

Interfacial Polymerization kinetics: new insights on film formation using in-situ microscopy and particle-tracking

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Water & Energy Technologies
Laboratory

Introduction



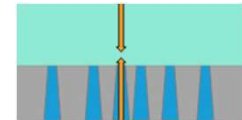
Interfacial Polymerization (IP)

Introduction

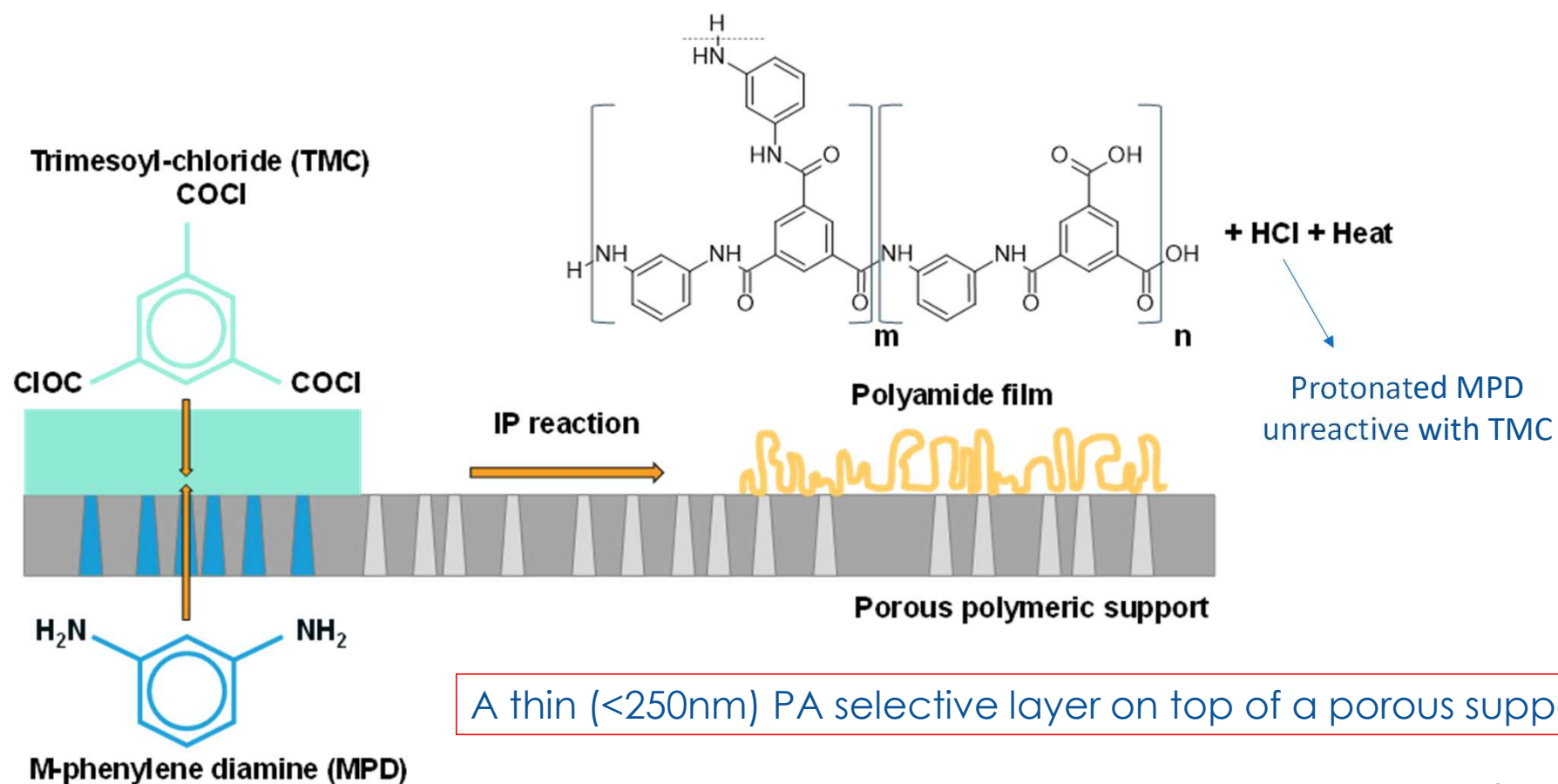


Interfacial Polymerization (IP)

- A polycondensation reaction occurs at the interface



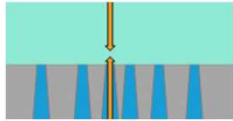
IP Interfacial Polymerization



Introduction



Interfacial Polymerization (IP)

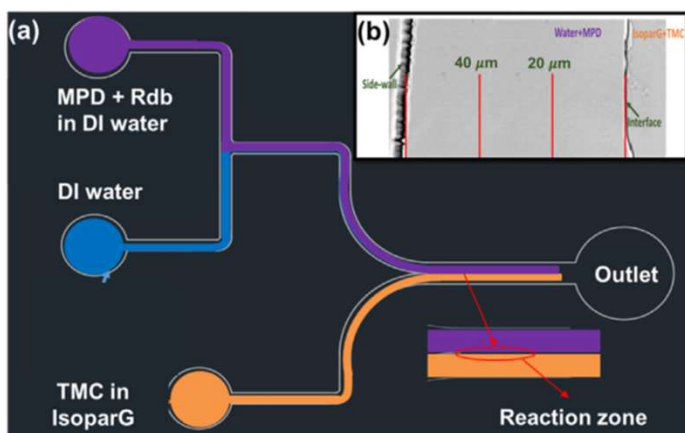
- A polycondensation reaction occurs at the interface 
- IP is used to fabricate thin-film composite (TFC) polyamide (PA) membranes first synthesized in late 70s by Cadotte et al.
- State-of-the-art desalination by reverse osmosis (RO) **>99% rejection**

Introduction

How can we understand more about IP?

