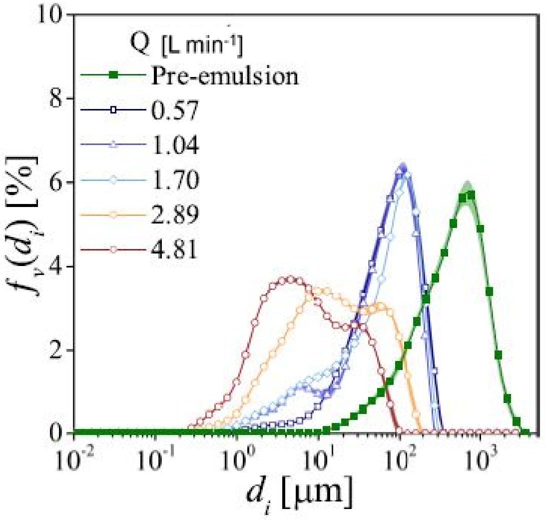
μ_d = 50 cSt



μ_d = 350 cSt



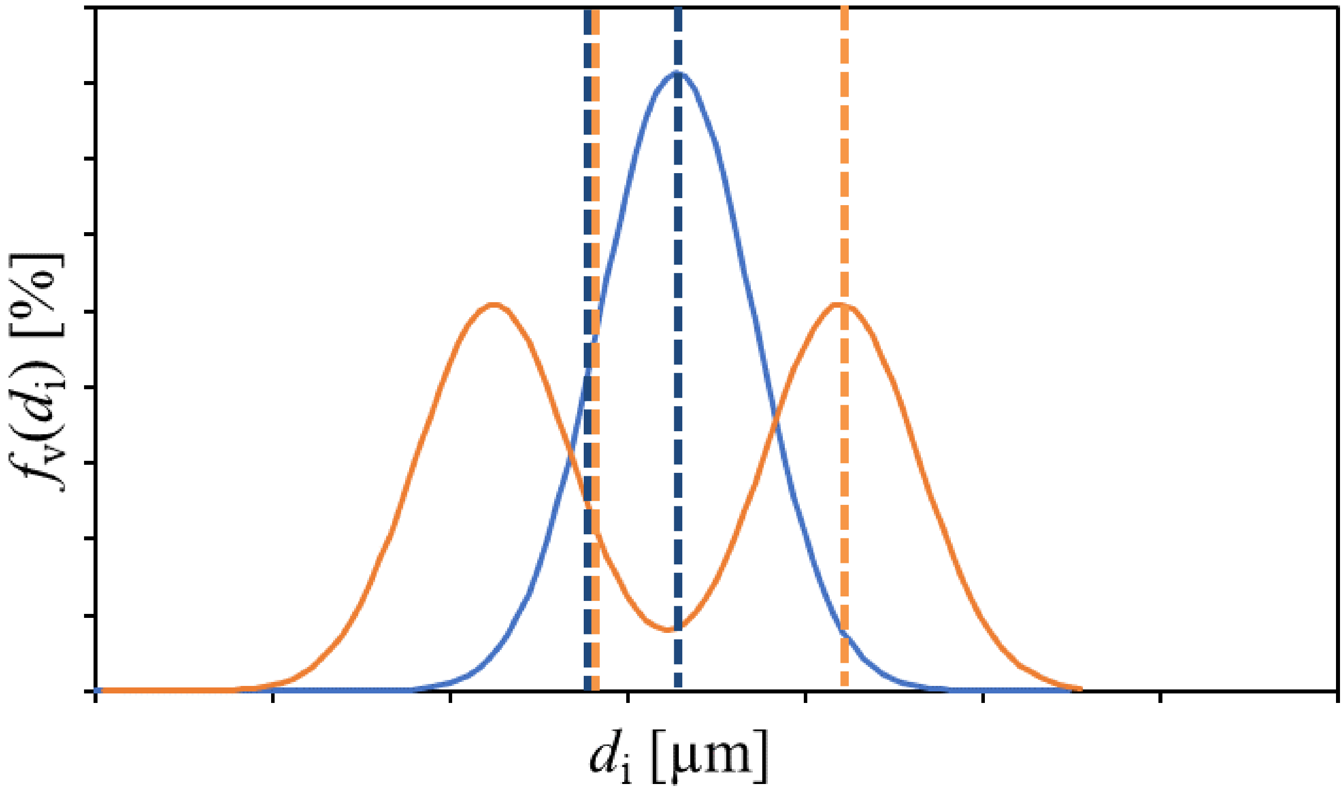
μ_d = 1000 cSt



μ_d = 2760 cSt

- Drop size decreases with flow/pressure and increases with viscosity.
- Most distributions are bi-modal – caused by formation of satellite or fragment droplets.

