Dynamic reactor technology:
Safe & efficient multi-phase chemistry

Dr Kevin van Eeten of **Flowid** looks at the implementation of dynamic flow reactors for multi-phase chemistry

Small skid SpinPro

Understanding the advantages and possibilities of continuous manufacturing at an early stage is essential to implementing flow in existing batch processes and during the development of new batch to flow conversions. R&D, product development, and scale-up to pilot and production levels are all still mostly done in batch, while there is a general unfamiliarity with flow technology during R&D and pilot-scale development

The transition from batch to flow is often advantageous whenever limitations can be overcome that are restraining the production capacity. Successful examples are found in accelerating gas-liquid mass transfer, the efficient cooling of strong exothermic effects, and improving the effects of mixing and selectivity. The very small reactive volumes used in continuous manufacturing bring advantages regarding compliance with safety and environmental regulations.

There is no one-size-fits-all flow solution for all of the problems in the chemical industry. Flowid is an expert in system integration in the field, selecting the best available technologies for the manufacturing of pilot and production flow skids. Thus it can design, manufacture and supply the best flow skid.

Flow systems can be divided into two categories: static and dynamic flow technologies. Each has its own benefits. Static flow reactors often have excellent heat transfer capabilities, give highly reproducible flow patterns and are relatively straightforward to implement and operate given the right flow controls. Dynamic flow reactors increase shear and turbulent mixing, and allow for precipitation reactions.

Flowid's dynamic flow reactor, SpinPro, is a type of rotor-stator spinning disc reactor that is already used in multiple continuous manufacturing skids. These reactors comprise a range of readily scalable dynamic flow reactors, covering the entire range from the initial screening of chemistry in the lab, via pilot-scale optimisation studies to full-scale production units.

For many applications from research to production phase, there is a suitable dynamic reactor. Another advantage is that scale-up is straightforward. Over the past decade, dynamic reactors have transitioned from promising technology for process intensification to becoming a reliable and proven continuous manufacturing system.

A rotor-stator spinning disc reactor (Figure 1) consists of a number of rotating discs (rotors), each inside a narrowly fitting encasing (stator). The disc is about the size of a DVD, rotating with a speed up to 4,000 rpm, while the typical rotor-stator distance is one millimetre. The very high rotational velocities lead to highly intense mixing, directly improving the mass transfer rate.

The large difference in velocity over the very narrow gap between the rotor and the stator leads to extremely fine dispersions of droplets and bubbles with a very high interfacial area, further enhancing mass transfer.[1] In comparison with other static flow reactors, the mixing intensity and level of turbulence do not depend on the flow rate, allowing an independent variation of both residence time and mass or heat transfer effects.

Successful applications of dynamic reactor technologies are found in highly exothermic reactions, such as nitrations and organolithium reactions.[2,3] The latter can even be performed under the formation of precipitates, as the rotor-stator spinning disc reactor is a dynamic type of flow reactor which does not suffer the drawback of clogging that is more common in static flow reactors.[4]

Aside from being able to safely perform highly exothermic reactions, the dynamic reactor technology also offers major benefits for another type of reaction in chemical processing: multi-phase chemical reactions. These reactions are ubiquitous steps in many synthesis routes in the speciality chemicals and pharmaceutical industries.

In a multi-phase reaction, one or more molecules from different physical phases (i.e. gaseous, organic, aqueous and/or solid) need to come together to react. First, a reagent molecule in a gaseous state has to be transported from the bulk of the gas, through the gas-liquid interface towards the bulk of the liquid.

From there, the molecules of interest then have to diffuse further through one or several film layers surrounding droplets, bubbles or catalyst particles, depending on the type of chemistry. Only then, when all molecules have come into close contact and at the right place, can the actual reaction take place.

Each step in this sequence takes a certain amount of time and effort. The total production rate of the chemical reaction not only depends on the intrinsic kinetics, but it must be described as a sort of resistance in series.

Although the intrinsic reaction rate between the molecules can be very high when (or if!) they are in contact, the total production rate often suffers from the time and effort required to get all the phases well dispersed and contacted. In a sense, overcoming the limitations related to mixing in this cascade of steps, brings total production rates much closer to the intrinsically fast reaction rates.

When comparing mass transfer rates between dynamic and batch reactors, it is evident that batch vessels suffer from drastically lower mass transfer rates, as gas-liquid mass transfer rates in rotor-stator spinning disc reactors are at least 40 times higher than in conventional reactors.[5,6] To reach an acceptable level of productivity,

