

SCALABILITY OF HETEROGENEOUS MW-HEATED PROCESSES

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Objective

Our research investigates how microwave (MW) heating can contribute to process intensification. The key issue is transformation of lab procedures to a MW oven in a fume hood setup (figures 1 and 2).

Microwave-assisted Heterogeneous Reactions

High concentrations or heterogeneous conditions may improve the space-time yields or productivity. Our setup (figure 3) offers apart from insights into the MW effect also an understanding of mass-, heat transfer and energy efficiency under MW heating conditions.

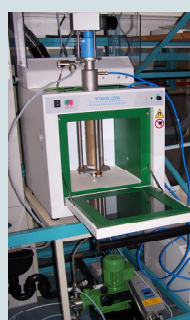


Figure 1:

Microsynth oven equipped with continuous-flow reactor from Milestone srl (Italy).

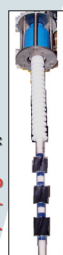


Figure 2:

Stirrer with Weflon blades to absorb microwaves and to indirectly heat reaction mixtures.

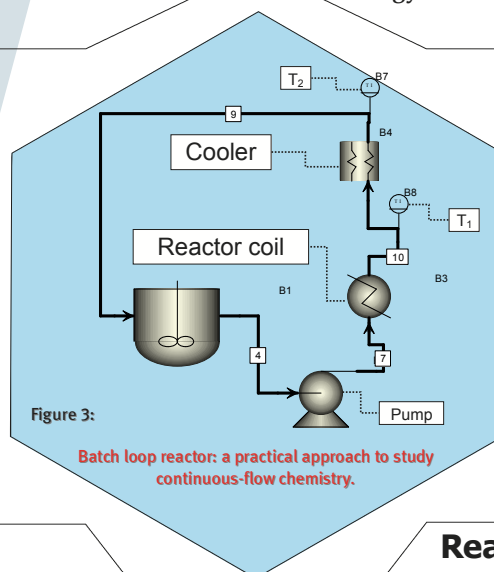


Figure 3:

Batch loop reactor: a practical approach to study continuous-flow chemistry.

Table 1:

Performance of batch loop reactor compared to calculated reaction time based on batch reactions (microwave heating (MW) and conventional heating (CH)).

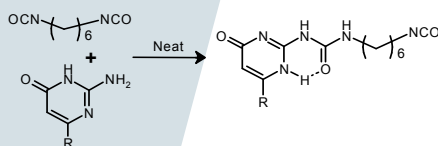
Racemization*	Conversion	Reaction time
Batch loop reactor	73 ee%	263 min.**
Calculated (MW)	73 ee%	628 min.
Calculated (CH)	73 ee%	932 min.

* See scheme 2 and text below

** Performance is higher as expected

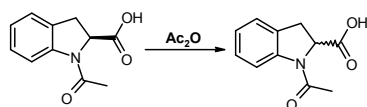
Vanishing MW Effects: Role of Heterogeneity

Previously we reported rate-enhancing MW effects in particular cases compared to conventional heating [1]. The examples in schemes 1 and 2 demonstrate that these MW effects may vanish by decreasing the heterogeneity of the system. The basic mechanism of rate-enhancement –crucial for scaling out these heterogeneous chemical processes– is being studied.



Scheme 1:

Urea formation.



Scheme 2:

Racemization of 1-acetylidoline-2-carboxylic acid.

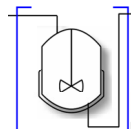
Reactor Performance

Given the limited penetration depth of microwaves, scaling out preferentially relies on continuous-flow equipment that allows to combine all demanding factors for a robust process.

The conversion-time profile in the batch loop reactor can be estimated from kinetic data of the batch process on a small scale (table 1). The batch loop reactor behaves as a n-number of CSTR's (Continuous Stirred Tank Reactor). This number is based on residence time distributions, which are correlated with the behavior of the reactor coil (see figure 4).

Figure 4:

Batch loop reactor simulated by n-number of CSTR's.



Increasing the productivity of the batch loop reactor is achieved by parallel circuits.

In Summary...

- Combination of flow chemistry and microwave heating offers a unique opportunity to scale out heterogeneous microwave-assisted organic reactions.
- The feasibility of this setup is depending on the presence of a microwave effect, safety issues and energy efficiency.