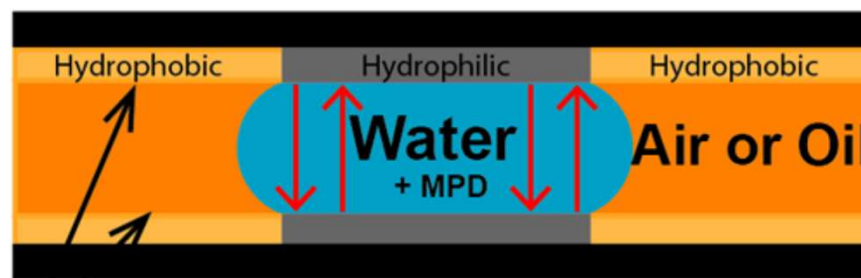
In-situ monitoring – insight of reaction kinetics

Heat transfer



Ukrainsky and Ramon, *JMS* (2018)

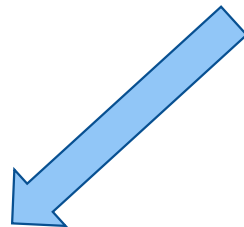
Mass transfer



Nowbahar et al., *J. Am. Chem. Soc.* (2018)

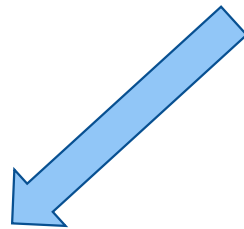
The product of IP:

Crumpled polyamide film



The product of IP:

Crumpled polyamide film



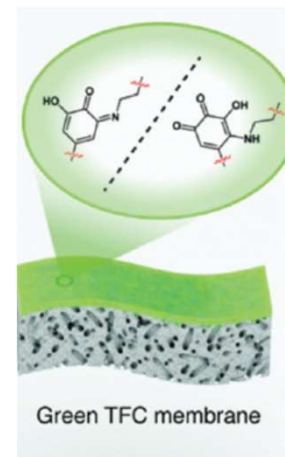
Why ?

Why ?

Synthesis — Morphology — Performance

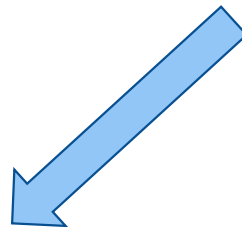


- ✓ Improve existing membranes
- ✓ Move towards 'green materials'

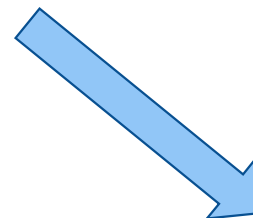


The product of IP:

Crumpled polyamide film



Why ?



How ?

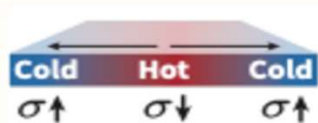
How ?

Instability mechanisms

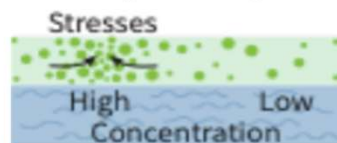
**Bubble
formation**



**Thermo-
capillarity**



**Soluto-
capillarity**

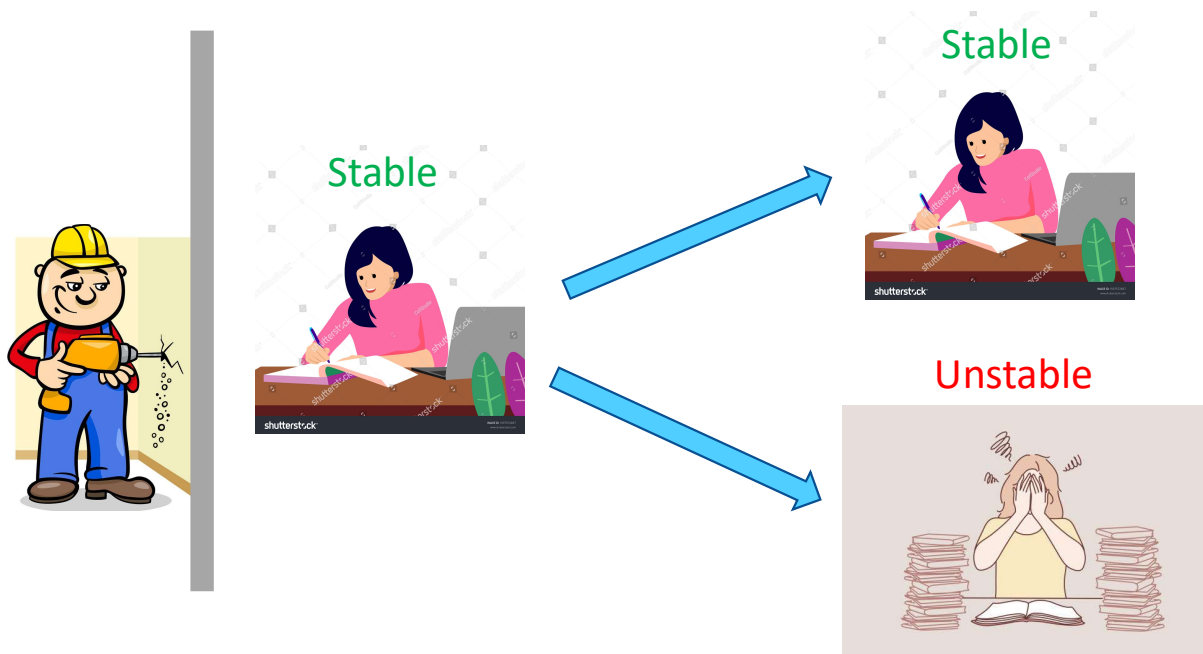


**Elastic
crumpling**

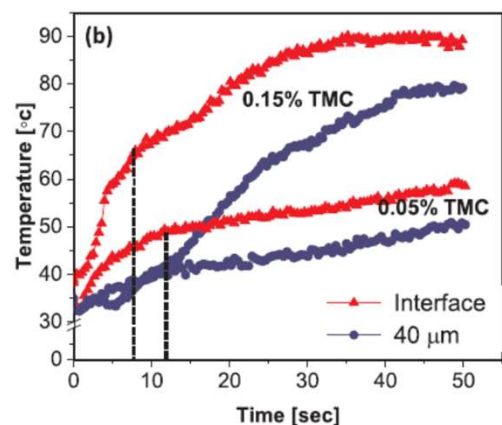


Instability

The **inability** of a system to **sustain** itself against small **perturbations**.



