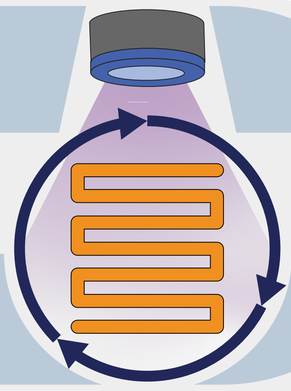
Characterization of continuous-flow photoreactors

The effect of gas bubbles on photon absorption in flow reactors



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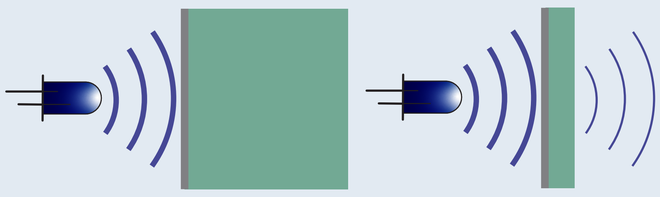
Determining photon absorption

- Photon balance:

$$\text{Incident} = \text{Transmitted} + \text{Absorbed}$$

- Relevant for small dimensions

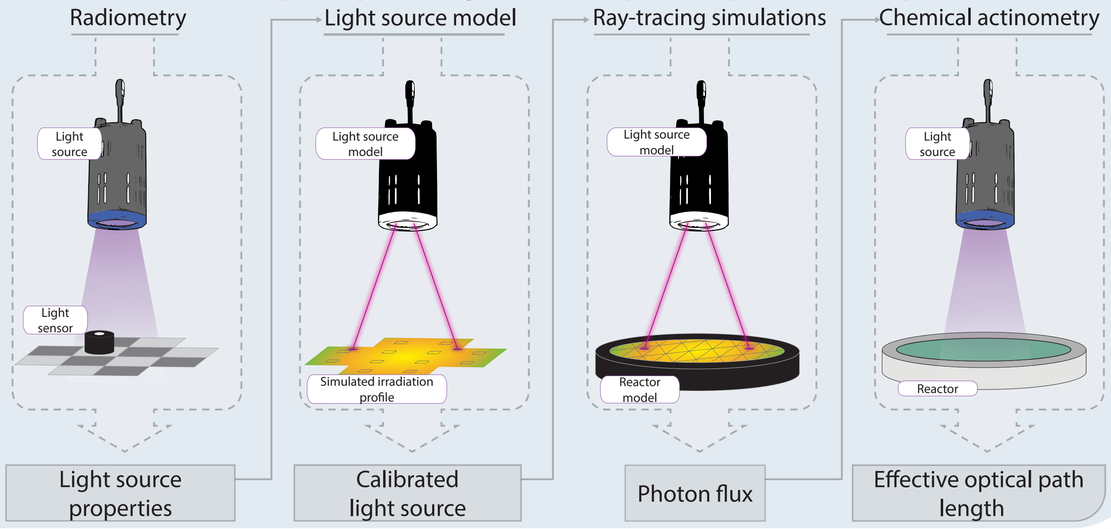


Large-scale batch:
Uses almost all photons
Incident = Absorbed

Continuous-flow:
Transmits photons

- Complex task to determine!

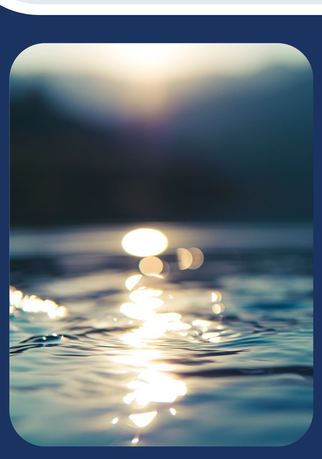
- Proposed strategy:
  - Decouple multiple parameters
  - Obtain effective optical path length as descriptor for photon absorption



Previous work:
Determining photon flux and effective optical path length in intensified flow photoreactors

Stefan D. A. Zondag, Jasper H. A. Schuurmans, Arnab Chaudhuri, Robin P. L. Visser, Cíntia Soares, Natan Padoin, Koen P. L. Kuijpers, Matthieu Dorbec, John van der Schaaf & Timothy Noël

Nat. Chem. Eng., 2024, 1, 462–471



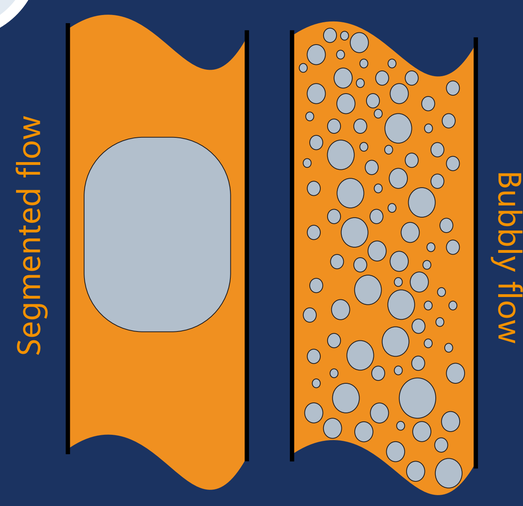
Gas-liquid interfaces

Light interactions

- Light reflects and refracts at interfaces
- Refractive index air ≈ 1.0
- Refractive index solvent ≈ 1.3
- Total internal reflection

