Speeding up time to market...

For biobased products using fast and efficient high throughput experimentation

Introduction

A series of recent surveys conducted by the nova-Institute (Hürth, Germany) concluded that green premium prices can be realized for novel biobased products within the coming years [1]. A short time to market is crucial in securing a significant share of the growing market for biobased products. It is clear that biobased products cannot rely solely on a societal *green bonus* but that they need to convince at a technical and economic level, as well as based on a comprehensive life cycle assessment. There are two factors that distinguish frontrunners in developing new products and technologies: a clear R&D strategy; and access to advanced R&D techniques either in-house or by bringing in external expertise. A strategy develops a clear understanding of the winning value proposition for the customer in the marketplace and follows a systematic approach to efficiently drive R&D work. It combines close steering of the development process and a simultaneous techno-economic analysis (see Figure 1 on the different development stages).

The R&D capabilities need to comprise efficient R&D tools and workflows applied by a dedicated staff with a high level of expertise. Over the last 20 years, high throughput experimentation (HTE) has become established as the most advanced tool for fast, efficient, and industrially relevant R&D. The key feature of HTE is a high degree of parallelization and automation combined with an efficient workflow to remove bottlenecks in daily R&D work. A fully integrated software workflow collects all data generated within the R&D cycle in one comprehensive database and eliminates manual documentation and data reporting. A closed software loop ensures fast handling and evaluation of the large amount of data generated within a very short time when using HTE. It significantly accelerates the proof of concept (PoC) and development phase (TRL 3–5, Figure 1) so that the demonstration phase can be reached more quickly. In hte's experience, the development of novel processes can be accelerated by up to one hundred times when applying HTE technology as compared to classical R&D (a few at a time), depending on the development stage and the actual application.

HTE R&D in action: From biomass to *green* nylon 66 via 1,6-hexanediol

In the following, HTE is demonstrated in action with a case study from the bioplastics area, namely producing Nylon 66 from a sustainable route via hydroxymethylfurfural (HMF) and 1,6-hexanediol (16HD) to hexamethylenediamine (HMDA) [4].

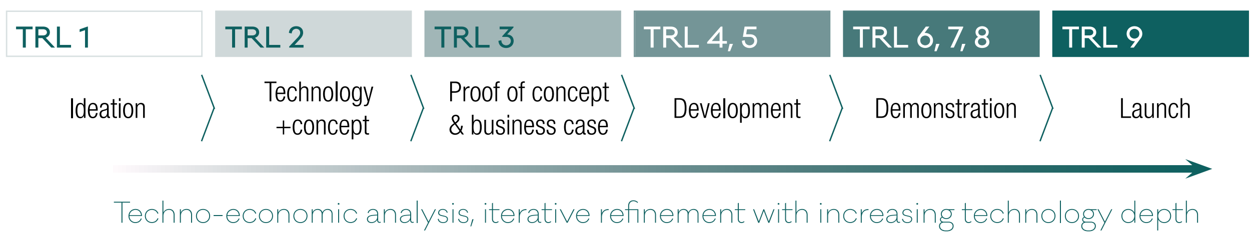
Lignocellulosic biomass from plant residues and agricultural waste is an ideal renewable and non-food feedstock basis [5]. The critical step in this process route is the production of 16HD from C6 polyols (PO) (see Figure 2). It requires a catalytic step involving selective hydrodeoxygenation and utilizing a heterogeneous catalyst in a continuous trickle-bed reactor system [4]. The economic feasibility of the process depends on identifying the optimum catalytic formulation with respect to activity, stability, and cost, and ideal operating conditions for maximum 16HD yield and selectivity. At the same time, the applicability of a broad range of viable feedstocks in the process is also important from an economic standpoint, considering large-scale commercial production.

Figure 3 shows the development cycle for producing 16HD from C6 polyols. The development work starts with the synthesis and screening of many different catalyst systems. The initial PoC screening and reaction network investigation is typically done with simple HTE batch reactor systems (milligram catalyst scale, in powder form). In a second step of initial up-scaling, the process is transferred from batch systems to continuous reactor systems utilizing HTE trickle-bed test units (gram catalyst scale, in powder form, with 16 reactor tubes in parallel, for instance) in order to closely represent actual continuous operation conditions.

The first two steps comprise several hundred catalysts to be tested with different feedstocks and under a variety of operating conditions in order to assess catalyst activity, selectivity, and stability, and to elucidate the reaction network.

It is important to define first performance targets based on a first detailed techno-economic analysis (Figure 1).

Figure 1: Market-driven product and technology development stages based on technical readiness level (TRL) classification [2] and the stage-gate model [3], as well as illustrating the importance of a simultaneous techno-economic analysis (TEA).



