DEVELOPMENT OF CHEMICAL PROCESSES

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Summary

The task of the process development and of the chemical engineering respectively is, to extrapolate a chemical reaction discovered and researched in the laboratory to an industrial scale, taking into consideration the economic, safety, and ecological boundary conditions.

The starting point is the laboratory equipment and the outcome of development is the production plant; in between, process development is required. The development of chemical processes is a complex procedure; therefore it does not take place in a one-way street. Assumptions are made for the individual development stages which are only confirmed or refuted when the next stage is being worked on. It may be necessary, therefore, to go through the individual stages several times with modified assumptions, resulting in a cyclic pattern. The first hurdle in establishing a new process is overcome when a promising synthetic route, usually with associated catalysts, is discovered. The conventional process development is carried out in three stages:

- Optimized laboratory synthesis
- Laboratory plant (the so called miniplant
- Pilot plant.

The scale-up factor from one stage to the next is always limited by the „minimum principle“, i.e., the process stage or piece of equipment with the lowest scale-up feasibility determines the maximum capacity of the next larger plant whose operating performance can be calculated. It is here that the engineer has an opportunity to save time and money. Consequently, efforts are now made to extrapolate directly from the miniplant to the production scale. An important tool to do this is the so-called integrated-miniplant-technology. This means the synergism between:

- Process pre-selection via microplants and process evaluation via short cut methods.
- Experiments via a continuously operating miniplant which includes all the recycling paths.
- Mathematical simulation of the experiments.

So, safety in scaling up is as good as that obtained by setting up a pilot plant.

1. Introduction

Chemical engineering is the science that deals with the development of chemical processes from a small-scale laboratory reaction vessel to a large-scale production process under economic, safety, ecological, and juristic boundary conditions. The chemical engineer has to develop and to improve the quality of the corresponding technical tools.

The bases of chemical engineering are:

- Mathematics
- Natural science such as physics, chemistry and biology
- Engineering science, especially mechanical and electrical engineering
- Business administration, especially cost accounting.

The basis was founded by important people such as:

- *Sadi Carnot*, French physicist (Thermodynamics of combustions in steam engines)

- *Rudolf Clausius*, German physicist (Thermodynamics of chemical systems at molecular scale)
- *Josiah Willard Gibbs*, American scientist (Developed much of the theoretical foundation that led to the development of chemical thermodynamics)
- *Hermann von Helmholtz*, German scientist (Thermodynamics of electro-chemical processes)

Henry E. Armstrong offered the first course in Chemical Engineering at the Imperial College in London in *1885*. *George E. Davis* produced the first Handbook of Chemical Engineering in *1901*.

The difference between laboratory and industrial chemistry can be highlighted by the following simple example:

The polymerization of acrylic acid takes place in a round bottom flask (5 cm diameter) and a production vessel (spherical, 2 m diameter).The heat of polymerization is 77.4 kJ mol^{-1}.The geometric parameters and heat produced for the reaction are shown in Figure 1.

The example shows that the heat liberated for the reaction on the laboratory scale can easily be removed by water cooling, whereas in the case of the production scale, the heat liberated will cause the vessel to explode.

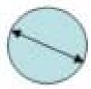
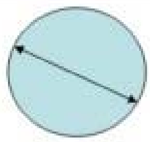
	Lab apparatus	**Production plant**
		
Diameter d	5 cm	2 m
Volume V	65.5 cm^3	4.19 m^3
Surface A	8.5 cm^2	12.6 m^2
Specific surface area a	120 m^2 m^{-3}	3 m^2 m^{-3}
Mass of acrylic acid m	68.0 g	4.36 t
Liberated heat Q	0.020 kWh	1 300 kWh
Specific heat charges Q_{spec}	2.55 kWh m^{-2}	103.5 kWh m^{-2}

Figure 1. Geometric and heat production conditions for the both cases: Lab scale and production scale. The heat flux density for water cooling is typically in the order of magnitude of 10 kW m^{-2}.

If we look at the production structure of the chemical industry from a bird eye view, it is seen that there are only a few hundred major basic products and intermediates which are produced on a scale of at least a few thousand to several million tones per annum worldwide. This so called building-blocks or intermediates, which are in turn produced from only about ten raw materials (especially crude oil), are the stable foundation on which the many braches of refining chemistry (dyes, pharmaceuticals, etc.), with their many thousands of often only short-lived consumer products, are based. This has resulted in the well-known "*ChemisTree*" (Figure 2).