Hydrodynamics

- Thin films (no straightforward path length)
- Different flow regimes
- Segmented flow
- Bubbly flow



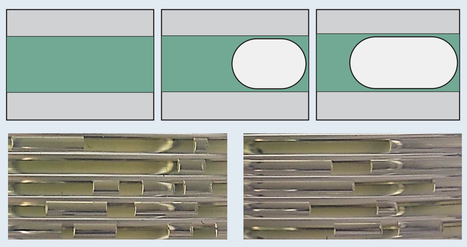
$$2\,[Fe(III)(C_2O_4)_3]^{3-} \xrightarrow{h\nu} 2\,[Fe(II)(C_2O_4)_2]^{2-} + 2\,CO_2 + C_2O_4^{2-}$$

• Addition of inert N_2

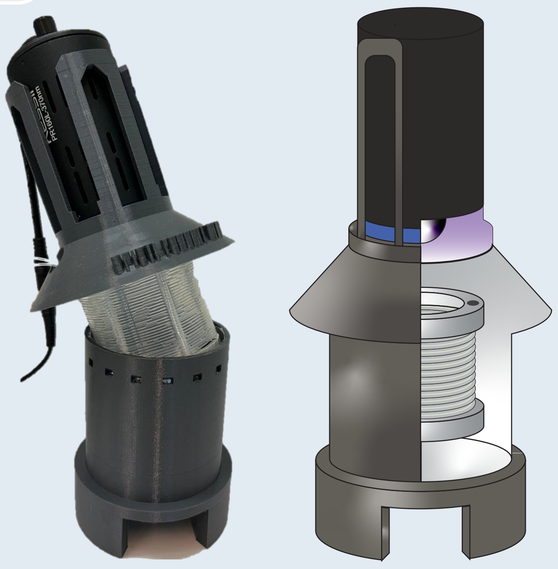
Capillary reactor

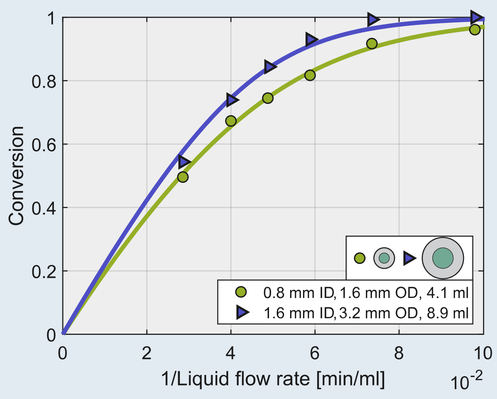
Uflow[1] reactor

- 3D printed custom design
- Available for everyone
- Benchmarked light source (Kessil)
- Upon gas phase introduction segmented flow is established

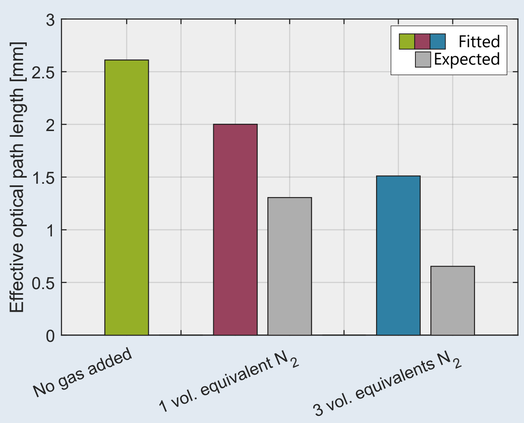


Segmented flow





- Different capillary sizes and reactor volume yield similar results
- Curvature and refraction of capillary
- Light concentation on reactor volume
- Higher space-time yield achieved

