Integrated continuous flow photoreactors: Photooxidation of (L)-methionine with singlet oxygen

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1 | INTRODUCTION

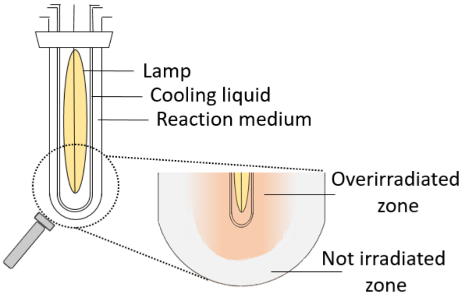
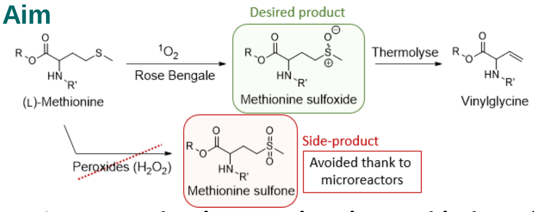


Figure 1 : Usual immersion well with a lack of homogeneous irradiation

Development of photochemistry in macroscopic **batch** reaction vessels is hampered due to inherent limitations: **superficial light penetration** and **poor heat exchange** result in inhomogeneous irradiation and hence to side-reactions or product degradation due to **overexposure**. The recent implementation of photochemical processes in **microreactors** under continuous-flow conditions appeared to be **much more powerful** than its batch analogue in terms of **irradiation efficiency**, light penetration and **excellent heat exchange**. Furthermore, the fine control of residence time ensures an accurate control of the irradiation time, avoiding side-reactions and degradation.

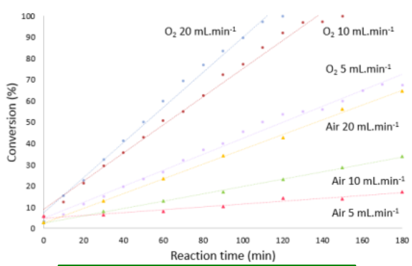
2 | Aim



The aim was to **implement the photooxidation of (L)-methionine with singlet oxygen in microreactor** under continuous-flow conditions to avoid the formation of undesired side-product and improve the efficiency of the reaction.

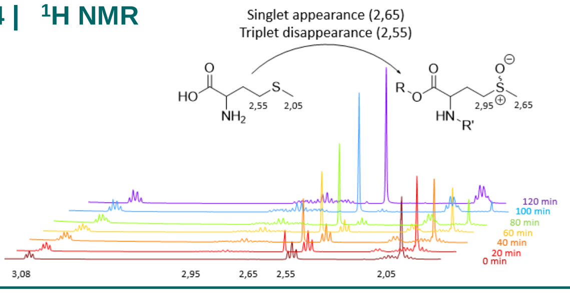
3 | Photooxidation in batch immersion well

Methionine + Rose Bengal in water with O_2 or Air bubbling

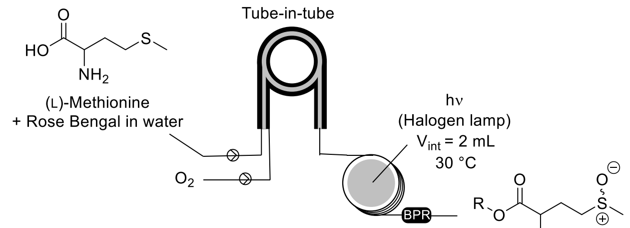


✓ ↗ [Rose Bengal]
↗ lamp intensity
✓ O_2 rather than air
Total conversion in 2 h

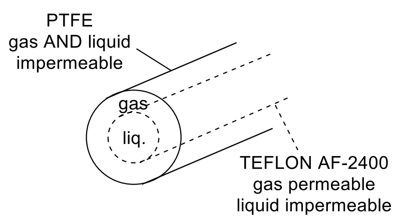
4 | ^{1}H NMR



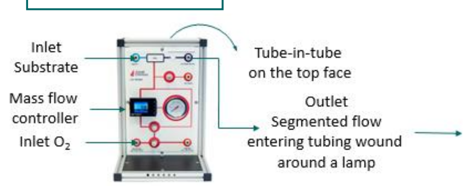
5 | Microreactors

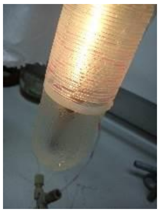


Tube-in-tube



General setup





6 | Results

Substrate solution	O_2	BPR (bar)	Residence Time	Conversion
0.1 ml.min^{-1}	15 mL.min^{-1}	8	20 min	100%

Total conversion of (L)-methionine into methionine sulfoxide could be reached in only **20 min**.
(Shorter residence times or lower O_2 flow rates were not sufficient and could not lead to 100% conversion)

7 | Conclusion

The photooxidation of (L)-methionine with singlet oxygen using Rose Bengal as a sensitizer was successfully implemented in a microreactor setup and led to total and selective conversion into methionine sulfoxide, an important building block for the organic synthesis of peptides or functionalized amino acid. The reaction was performed **in 20 min** while the same reaction in batch took 2h.

8 | Acknowledgements

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