Transition from **stable** to **unstable**: e.g., laminar to turbulent flow



Ukrainsky and Ramon, *JMS* (2018)

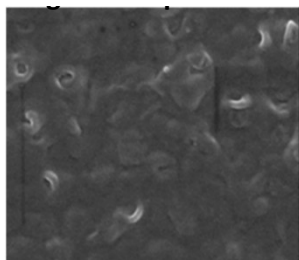
Local overheating in the reaction zone

Bubble
formation

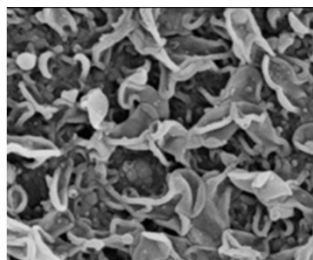


Nanobubbling

No soluble gasses



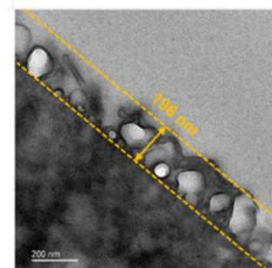
With soluble gasses



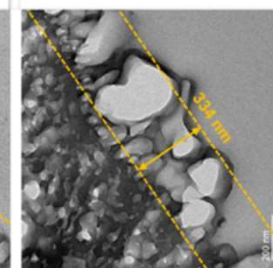
Ma et al., *Environ. Sci. Technol. Lett.* (2018)

Vaporization

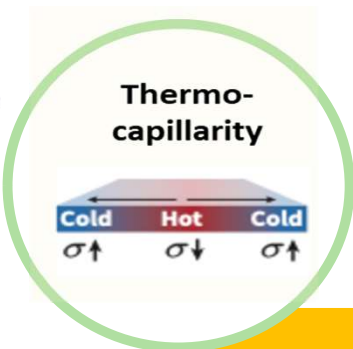
TFC-hexane



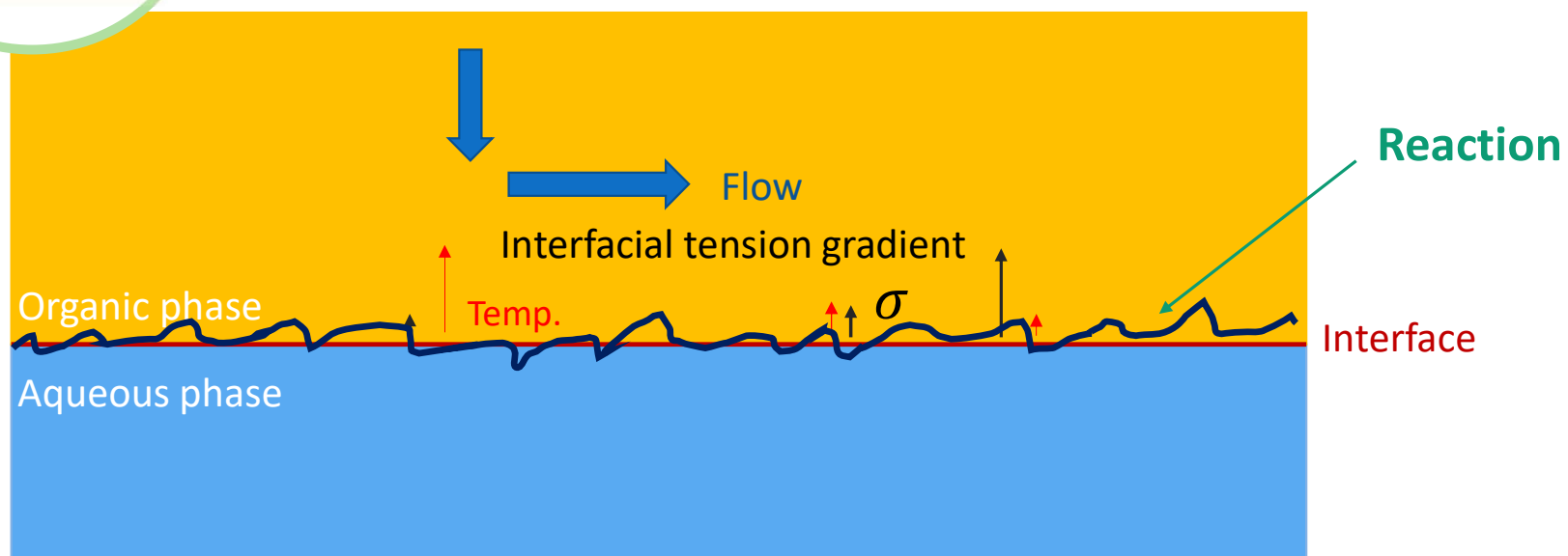
TFC-pentane



Peng et al., *JMS* (2021)

