Summary of continuous flow synthesis of zeolite FAU in an oscillatory baffled reactor

Arkema, a French headquartered multinational specialty chemicals and advanced materials company, has for the first time achieved the continuous flow synthesis of zeolite NaX. The successful pilot operation (up to 100 litre/hour) was performed using a NiTech© continuous oscillatory baffled reactor (COBR).

The company wanted to investigate the synthesis of zeolite NaX faujasite (FAU) type in a COBR under different operating conditions. It also wanted to study the effect of mixing intensity on crystal properties along with the steady state of zeolite concentration in both temporal and spatial domains.

Zeolites are crystalline aluminosilicates, which are conventionally synthesized by hydrothermal treatments where a reactive gel composed of sources of aluminium and silica is aged at low temperature. Then, a crystallization step takes place at high temperature yielding a crystalline solid. This process is usually carried out in batch reactors, taking several hours to several days.

Continuous flow synthesis has been proposed over the past years as an alternative solution, offering advantages such as enhanced mass/heat transfer, controllable flow patterns, operational flexibility and ease of scale-up.

Why choose NiTech's© COBR technology?

- The slow crystallization kinetics (= relatively long reaction time) can be performed in a reactor of a length that is practical for production scale;
- The reactor's performance is not hindered by solid particles in the process fluid, unlike some other flow reactors that can be prone to blockages from solids;
- Superior process fluid mixing prevents problematic solid-liquid separation;
- Enhanced heat transfer accelerates the heating and cooling rates, reducing the total process time;
- A controllable, consistent flow regime avoids the variations in product quality that are common in batch systems;
- Reliable, linear scale-up makes process scale-up simple and predictable.

The reactive crystallization of zeolite NaX was carried out in a horizontally oriented NiTech© DN43 (43mm diameter) COBR, which consisted of 10 jacketed baffled straight tubes and 10 jacketed baffled bends.

The raw materials used to prepare the zeolite NaX were powder alumina, sodium silicate, and sodium hydroxide as sources of aluminium, silica and alkali respectively.

Crystallization

Solutions of sodium aluminate (tank 1) and sodium silicate (tank 2) as well as the seeds (tank 3) were pumped through an in-line mixer and then into the COBR. The reaction temperature was set to 100^{o}C. During the process, samples were taken at three points along the reactor at regular times.

The COBR's consistent mixing environment was found to be suitable for handling the zeolite crystals – avoiding sedimentation and preventing blockages. Both the crystallinity of the zeolite at different points and the system's pressure log demonstrated that a stable continuous synthesis was achieved. The effective mixing was also beneficial in suppressing fouling.

Positive results

The COBR's enhanced heat transfer and plug flow behaviours resulted in a controlled temperature profile along the reactor during operations. This ensured a constant and repeatable environment, essential to obtain a good crystallinity and avoid the co-crystallization of other zeolite phases. After five hours of operation, the reactor walls were free of encrustations.

Results showed that the combination of a seed-assisted method, in-line mixing of the reactants at high temperature and effective heat transfer along the reactor allowed a complete synthesis in less than 100 minutes.

In addition, the crystalline zeolite yields produced were consistently close to the theoretical yield of 98-100%.

The trials also demonstrated that a high flow rate resulted in less dispersion of the synthesis gel and faster stabilization of the process. **"A continuous system for zeolite synthesis integrating a COBR is attractive from a productivity point of view", said the authors[1]. "The validation of the system in a pilot-scale (so far up to 50 litres/hour) provides the potential for reliable scaling up to industrial-sized production."**

This summary is based on work published in the following journal article: Ramirez Mendoza H, Valdez Lancinha Pereira M, Van Gerven T, Lutz C. Continuous flow synthesis of zeolite FAU in an oscillatory baffled reactor. Journal of Advanced Manufacturing and Processing. 2020;2:e10038.