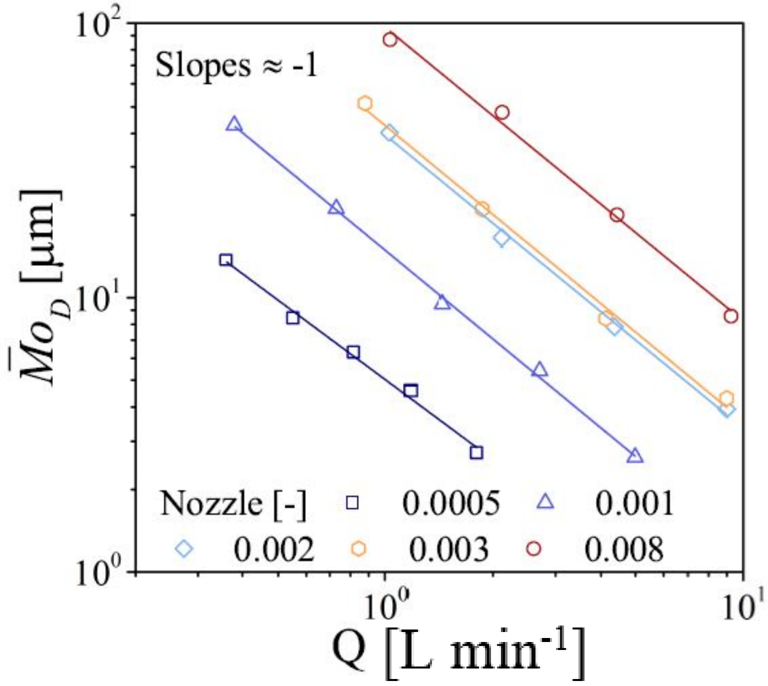
Why use mode?

