Intensification of an exothermic sulfur insertion reaction in the continuous Scalable Agitated Baffle Reactor (SABRe) system

Continuous flow chemistry opens a way to intensify exothermic processes and produce materials of high quality at a fraction of the costs and plant footprint.

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21 Jun 2022, By Nikolay Cherkasov

Why exothermic processes are particularly popular in flow chemistry?

Flow reactors such as microreactors are shown to provide exceptional heat and mass transfer. Therefore, the processes that benefit from such properties are the primary application focus. Microreactors, however, suffer from one major problem – their superior performance is tied to the small dimensions rendering the **scale-up of microreactors exceptionally challenging**.

An insurmountable problem is observed when the viscosity of the medium increases and requires a higher pressure to pump the materials. If some solid formation is possible, most flow reactors become unusable with a major risk of irreparable damage if precipitation occurs.

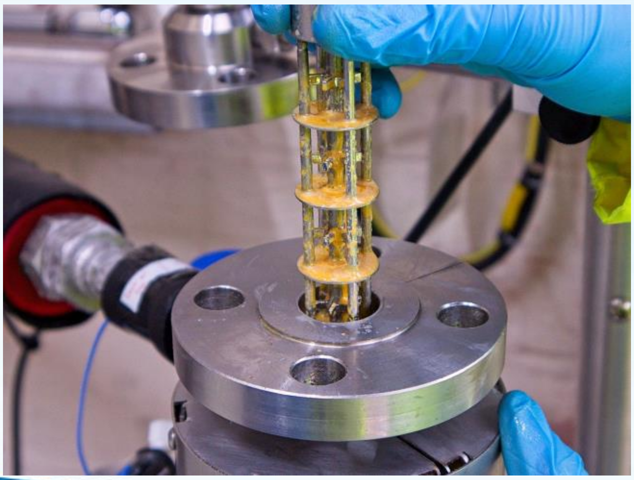
The multi-CSTR SABRe system avoids such problems and **provides rapid heat & mass transfer performance with the ability to handle solids**. In the worst-case scenario of the reactor blockage, the reactor could be opened and cleaned in minutes.

The process studied

We have studied an exothermic (~200 kJ mol^{-1}) sulfur insertion reaction between an aqueous phase and a solvent-free sulfur compound.

A 25wt% aqueous solution	+	Solvent-free sulfur compound (liquid)	⟶	Product (oil phase) Aqueous solution Sulfur (solid) by-product 0.25 kW heat @ 0.1 L/min flow

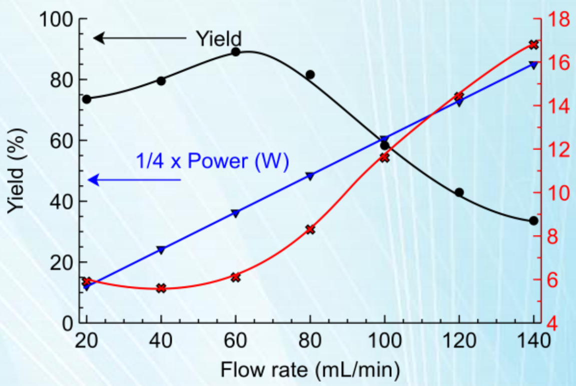
Performing such a process efficiently requires rapid **heat transfer to maintain the product below +10 °C**, otherwise the product decomposes reducing yield and forming by-products. Between the aqueous and sulphur phases, **fast mixing is essential** to form the desired product. Slow mixing increases solid sulfur formation and creates local hotspots. A small amount of sulfur formed even under the optimal process conditions render conventional plate reactors too risky due to the possibility of their blockage and permanent damage.



Experimental procedure

We have performed the study at the customer's premises using our Hastelloy 0.1L reactor system. The substantial amount of HCl formed as one of the products meant that the SABRe in Hastelloy was required

The aqueous phase was introduced into the reactor with a Knauer 80P pump with a maximum flow rate of 1 L min^{-1}; the sulfur phase was introduced with the Knauer P4.1S pump with the ceramic head to handle corrosive medium. The reactor jacket was cooled with a Huber chiller with the temperature monitored at inlet/outlets of the jacket and process fluids.

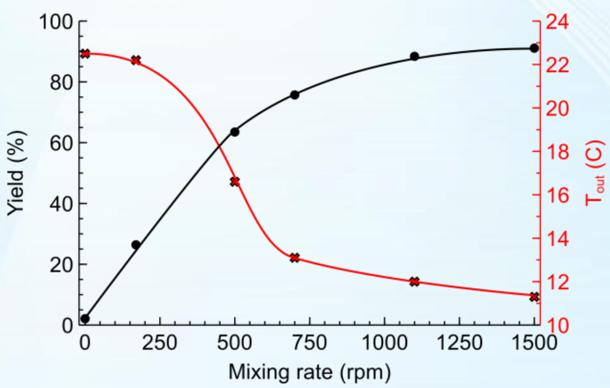


Process development and intensification results

At the first step of the process development, we have maintained the reagent stoichiometry and increased the total flow rate. In this study, a moderate mixing rate of 500 rpm, 0°C jacket fluid temperature, and an intermediate cooling fluid flow rate were fixed.

With the increasing total flow rate, the thermal power of the chemical reaction increased reaching 320 W at the highest flow rate studied. Such a high thermal power resulted in a substantial increase in the process stream outlet temperature of 17 °C, higher than the typical threshold of 10 °C used for the batch process. At the lower process flow rates, the reaction heat was smaller resulting in a lower stream outlet temperature.

The product yield increased with the higher flow rate due to faster mixing. The moderate agitator speed of 500 rpm was selected intentionally to study mixing rate separately. Importantly, we observed yield above **90%** (maximum for the process) with the product fulfilling the specifications.



The next step included a study of the mixing rate on the process performance. In this study, the jacket fluid flow was maintained and the total fluid velocity fixed at 100 mL min^{-1}.

This plot shows the SABRe's ability to **maximise mass & heat transfer performance by changing the stirring rate** – something impossible to do in conventional flow reactors.

The product yield was negligible and outlet process temperature above 22 °C when no stirring was performed. The increasing stirring rate dramatically improved heat transfer performance as evidenced by the decreasing process outlet temperature. Rapid heat removal and mixing of the materials resulted in an increasing process yield reaching the plateau at the maximum value of 90% above 1200 rpm.

The process under the optimised conditions processed above **150 kg a day** of the reactant stream and was proven to operate efficiently for long periods on stream.

The preliminary results show that replacing batch with continuous manufacturing provides an opportunity to halve the manufacturing costs and the energy consumption with exceptional process control and product quality.

The SABRe system enables rapid heat & mass transfer to handle exothermic processes with the throughput of above 100 kg a day.

The SABRe system (available in steel, Hastelloy or glass) is suitable for a wide range of chemical applications. Combining simplicity with superb reaction control, SABRe is the best choice for simple, safe and cost effective chemistry.

What can the SABRe do for you today? Get in touch and arrange a trial.