AM Technology
Engineering Chemistry

Hydrogenation: Technical Note

Hydrogenation: Introduction

Catalytic hydrogenation remains a difficult process to manage using conventional plant equipment, largely due to the safety concerns and costs associated with processing hydrogen at high pressures using large reaction vessels. Many safety concerns can be lessened or negated by switching to flow hydrogenation using a limited volume flow reactor, but this naturally brings about its own challenges. Hydrogenation in flow has been achieved using the Coflore® ATR continuous flow reactor offering significant advantages over other process technologies.

Why use a flow process ? Safety management

Hydrogenations are usually regarded as being hazardous and difficult to manage at production scales due to the dangers inherent in handling hydrogen at large volumes and pressures, especially in the presence of pyrophoric catalysts and/or flammable solvents. In a typical batch process using an induction reactor, hydrogen is reacted in vessels of several thousand litres in size, pressurised to up to 100 Bar, and reacted over many hours in order to achieve sufficient yields. The inventory of solvent, which in many cases is flammable, and catalyst, which can be pyrophoric, must be large enough to account for the size of reactor vessel. Clearly, this requires a multitude of costly safety management technologies and procedures, which can drive capital and operating costs to high levels.

In flow, the process throughput is determined by the flow rate needed to achieve a given residence time rather than the size of the overall reactor. The size of any given flow reactor is greatly reduced in comparison to an equivalent throughput batch reactor, greatly reducing the hazards and risks associated with any given chemical process.

For flow hydrogenation, the active volume of hazardous materials is minimised. Also, the reactants spend less time in contact with each other, allowing for each reactant to be stored safely, and in isolation, for the majority of the operation time.

This greatly reduces safety risks and a well-designed flow reactor can greatly lower costs associated with the relevant equipment set-up and procedures. This, on top of the usual benefits offered by intensified processes, makes flow processing an attractive solution to the typical challenges faced in production-scale hydrogenation.

The Coflore® ATR continuous flow reactor

Hydrogenation using the Coflore® ATR

Many flow reactors currently rely on small tubes, static mixers, or complex mechanical elements to achieve adequate levels of agitation and mixing. While certain hydrogenations in flow reactors are possible at small scales, all are found to be unsuitable for continuous production at high throughputs and residence times, typically due to the problems associated with processing a particulate solid catalyst in the form of slurry.

The ATR can be set-up as a fully automated system that has been specifically engineered to increase durability and simplicity, whilst adding an extra layer of safety.

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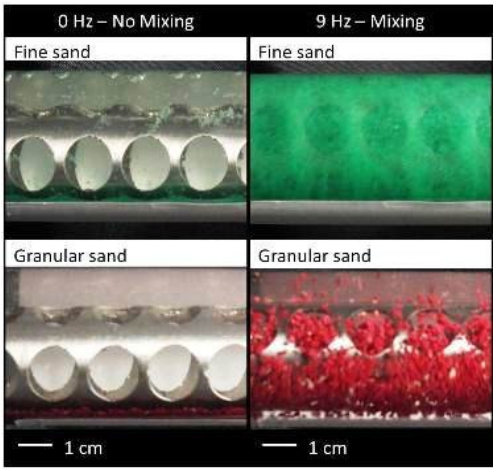
In small tubes or systems with static mixer elements, the solids tend to settle and block the reactor components. If the process fluid is pumped through at a high enough speed to prevent this, the achievable residence time range becomes severely limited. Countering the residence time issue by numbering-up reactors in series leads to significant energy expenditure due to the high pressure loss incurred in the small channels.

The ATR's dynamically mixed system circumvents these problems by decoupling plug flow from net flow. The tubes used are of a relatively large diameter, and allow for long residence times by using internal agitators. These agitators are free-floating within the tube, and efficient mixing is achieved using external oscillatory motion of the entire reactor. The need for mechanical seals and conventional shaft-driven agitators is entirely removed. Dynamic agitation ensures that solids are kept suspended within the process slurry, and that gases are thoroughly dispersed within the liquid phase, making the ATR system ideally suited to handling multi-phase mixtures like those found in hydrogenations.



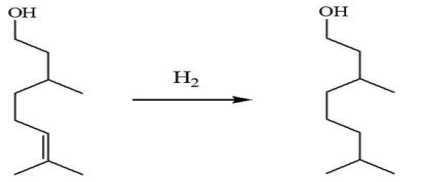
Static vs dynamic mixing in the Coflore® ATR

Hydrogenation in the Coflore® ATR is achieved using a novel counter-current slurry-to-gas flow method; a core function of the Coflore® ATR in hydrogenations as maintaining a uniform gas-liquid distribution is an essential attribute in this process.

Gas presence throughout the system is extremely important in providing efficient performance and the Coflore® ATR achieves this by flowing gas from the bottom-up. At the same time, the slurry (solid-liquid) is distributed uniformly to promote maximum preformation. The Coflore® ATR handles both of these with ease due to the main reaction zone being in a horizontal configuration.

The gas and slurry are in contact within the reactor for a short time relative to current methods, reducing the contact time of flammable or explosive reagents. This reduces safety management requirements and costs, and creates critical advantages for the ATR system over and above those already described. Separation of the catalyst solid from the solvent/product mixture can be achieved quickly and easily without a filter at the reactor outlet, as can final product isolation. Overall, this isolates each component for much more of the processing time than with conventional batch methods.

Hydrogenation of Citronellol to 3,7-Dimethyl octanol using the Coflore® ATR



The hydrogenation of Citronellol to 3,7-Dimethyl octanol was performed according to the following reaction conditions:

10% reactant solution in IPA solvent by mass; 1% Pd/C catalyst content by mass; 40°C; 3 ATM; 15 minute residence time.

These conditions were sufficient to achieve 100% conversion. An 8-tube 10 L reactor assembly gives a pure dimethyl octanol production rate of 57 L/day under 24 hour operation.