A special characteristic of the basic products and intermediates is their longevity. They are statistically so well protected by their large numbers of secondary products and their wide range of possible uses that they are hardly affected by the continuous changes in the range of products on scale. Unlike many end products, which are replaced by better ones in the course of time, they do not themselves have a life cycle. However, the processes for producing them are subject to change. This is initiated by new technical possibilities and advances opened up by research, but are also dictated by the current raw material situation.

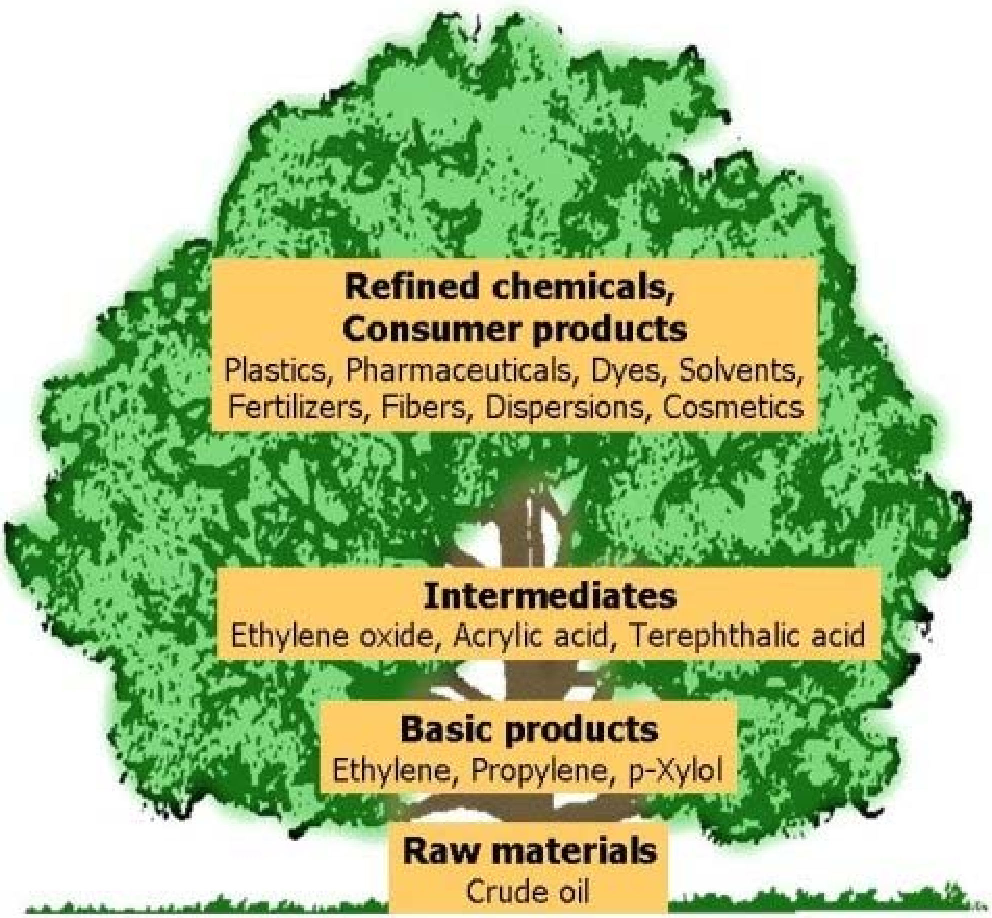


Figure 2. Example for a so called *ChemisTree*: starting from the raw material crude oil and progressing through the basic products and intermediates, to the refined chemicals and final consumer products, as well as specialty chemicals and materials.

In the longer term, an oil shortage can be expected in 40 to 50 years, and this will result in increased use of natural gas or biomass. The fossil raw material with the longest future is coal, with reserves for more than 200 years. The question whether natural gas reserves in the form of methane hydrate, in which more carbon is stored than in other fossil raw materials, will be recoverable in the future cannot be answered at present, since these lie in geographically unfavorable areas.

In the case of basic products and intermediates it is not the individual chemical product but the production process or technology which has a life cycle (Figure 3): To remain competitive at this point the producer must be the price leader for his process. Therefore, strategic factors for success are:

- Efficient process technology
- Exploiting economy of scale by means of world-scale plants
- Employing a flexible integrated system at the production site
- Professional logistics for large product streams.

