# Master's Thesis Engineering Technology 2023-2024

# Characterization of micromixing in batch and SSE reactor for high viscous systems

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## Introduction

**Goal:** Sustainable processes

**Requirement:** Solvent-free process intensification

**Challenges:** High viscosity & inefficient mixing

**Solution:** High shear mixing in single screw extrusion reactor with specialized mixing elements

**Thesis goal:** Characterization of micromixing in screw reactor

**Protocol:** Villermaux-Dushmann method

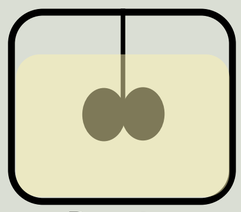
## Principle: Villermaux-Dushmann method

**Parallel competitive reactions** [1]

**(1)** $H_2BO_3^- + H^+ \rightleftharpoons H_3BO_3$

**(2)** $IO_3^- + 5I^- + 6H^+ \rightleftharpoons 3I_2 + 3H_2O$

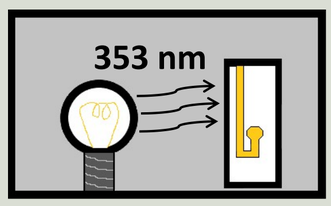
Excess $I^-$ → $I_3^-$



Reactor

Degree of $H^+$ excess relates to amount of $I_3^-$ formed

Spectrophotometry




Absorbance

Low concentration of triiodide $I_3^-$ = Lower Segregation index $X_s$

More efficient micromixing

## Batch experiments

Figure 1 shows a schematic representation of the batch setup.

- Solution A (Acid) was injected into the reactor filled with solution B (Buffer) while stirred.
- After injection of acid, samples were taken and analyzed spectrophotometrically.
- Both 1 mPa·s and 100 mPa·s solutions were tested.
- Benchmark for flow-experiments.

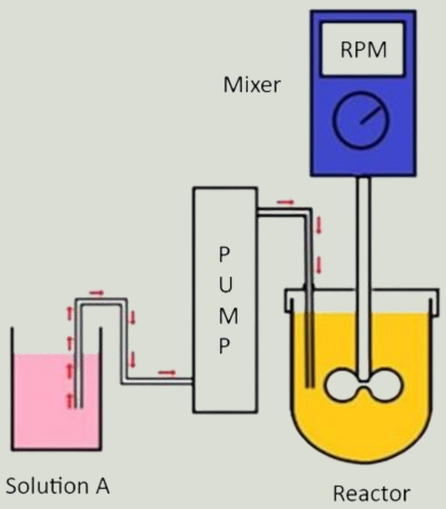



*Figure 1: Batch setup [1].*

## Flow experiments

Figure 2 shows flow reactor setup and the used mixing elements and one of the studied configurations

- Buffer and acid solution were injected into the reactor.
- The screw rotation transports and mixes the reagents.
- After mixing, the mixture was analyzed spectrophotometrically.
- Both 1 mPa·s and 100 mPa·s solutions were tested.
- Alternative screw configurations were tested.

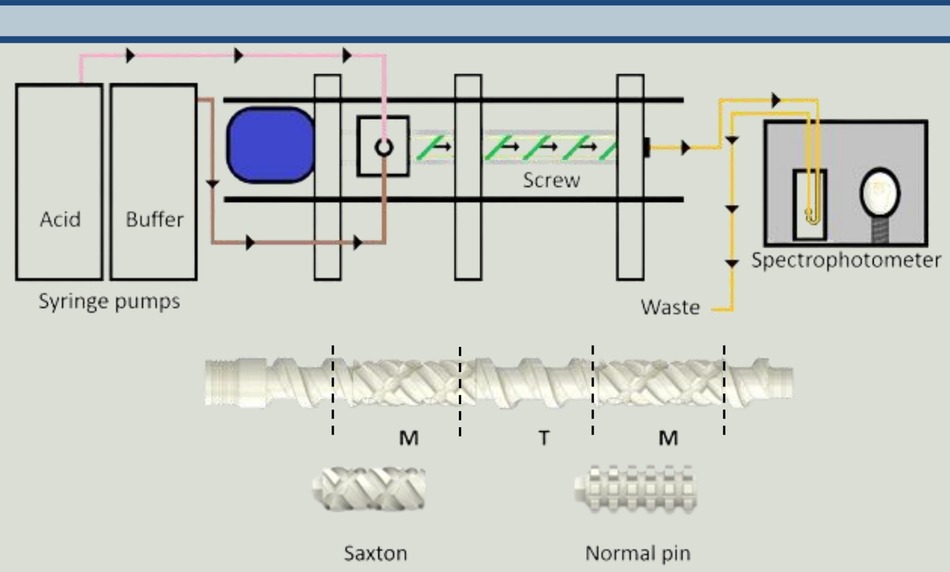



*Figure 2: Flow experiment setup (top), used mixing element Normal pin mixer and Saxton mixer and assembled screw with configuration (bottom) M-T-M.*