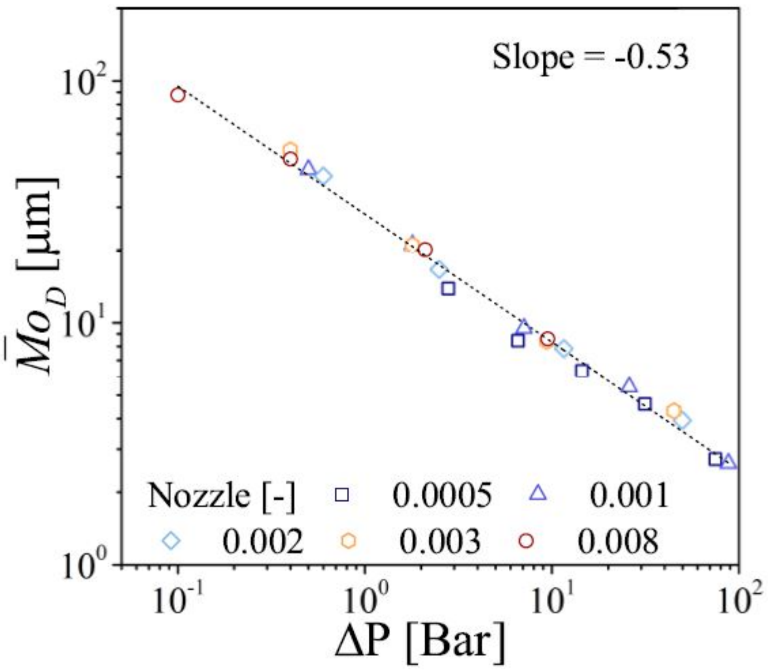


$$d_{3,2} = d_{3,2} \propto Mo_D \propto d_{max} \neq Mo_D$$

$\mu_d = 50$ cSt

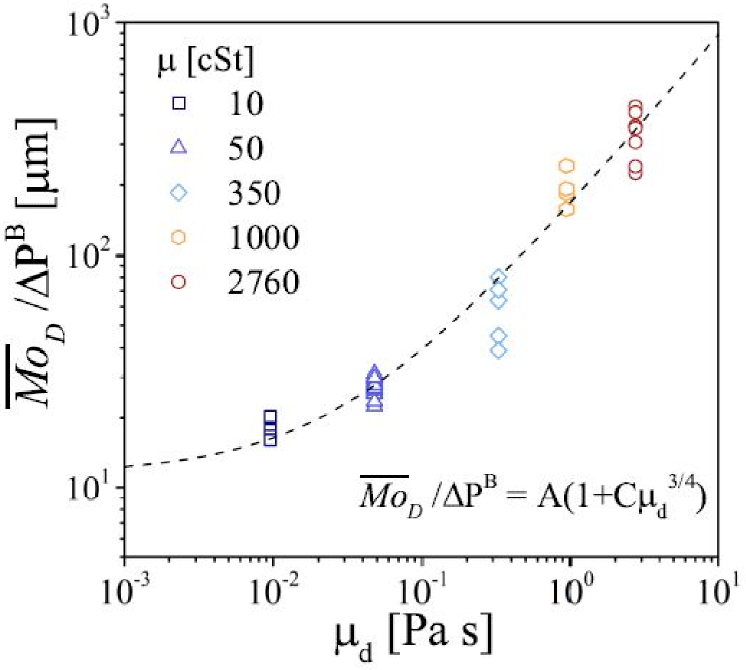
Effect of Q and ΔP on drop size





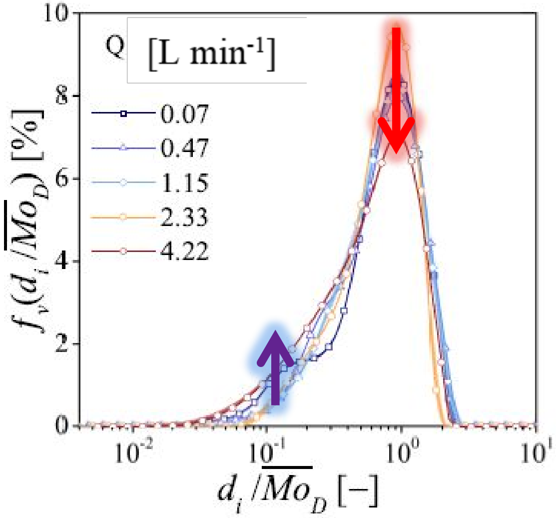
- Mode of daughter droplets is proportional to Q^x, where $-1.2 > x > -0.75$.
- Drop size scales with ΔP.

Effect of μ_d on drop size

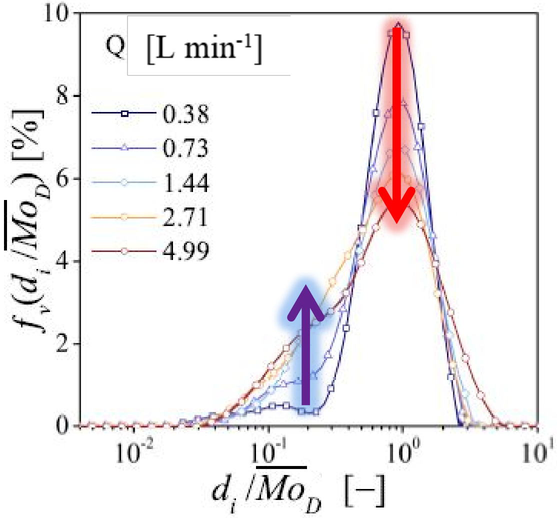


- Mode is independent of viscosity at low viscosities and is proportional to viscosity to the power of 0.75 at higher viscosities.
- Follows trend expected from theory.