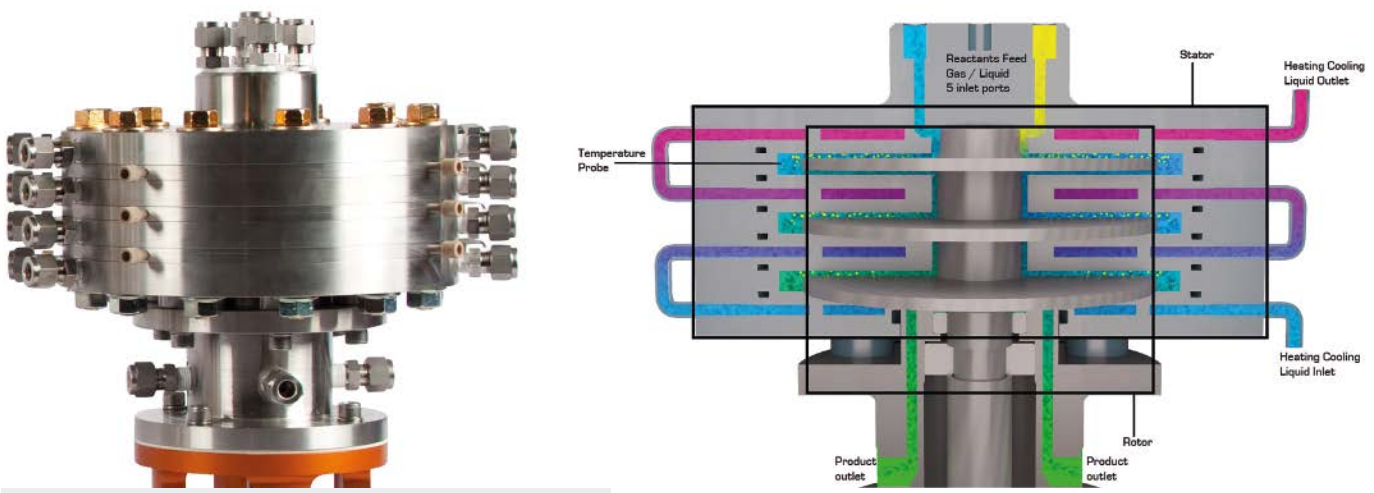

Figure 1 - SpinPro R300 *(left)* & schematic cross-sectional view *(right)*

Table 1 - Industrial cases with major advantages from going to flow

Phases		Pressure	Temperature	Rotation speed	Residence time	Production rate*	Advantages of going to flow
Gas	Liquid(s)	[barg]	[°C]	[rpm]	[min]	[ton/yr]	
HCl	Org.	7	30	2,500	0.50	130	More than 80% reduction of energy cost and CO_2 emissions while doubling production capacity. Large reduction in hold-up of corrosive gas.
CO_2	Org.	5	75	500	0.50	240	Large increase in production rate. The old process was cryogenic, while this was no longer necessary in the SpinPro, leading to a large cost reduction for energy.
H_2S	Org. & Aq.	6.5	50	2,500	20	6	Elimination of toxic gas hold-up and thus an intrinsically safe process. This allowed room to expand production capacity without problems with safety regulations.

* Based on 8,000 hr/yr of continuous manufacturing

these batch reactors are then made needlessly large, and are often fed with a large stoichiometric excess of gas at elevated pressures.

This not only leads to an inefficient use of equipment and resources, but also gives rise to dangerously high gas hold-up in the reactor as well. This especially proves problematic for production with ever more stringent safety regulations or even restricts scale-up ambitions when dealing with toxic or highly reactive gasses.

Flowid has engineered and manufactured several pilot and production skids with multiple dynamic flow reactors per skid in series or parallel for gas-liquid reactions. These reactions are all either highly exothermic and/or impose safety or energy concerns in conventional production.

For some clients, this meant that they would otherwise have been unable to increase production capacity to meet increased market demands by scaling up conventional batch processes, as this would lead to safety end regulatory problems.

Table 1 shows three industrial cases of multi-phase processing in which a production skid from Flowid has been installed on an industrial scale to solve these problems. In the first, where gaseous HCl was used in the production of an aroma molecule, the advantages were:

- Major energy savings and reduction in CO_2 emissions
- Higher yield and product of higher purity
- Lower capex investment due to intrinsic safety

After the transition from batch to a continuous gas-liquid production, the production process is significantly safer and more economic, and resulted in a more than 80% reduction in energy requirements and CO_2 emissions. The conventional batch process was performed in a 10 m³ stirred, glass-lined batch reactor, entailing a nine-day batch cycle with cooling at 0°C.

These long cycle times and the large amount of reactive volumes not only pose a significant safety risk, they also require a lot of energy to keep the reactor at the required low temperature. In fact, the energy consumption for this process amounted to 130 MWh/year, with CO_2 emissions reaching 75 tonnes.

Using our compact dynamic flow reactor, Flowid has designed a production skid of about 1 m³ with a reactive hold-up of only 300 mL *(pictured)*. In stark contrast to the batch process, the continuous system has resulted in an intrinsically safe, more accurately controllable and highly efficient process.

Due to continuous production at ambient temperature production capacity has effectively doubled, while circumventing the need for expensive cooling measures. This has led to substantial reductions in energy consumption (from 130 to 22 MWh/year) and CO_2 emissions (from 75 to 12.5 tonnes).

Conclusion

By combining the advantages of dynamic technology with flow chemistry, the dynamic flow reactor increases mass transfer rates and thus intensifies multiphase chemical reactions. Recent applications in multi-phase processing have allowed customers to meet increased demand while still complying with safety and environmental regulations.

In addition, their processes can now be performed inherently safely, while simultaneously running more economically. With its proven reliability and demonstrated advantages, the SpinPro reactor is becoming increasingly prevalent in production sites worldwide, underscoring its position as a valuable tool in the chemical industry's transition to continuous manufacturing. ●

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