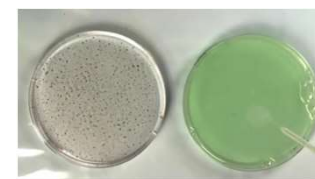


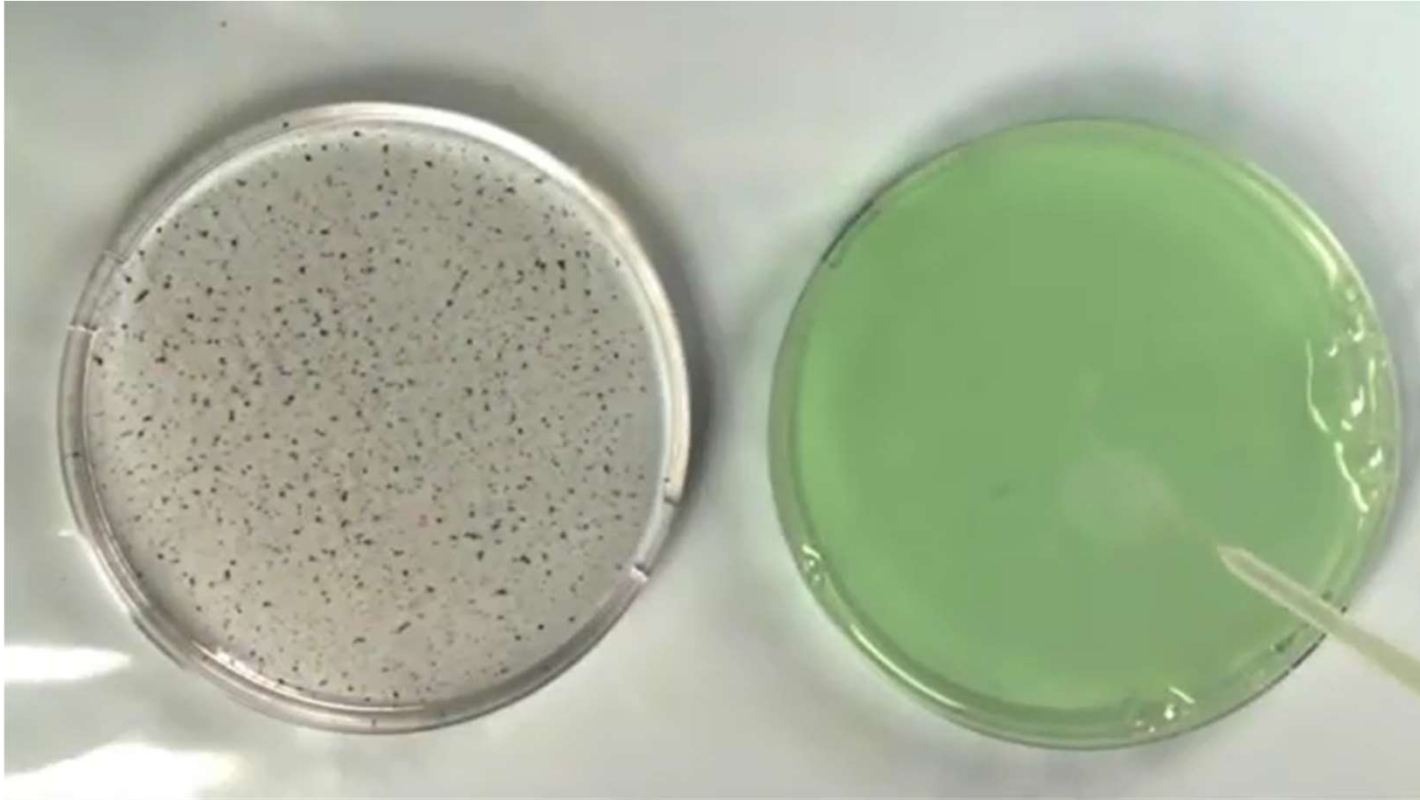
IP system



Gradients in interfacial tension drive a flow:

Marangoni flow



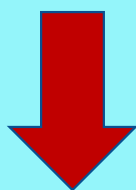


How ?

During film formation

After film formation

Instability= flow in IP system



Film Crumpling



Local
the reaction zone
causes 'degassing'

**Elastic
crumpling**



Wrinkling of the formed
film due to different
elastic properties
between the film and
the support

The product:

Crumpled polyamide film



Journal of Membrane Science

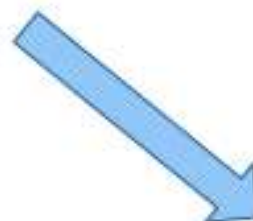
Available online 10 May 2022, 120593

In Press, Journal Pre-proof ?



Re-thinking polyamide thin film formation: How does interfacial destabilization dictate film morphology?

Ines Nulens^{a, 1}, Adi Ben-Zvi^{b, c, 1}, Ivo F.J. Vankelecom^a, Guy Z. Ramon^{b, c, d}  



How ?

Methods

How can we observe a flow in IP ?