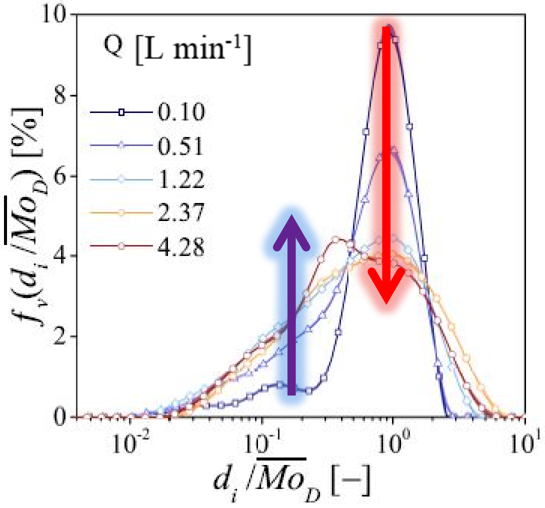
Effect of ΔP and μ_d on φ



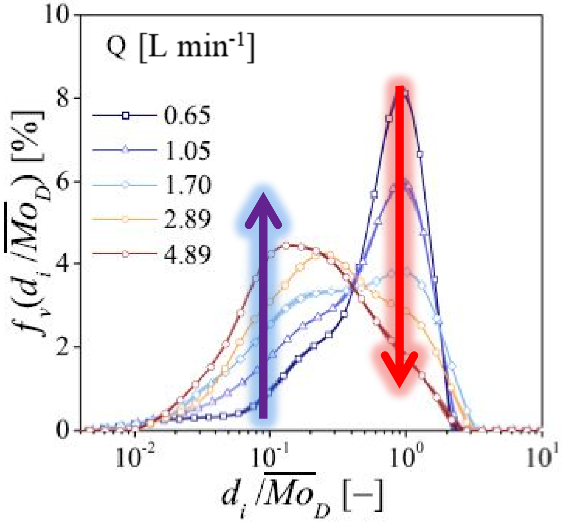
10 cSt



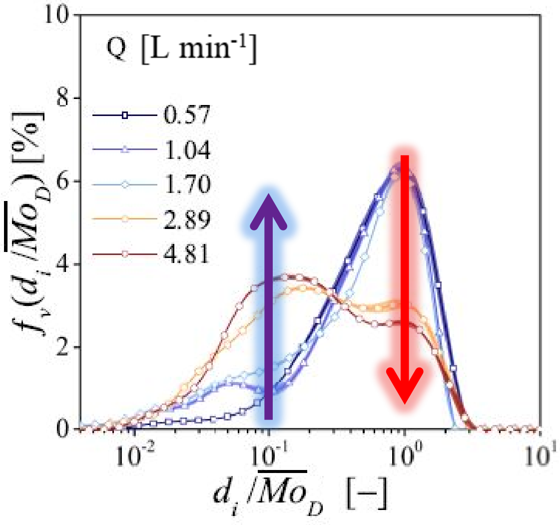
50 cSt



350 cSt

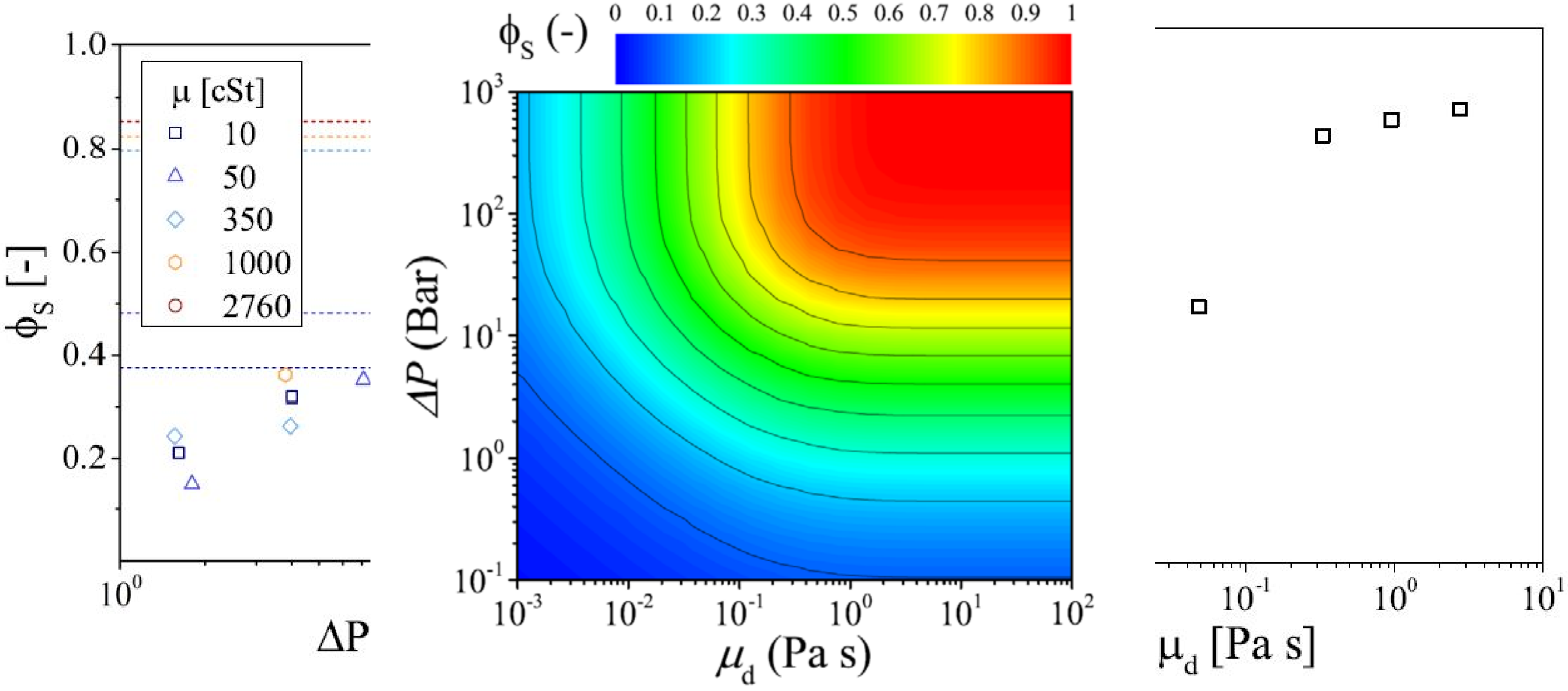


1000 cSt



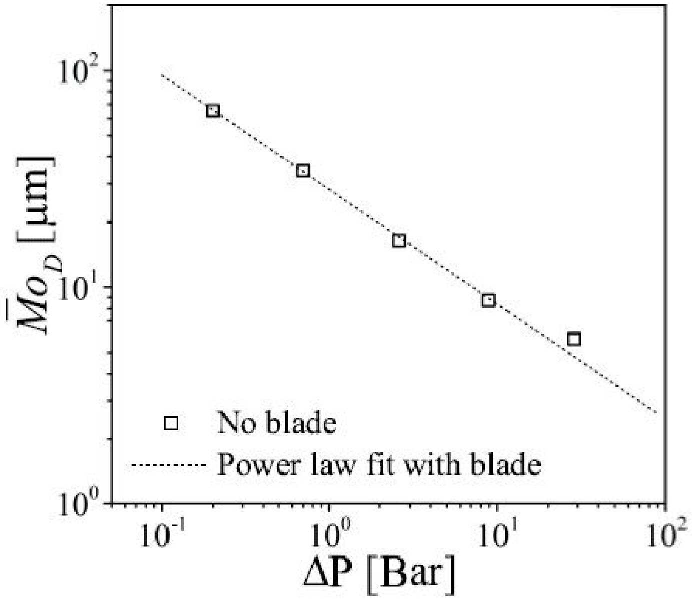
2760 cSt

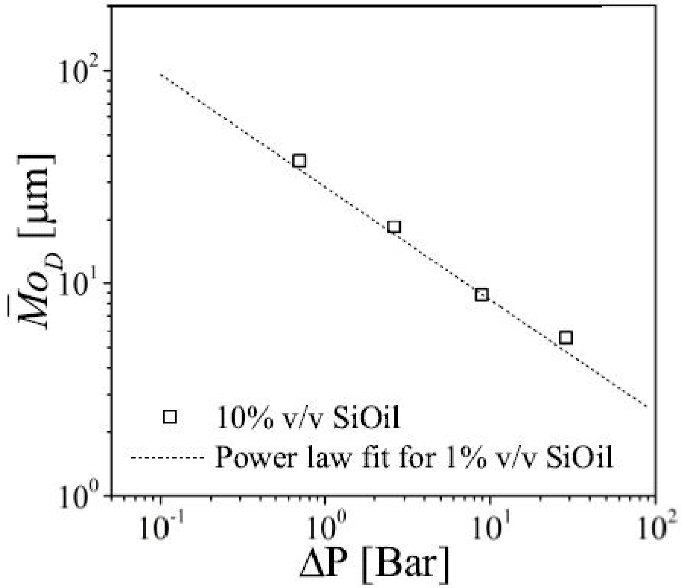
Effect of ΔP and μ_d on φ



- Volume fraction of satellite droplets follows a cumulative Weibull distribution as both pressure and SiOil increase.

Effect of blade presence and SiOil VF on drop size





- Blade has no effect on emulsification under these operating conditions. Potentially due to the low volume fraction of SiOil.
- Volume fraction does not affect emulsification between 1% v/v and 10 % v/v. This confirms the amount of surfactant used in this study was adequate.