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In the next step and after identifying several lead catalyst systems, the catalysts are upscaled to industrial size and shape (as pellets, extrudates, or spheres) and further optimized by additional testing (typically several tens of catalysts). Note that this step can still be performed in HTE equipment in the range of a few grams.

The HTE test protocols have been optimized over the years to deliver relevant results to support demonstration and industrial scale. It is important not to postpone this step for too long within the development stage because the apparent kinetics of commercial catalyst sizes and shapes will deviate from the powder kinetics and will affect product yield and selectivity.

In the last development stage, the catalyst amount is typically increased into the hundred-gram scale using bench-scale test systems. These systems have a lower degree of parallelization but a high degree of flexibility for each catalyst system. In this stage, many different real feedstocks are tested to widen the feedstock portfolio. The catalyst systems are operated individually in their optimum operation range and over a longer period with industrially relevant feedstocks to assess long-term catalyst stability.

Larger product quantities are collected to perform more detailed product analytics. Reactor design and material are finally evaluated, and necessary process steps around the catalytic reactor such as gas and/or liquid recycling are investigated. The results obtained form the basis for further refinement of the techno-economic evaluation and act as input for the layout of the successive pilot demonstration phase (see Figure 1).

In the given case study, advanced HTE technology and workflows could be utilized to establish a comprehensive technology platform for converting non-food biomass into valuable biobased building blocks such as 1,6-Hexanediol. By means of high throughput experimentation, the PoC and development phase (TRL 3–5) could be shortened to around 3 years covering a broad range of feedstocks, identifying and optimizing several lead catalysts to commercial size and establishing a platform technology for renewable chemical intermediates. At the same time, a comprehensive set of information could be generated for a detailed techno-economic evaluation.

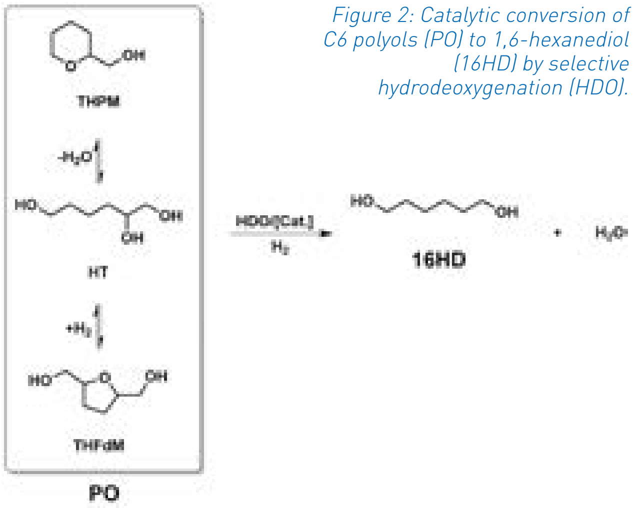

Figure 2: Catalytic conversion of C6 polyols (PO) to 1,6-hexanediol (16HD) by selective hydrodeoxygenation (HDO).

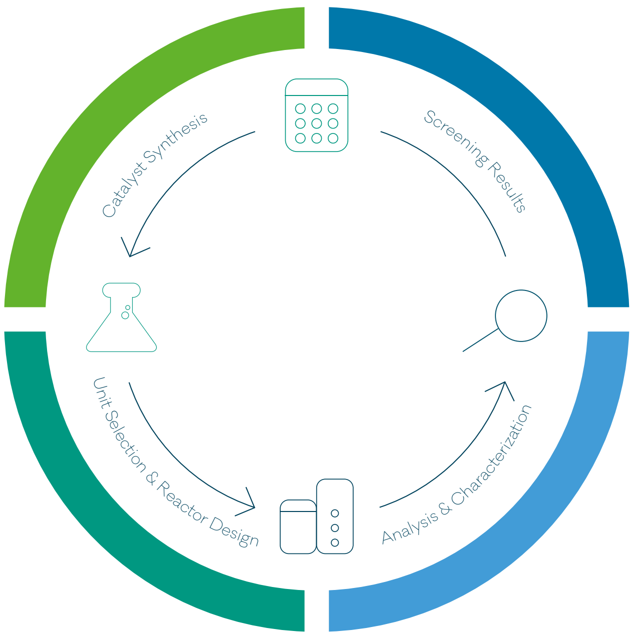

Figure 3: Development cycle for producing 16HD from C6 polyols. The catalyst scale-up in this example involves shell impregnation of commercial carriers. The product analysis involves detailed GC-MS and GC-FID/TCD analysis identifying more than a hundred compounds within the reaction network. The testing comprises HTE as well as flexible bench-scale equipment.

References

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