## Results

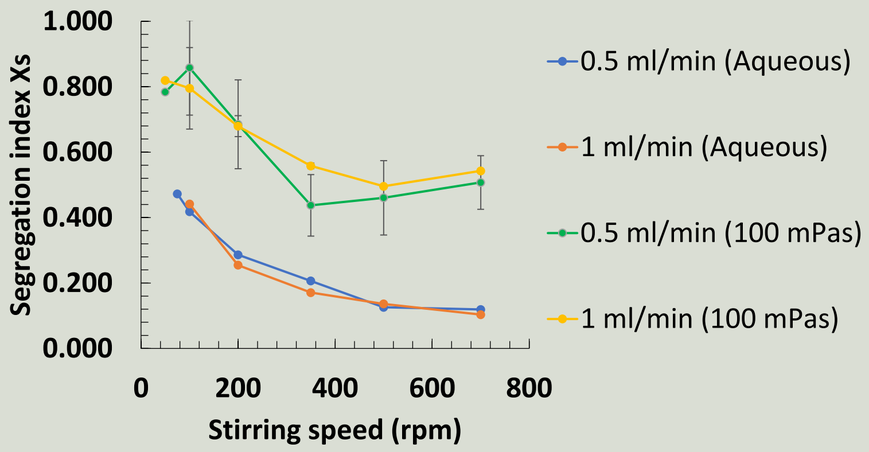



*Figure 3: Results from batch experiments in OptiMax reactor for aqueous and 100 mPa·s solutions.*

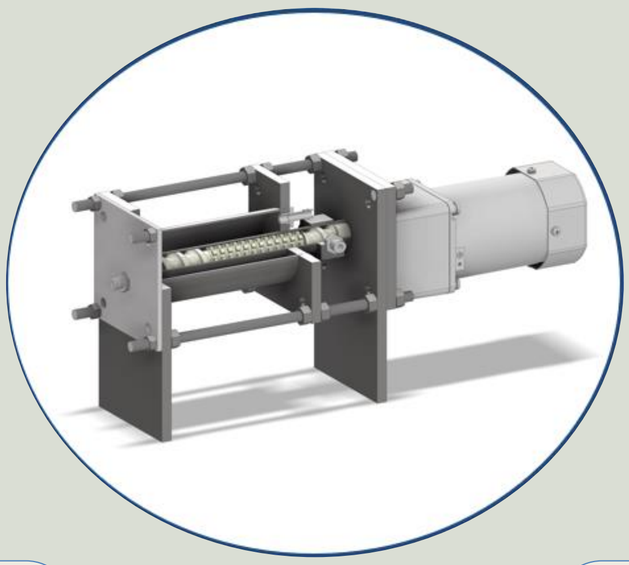


*Figure 5: Benchtop single screw extrusion reaction.*

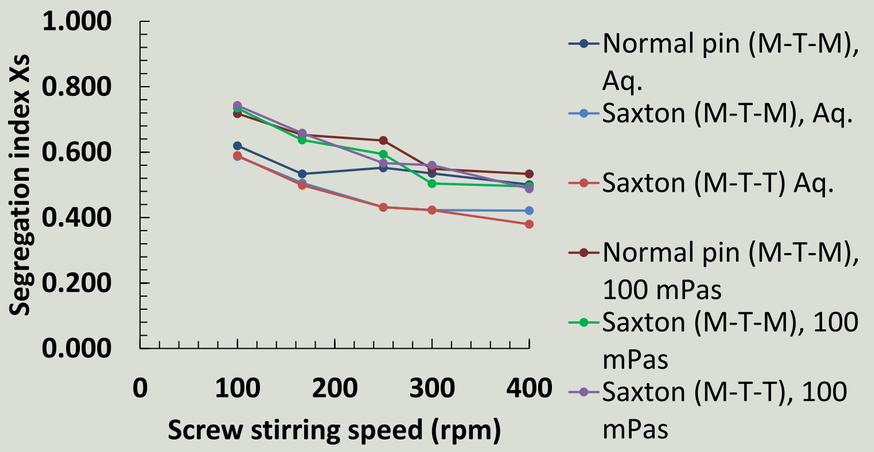



*Figure 4: Results from flow experiments in screw reactor for aqueous and 100 mPa·s solutions. Injection flow rates of the buffer and acid are 20,0 and 0.160 ml/min*

Figure 3 shows that micromixing efficiency in the OptiMax reactor is larger in aqueous solutions than in 100 mPa·s solutions. Injection flow rates 0.5 ml/min and 1 ml/min exhibit no macromixing limitations and are thus suitable for studying micromixing. The segregation index follows a decreasing exponential with increasing stirring speed which corresponds with literature [1].

Figure 4 shows that the segregation index follows a similar trend with increasing stirring speed as observed for the batch reactor.
Additionally, this figure shows:
that micromixing efficiency in the screw reactor is optimal if:

- Saxton mixing elements are used,
- Aqueous solutions are mixed

Micromixing efficiency is independent of the screw configuration

## Conclusion

- Micromixing is **more efficient** in **aqueous solution** and **less efficient** in **viscous solution** for both batch and screw reactors.
- **Saxton mixing elements** provide better micromixing than Normal pin mixing elements.
- **Screw configuration does not affect** micromixing efficiency.

## Opportunities

- Investigating other mixing elements and higher viscosities (1 Pa·s and 10 Pa·s ).
- Optimization of viscosifying agent (use vs cost).
- Comparing batch and flow data through python programming.

**Dulmage mixer** **Pineapple pin mixer**

*Figure 6: Alternative mixing elements.*

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ing. Jonas Loncke

## References:

[1] P. Guichardon and L. Falk, "Characterization of micromixing efficiency by the iodide-iodate reaction system. Part I: Experimental procedure," Chem Eng Sci, vol. 55, no. 19, pp. 4233–4243, 2000, doi: 10.1016/S0009-2509(00)00068-3.

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