Modelling of DSDs

1	$p_v(d_i) = \frac{1}{d_i s_z \sqrt{2\pi}} \mathrm{EXP}\left[-\frac{[\ln(d_i)-\overline{d}]^2}{2{s_z}^2}\right]$
2	$f_v(d_i) = 100 \times \frac{p_v(d_i)}{\sum p_v(d_i)}$
3	$f_{v,T}(d_i) = \emptyset_s f_{v,S}(d_i) + (1-\emptyset_S) f_{v,D}(d_i)$
4	$\bar{d}_D = ln\left[A\Delta P^B\left(1 + C{\mu_d}^{3/4}\right)\right] + {s_{z,D}}^2$
5	$\overline{Mo}_S = \frac{\overline{Mo}_D}{D} \therefore \bar{d}_S = ln\left[\frac{A\Delta P^B\left(1 + C{\mu_d}^{3/4}\right)}{D}\right] + {s_{z,S}}^2$
6	$s_{z,S} = E{\mu_d}^F$
7	$s_{z,D} = G$
8	$\emptyset_S = \left(1 - EXP\left[-\left(\frac{\mu_d}{\beta_1}\right)^{\alpha_1}\right]\right) \times \left(1 - EXP\left[-\left(\frac{\Delta P}{\beta_2}\right)^{\alpha_2}\right]\right)$

$\}$ Log-norm distributions around each mode.