Microfluidic device

Aqueous phase: fluorescent particles ($1\mu\text{m}$) + MPD

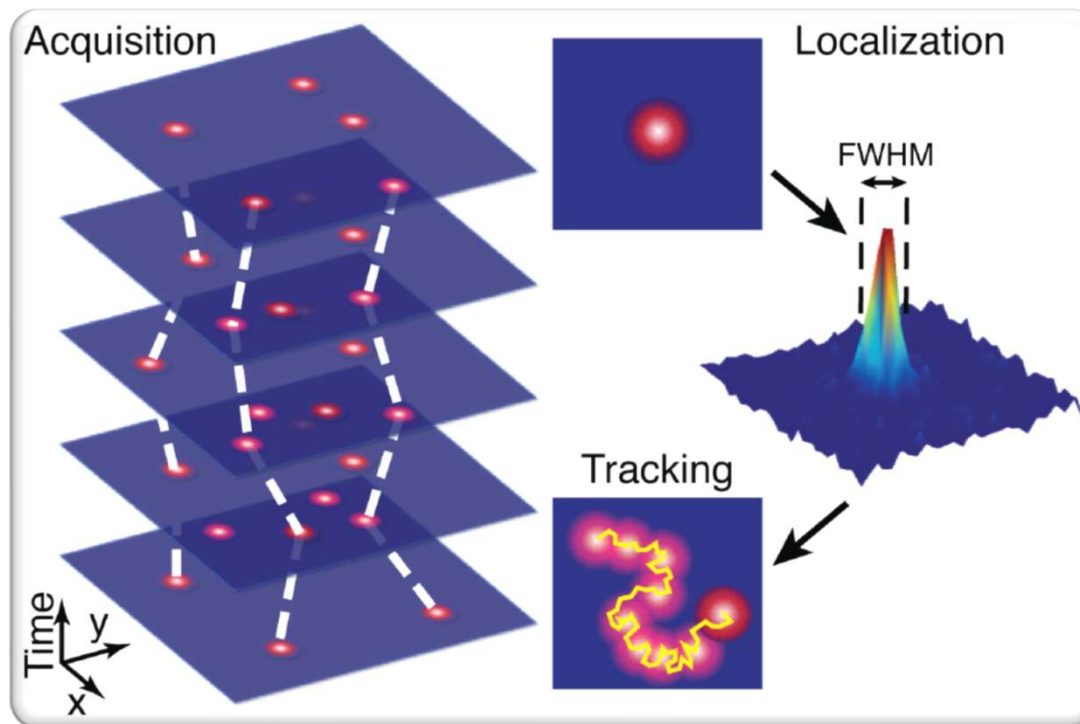
Organic phase: Isopar-G + TMC



Confocal Microscopy

Videos of 2D image over time

Particle Tracking



Acquisition of the displacement using confocal microscopy

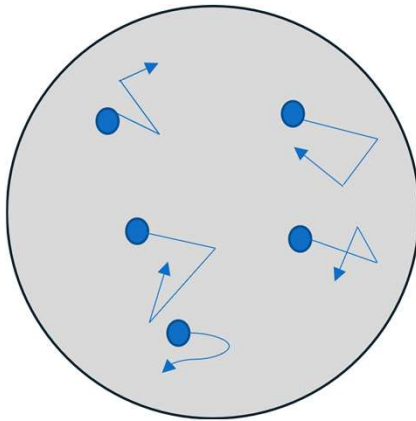
Manzo et al., Rep. on Prog. in Phys. (2015)

What do we expect to see?

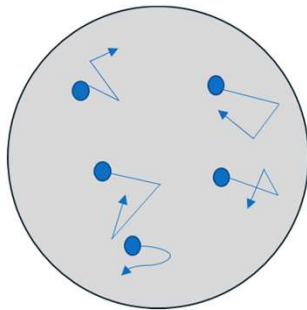


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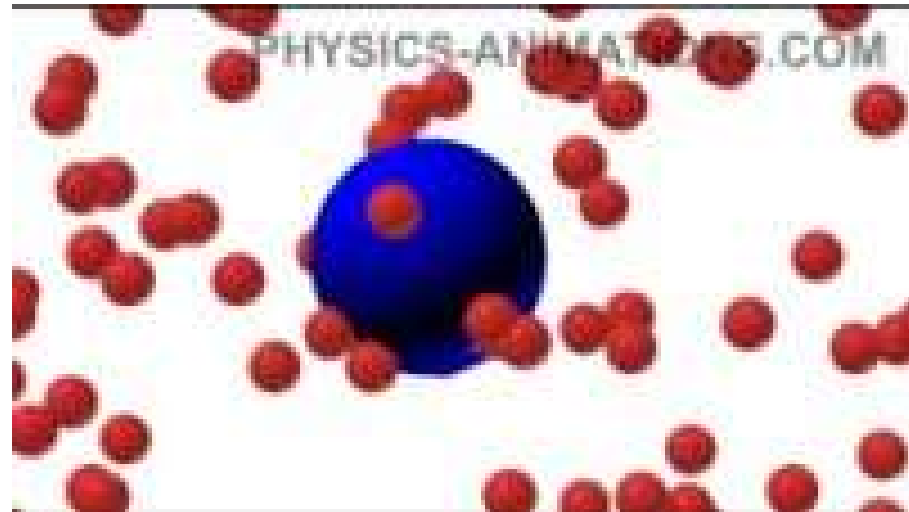
Random motion (Brownian)



Brownian motion



- No bulk flow.
- The motion is thermal-driven.



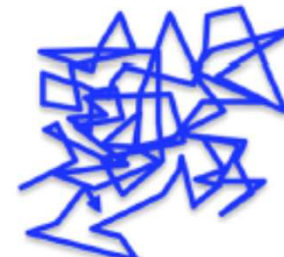
Stokes-Einstein relation:

$$D = \frac{k_B T}{6\pi \eta r}$$

Thermal energy
Viscous friction

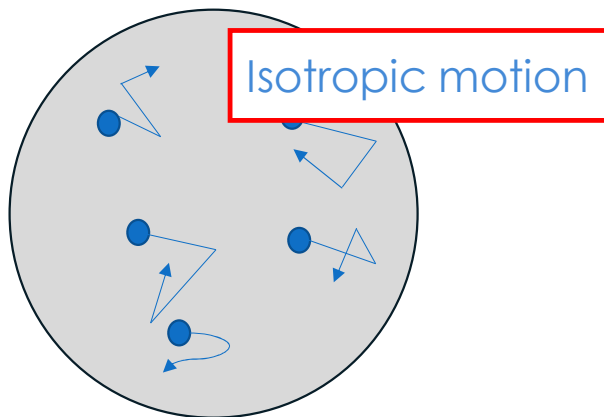
k_B Boltzmann constant
 T temperature
 η dynamic viscosity
 r radius of particle

Trajectories:



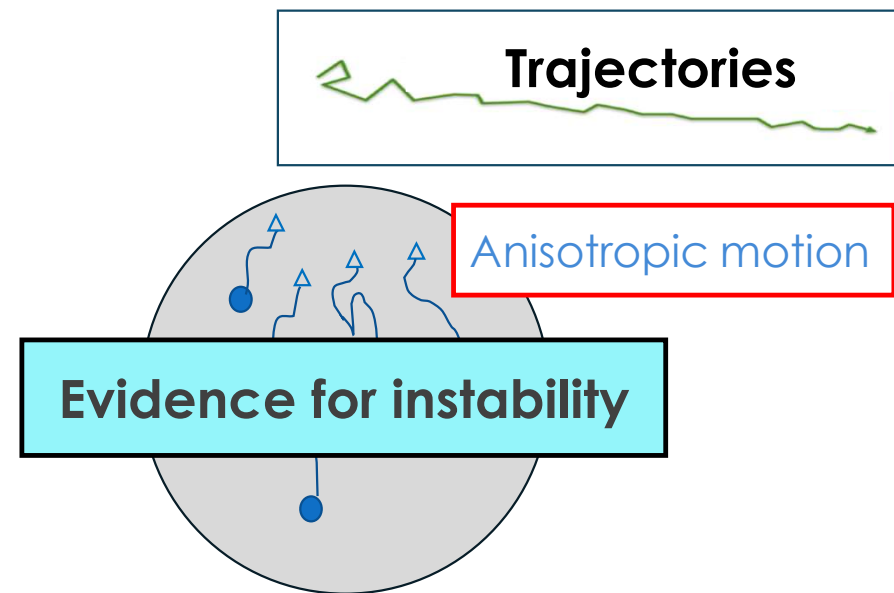
What do we expect to see?

Random motion (Brownian)



- No bulk flow.
- The motion is thermal-driven.

Directed motion



- Particles act as tracers that move with the bulk.
- **Brownian + bulk directed motion**

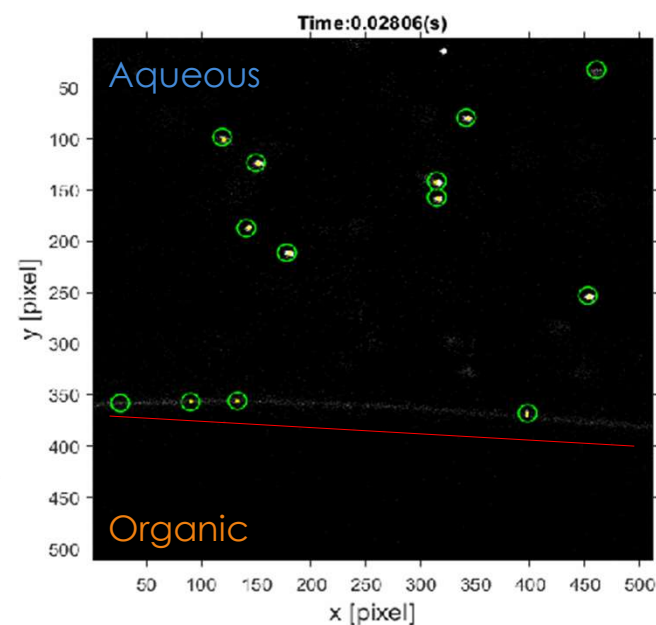
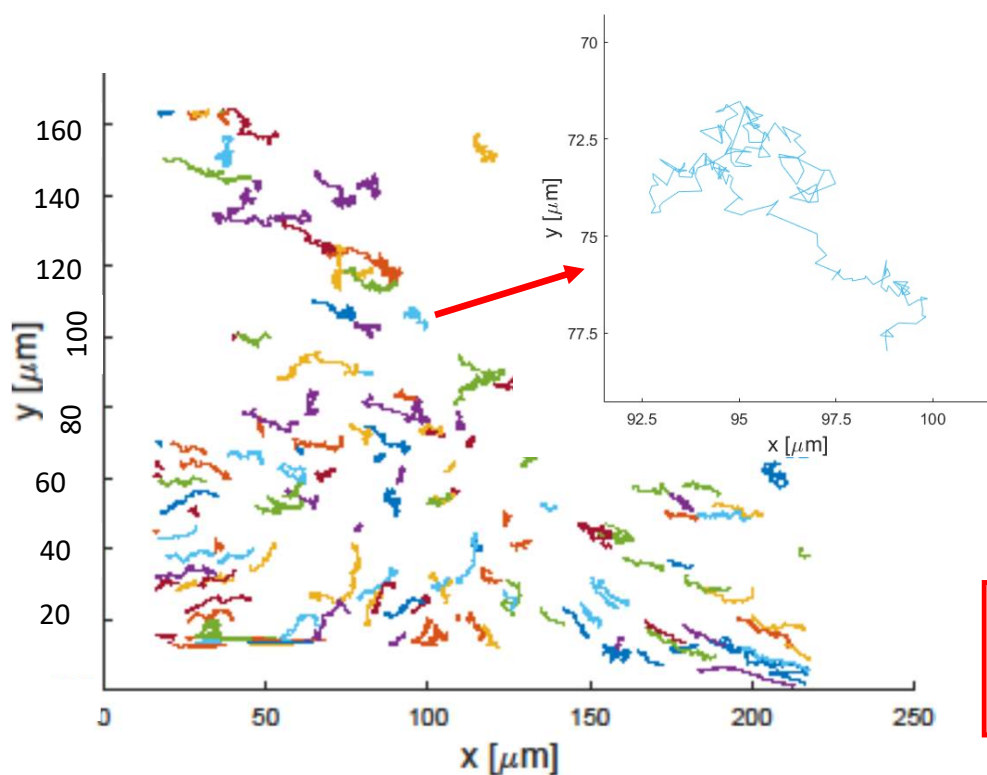
Results