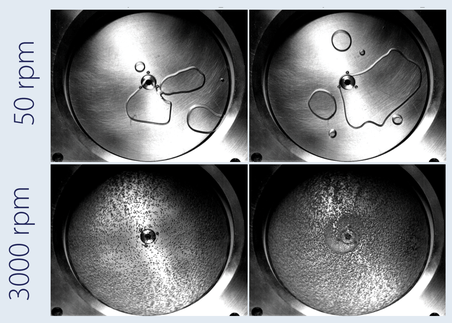


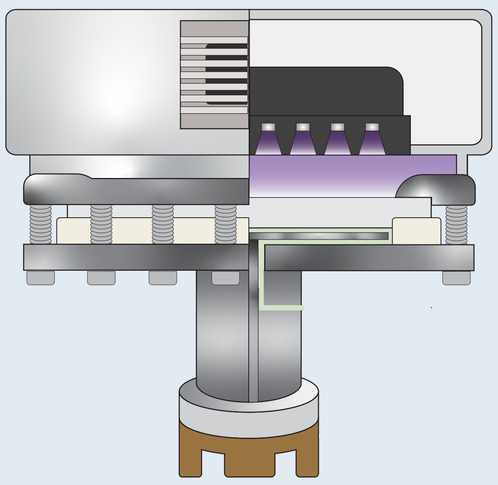
- Effective optical path length as direct equivalent for photon absorption
- Gas leads to photon absorption losses
- Losses increase with slug size
- Effect is lower than expected

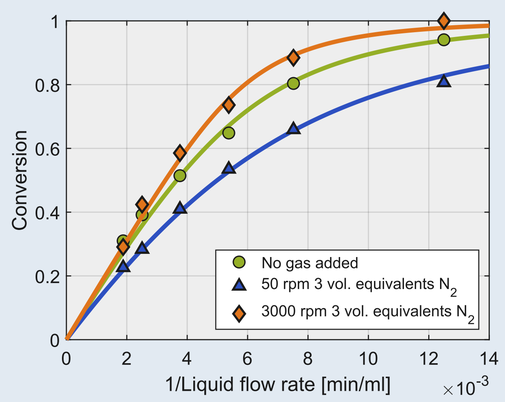
High-shear reactor

Rotor-stator spinning disk reactor

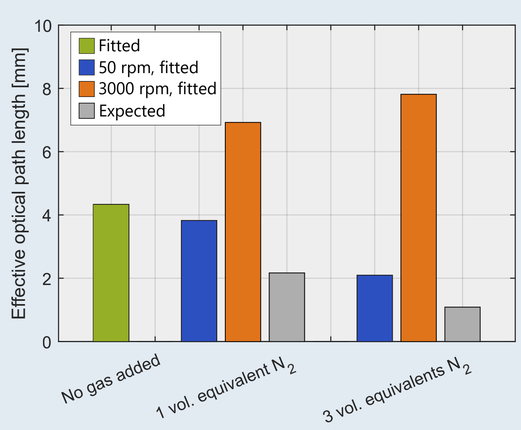
- Rotating disk, high rpm possible
- Decouple mixing from residence time
- Varying bubble behavior;
  - Low rotation speeds large bubbles
  - High rotation speed small bubbles







- Gas introduction shows two effects
- High rotation speed increases conversion
- Throughput can increase with the introduction of a gas



- Gas dispersed in small bubbles can increase photon absorption
- Large bubbles at low rotation speed show similar behavior to segemented flow
- Effect is lower than expected

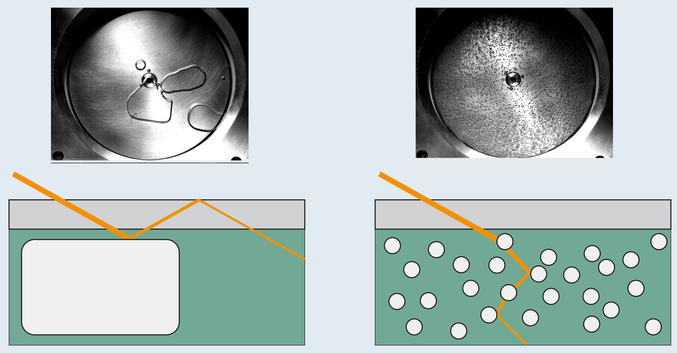
Interaction of Light with Gas-Liquid Interfaces: Influence on Photon Absorption in Continuous-Flow Photoreactors

Jasper H. A. Schuurmans, Stefan D. A. Zondag, Arnab Chaudhuri, Miguel Claros, John van der Schaaf & Timothy Noël

Bubbles contrentrate light on the liquid

Large bubbles

- Photon absorption losses, nonetheless lower than expected
- Photons leaving after single pass find their way back in the liquid
- Total internal reflection between multiple interfaces:
  - Liquid-gas
  - Reactor material-surrounding air



Small bubbles

- Photon absorption increases
- Gas bubbles can elongated optical path lengths
- Reflection and refraction can contribute:
  - Randomization of directions
  - Increased total interal reflection

[1] T.M. Masson, S.D.A. Zondag, J. H. A. Schuurmans, T. Noël, Open-source 3D printed reactors for reproducible batch and continuous-flow photon-induced chemistry: design and characterization. React. Chem. Eng. 9, 2218–2225 (2024)

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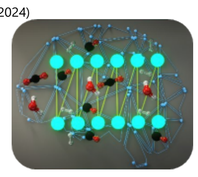

CATART PROJECT

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