Total dist = volume weighted sum of both sub-dists.

Mode of large drops follows theory.

Mode of small drops is constant scale with large drops.

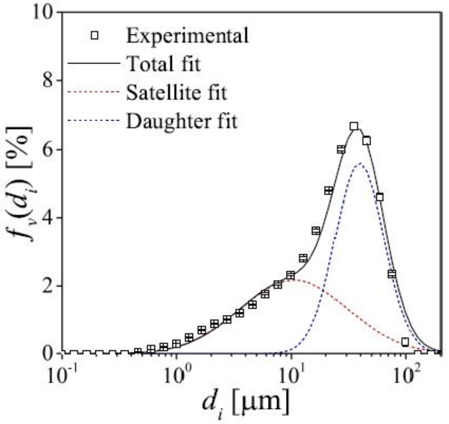
St Dev of small drops varies with viscosity.

St Dev of large drops is constant.

Vol frac of small drops varies with pressure and viscosity, as seen in contour plot.

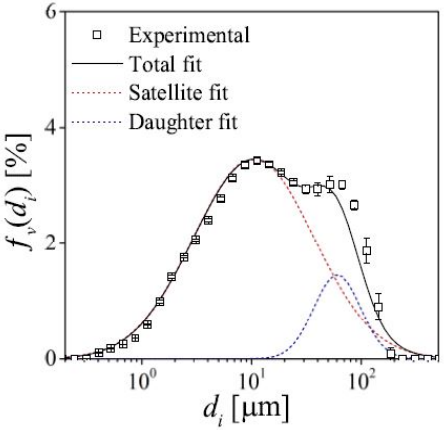
Fitted DSDs

0.003



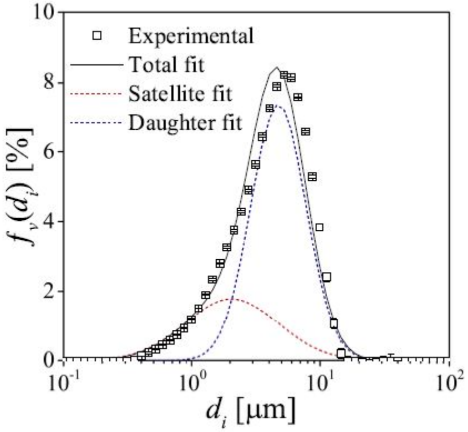
4.13 l min^{-1}, μ_d - 50 cSt.

0.001



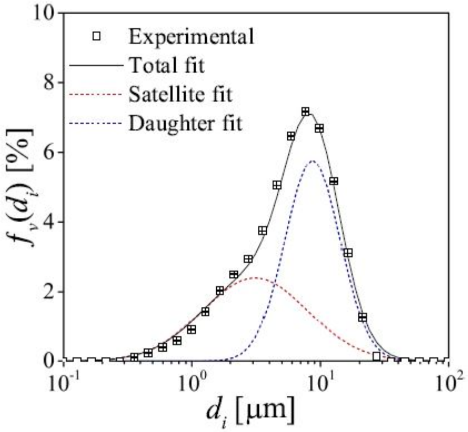
0.51 l min^{-1}, μ_d - 2760 cSt.

0.001



1.15 l min^{-1}, μ_d - 10 cSt.

