

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

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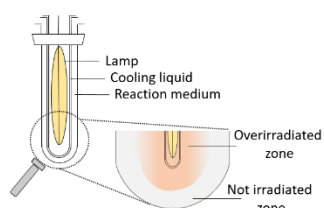


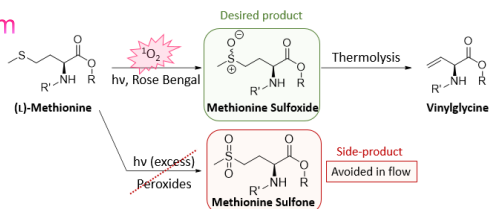
Figure 1 :

Usual immersion well with a lack of homogeneous irradiation

1 | INTRODUCTION

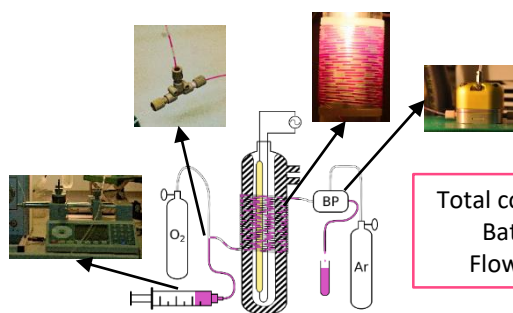
Development of photochemistry in macroscopic **batch** reaction vessels is hampered due to inherent limitations: **superficial light penetration** and **poor heat exchange** result in inhomogeneous irradiation and hence to side-reactions or product degradation due to **overexposure**. The recent implementation 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



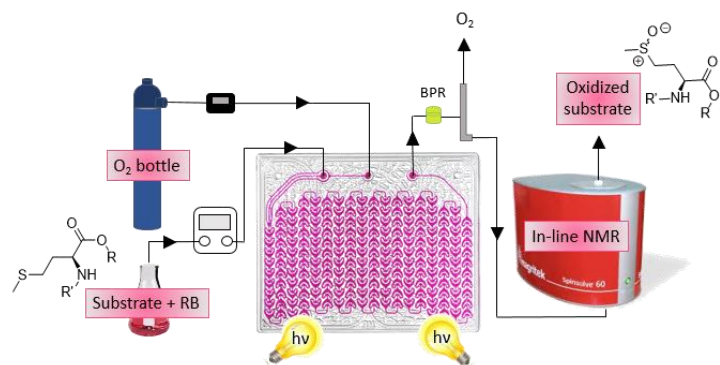
The aim was to **implement the photooxidation of (L)-methionine with singlet oxygen in microreactor** under continuous-flow conditions to avoid the formation of undesired side-product and improve the efficiency of the reaction.

3 | Home-made microreactor



Total conversion :
Batch 2 h
Flow ~1 min

4 | Corning® Advanced-Flow™ Lab Photo Reactor

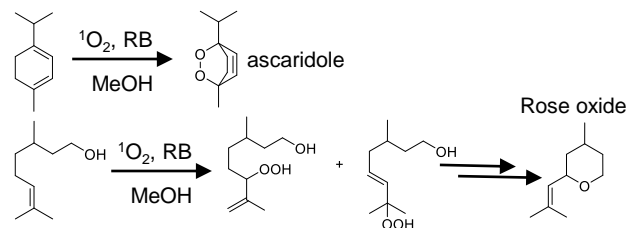


¹ N. Emmanuel et al., *Org. Process Res. Dev.*, **2017**, 21 (9), pp 1435–1438

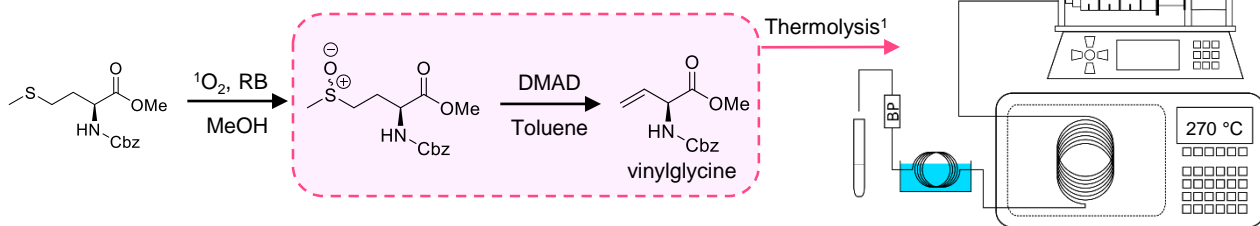
- ✓ Control of exposure time (residence time)
- ✓ Efficient and easy **scale-up**
- ✓ Segmented flow : excellent gas and liquid **mixing**
- ✓ **In-line analysis**

Total conversion in ~1 min (132 g.day⁻¹)

Other substrates:



5 | Thermolysis



¹ N. Lamborelle, J. F. Simon, A. Luxen, J.-C. M. Monbaliu, *Org. Biomol. Chem.*, **2015**, 13, 11602

6 | Conclusion

The **scalable photooxidation of (L)-methionine with singlet oxygen** 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 **~1 min**. Cbz-methionine methyl ester was also photooxidized and successfully thermolyzed into vinylglycine.

7 | Acknowledgements

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