Observed trajectories

Blank

DW; Isopar-G



- Non-directed motion of particles
- The motion has Brownian behavior

Results

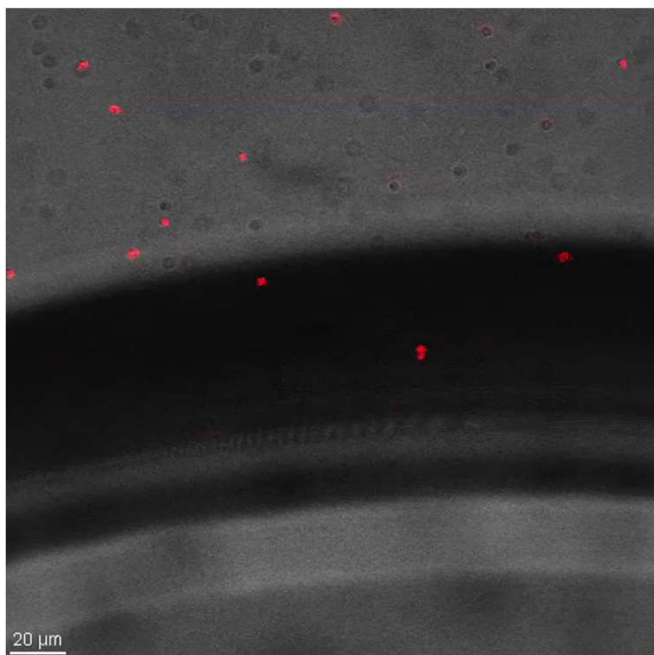
Videos



Low concentrations:

DW+ 0.02% MPD

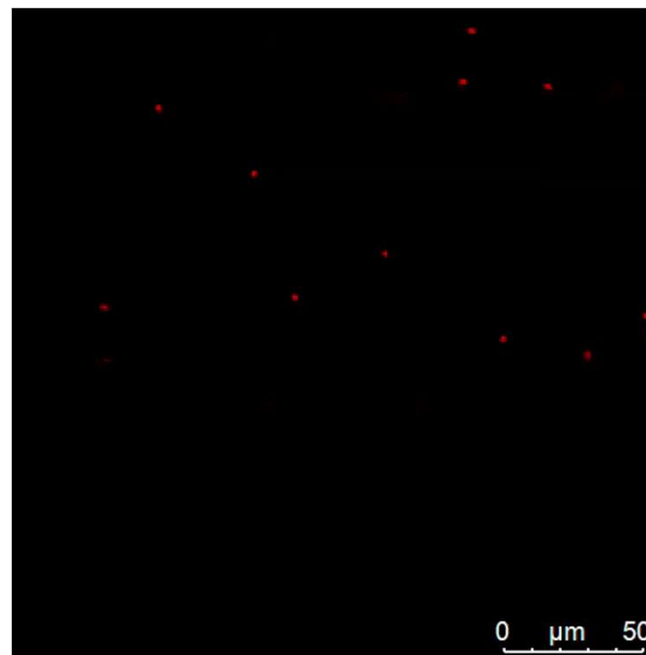
Isopar-G + 0.001% TMC



High concentrations:

DW+ 2% MPD

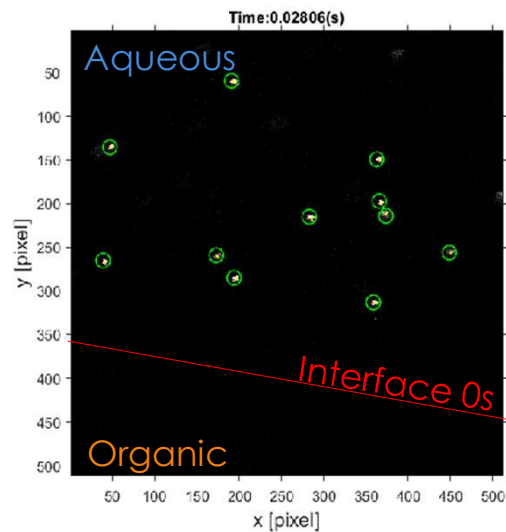
Isopar-G + 0.15% TMC



Results

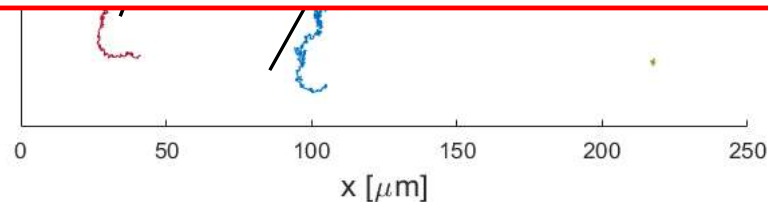
Observed trajectories

Low concentrations

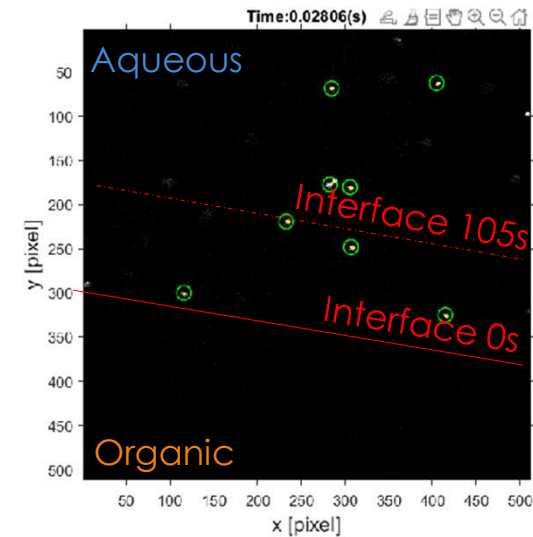


210

- More directed motion of particles than the blank
- The particles that are closer to the interface have more directed flow