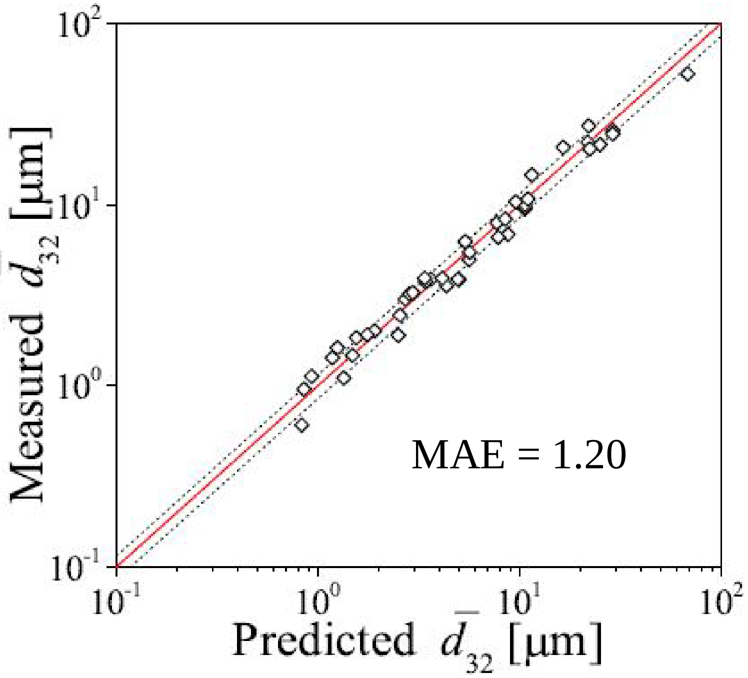
0.001



1.22 l min^{-1}, μ_d - 350 cSt.

- Model is able to accurately predict the DSD over a wide range of processing conditions.

Accuracy of the developed model



- Model is able to accurately predict the DSD over a wide range of processing conditions.
- We can therefore tune the DSD of our product to improve product quality and stability.

Scale-Up

$$Q = C_d A_o \sqrt{\frac{2\Delta P}{\rho_c}}$$

Scale-up based on constant pressure

$$\frac{Q_2}{Q_1} = \frac{A_{o2}}{A_{o1}}$$

To keep the same drop size distribution, need to keep pipe/orifice diameter constant.

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Conclusions

- We have developed a model that predicts the entire DSD of emulsions produced in a Sonolator for a wide range of process conditions.
- Blade presence had no effect on the DSD for the conditions investigated.
- Mechanistic droplet break-up models appear to be accurate at predicting relationship between drop size and μ_d in high pressure homogenisers.

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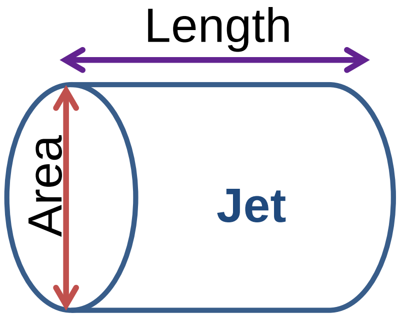
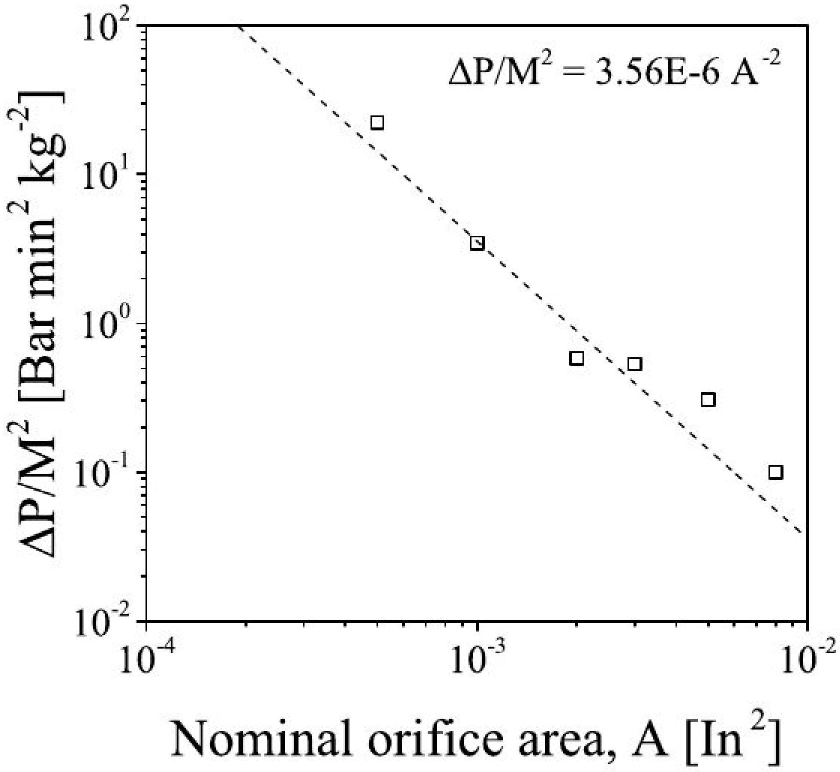


Why ΔP?

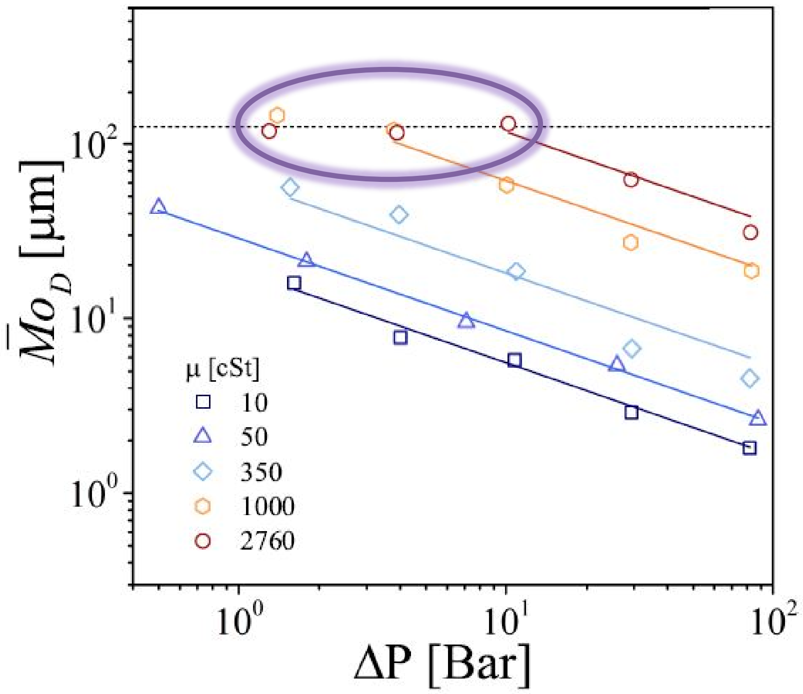
$\Delta P = \frac{\rho}{2{C_d}^2 A^2} Q^2$ Orifice plate theory $\bar{\varepsilon} = \frac{\Delta P\, Q}{\rho A L} = \frac{\Delta P^{3/2}\sqrt{2}C_d}{\sqrt{\rho}} \therefore d_{max} \propto \Delta P^{-0.6}$

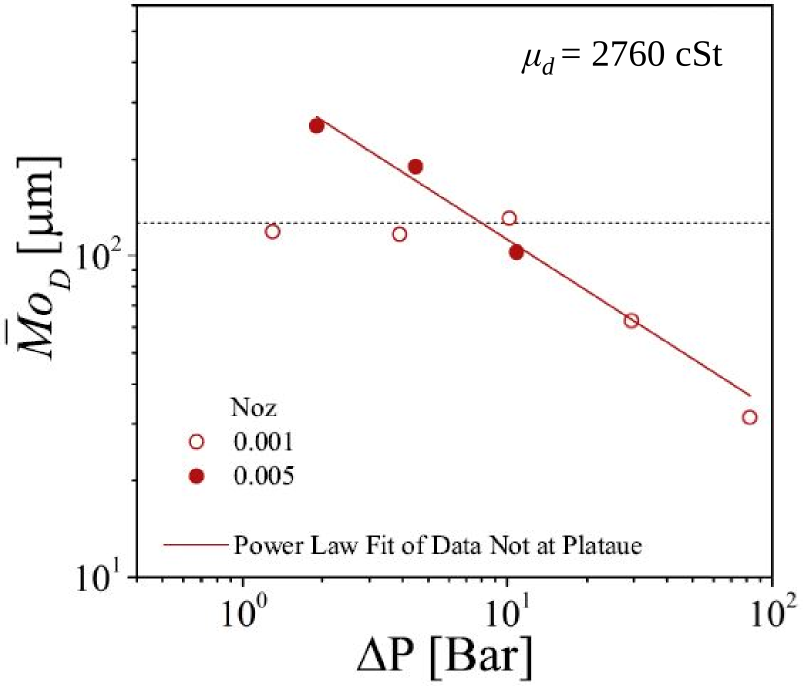


C_d = 11,850

Nominal area (In^2)	Predicted area (In^2)
0.0005	0.00040
0.001	0.0010
0.002	0.0025
0.003	0.0026
0.005	0.0034
0.008	0.0060

Effect of μ_d on drop size





- Effect of ΔP on mode of daughter droplets doesn’t change with SiOil viscosity, which is not expected from theory.
- Nozzle causes plateau in droplet size at low pressure drops\flow rates.