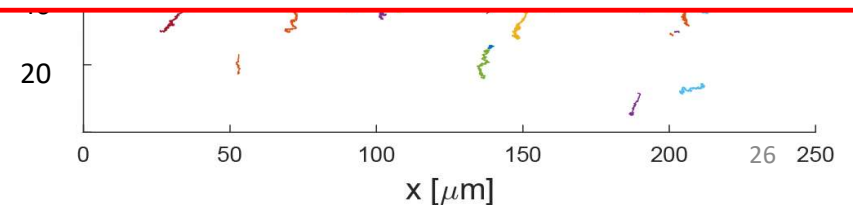
High concentrations



160

140

- Directed motion of particles towards the interface
- The particles reach the interface and remain there

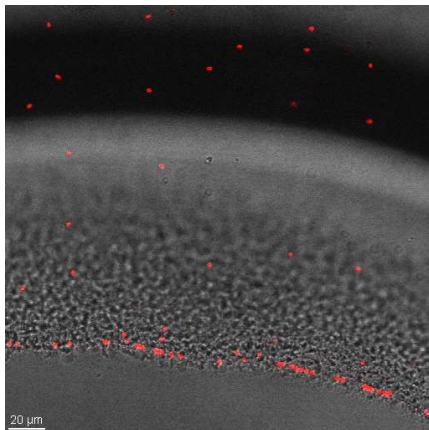


Results

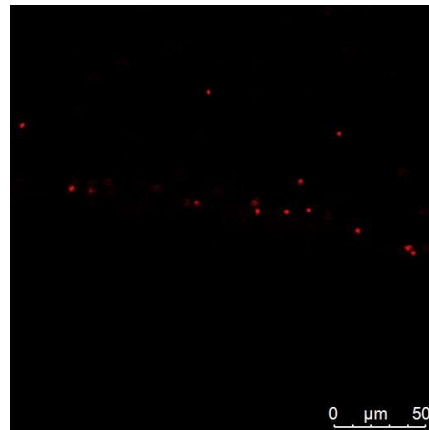
Images

High concentrations

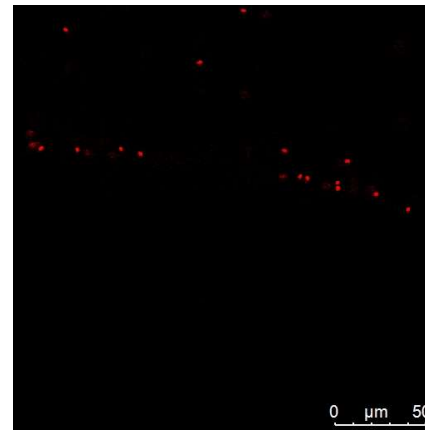
Hight 0



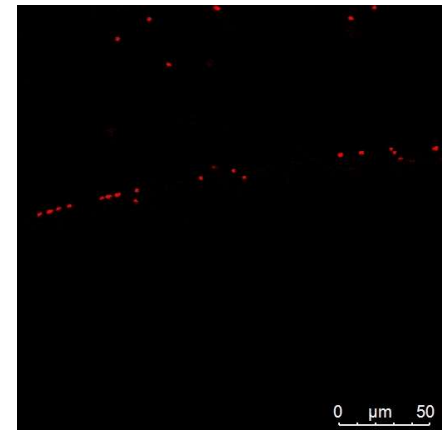
Hight 50 µm



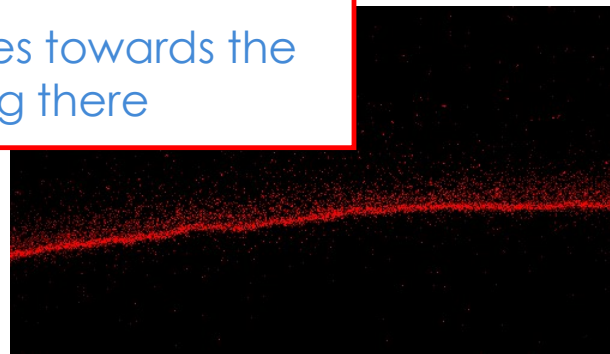
Hight 100 µm



Hight 450 µm



- A movement of particles towards the interface and remaining there



Hight 0
0.1 µm particles

Conclusions

- Different motion behaviors between blank, low, and high monomer concentrations.
- At higher monomer concentrations a directed motion was observed.
- At high monomer concentrations there is a motion towards the interface.
- Tracking particles provides us with new insights about IP.

Future work:

- Data analysis.

Conclusions

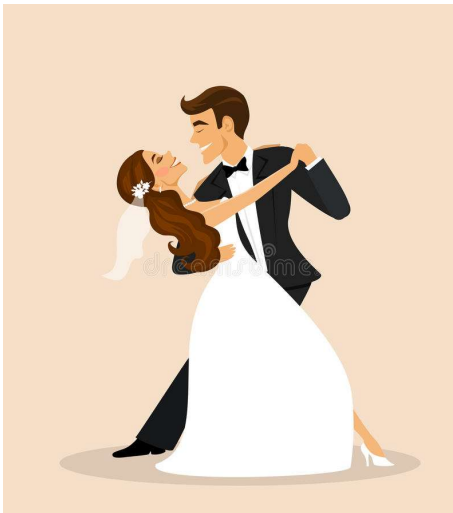
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Future work:

- Data analysis.
- The other side of the picture.



Aqueous phase



?

Organic phase

interface



Conclusions

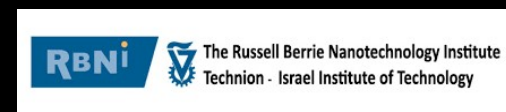
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Future work:

- Data analysis.
- The other side of the picture.
- Kinetics of film formation?



Acknowledgements



Thank you for
Listening 😊