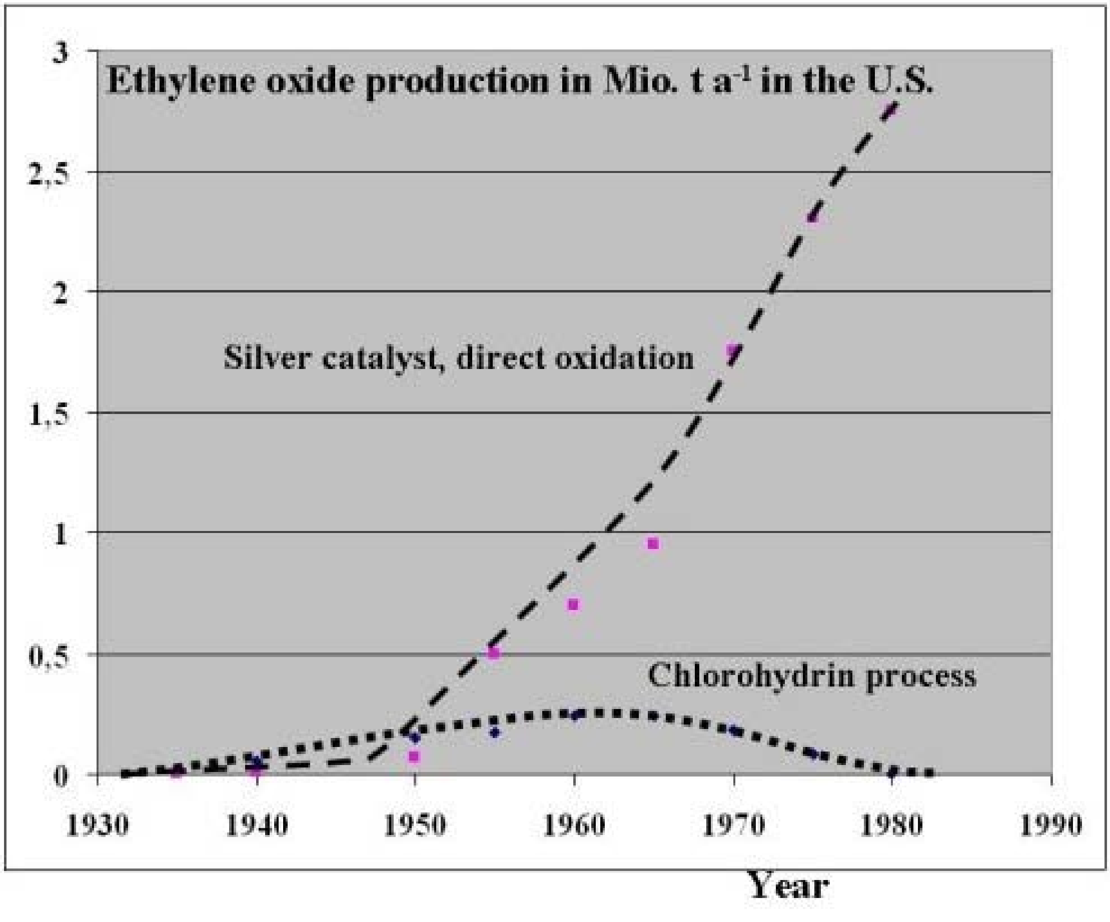


Figure 3. Life cycles of the Ethylene oxide process. In 1999 the ethylene oxide production in the U.S. was over 4 Mio. t a^{-1} [Weissermel 2003].

The demands made on process development for fine chemicals differ considerably from those of basic products and intermediates. In addition to the boundary conditions of better and/or cheaper, time to market (production of the product at the right time for a limited period) and focused R&D effort are of importance here. Further strategic factors for success are:

- Development partnerships with important customers
- The potential to develop complex multi step organic syntheses
- A broad technology portfolio for the decisive synthetic methods
- Certified pilot and production plants
- Reputation as a competent and reliable supplier.

Special chemicals are complex mixtures whose value lies in the synergistic action of their ingredients. Here the application technology is decisive for market success. The manufacturer can no longer produce all ingredients, which can lead to a certain state of dependence. Strategic factors for successful manufacturers are:

- Good market knowledge of customer's requirements.
- A portfolio containing numerous magic ingredients
- Good technical understanding of the customers systems
- Technological breadth and flexibility.

Active substances such as pharmaceuticals and agrochemicals can only be economically marketed while they are under patent protection, before suppliers of generic products enter the market. Therefore, producers of such products cannot simply concentrate on costly research. As soon as possible after clinical trials and marketing approval, worldwide scales of the product must begin so that the remaining patent time can be used for gaining customers. In contrast, the actual chemical production of the active substance is of only background importance. The precursors can be farmed out to other companies. Strategic factors for success of active substance manufacturers are:

- Research into bimolecular causes of disease and search for targets for pharmacological activities
- Efficient development of active substances (high-throughput screening, searching for and optimizing basic structures, clinical development)
- Patent protection
- High-performance market organization.

Enterprises which already have competitive advantages must take account of the technology –S-curve in their research and development strategy (Figure 4).

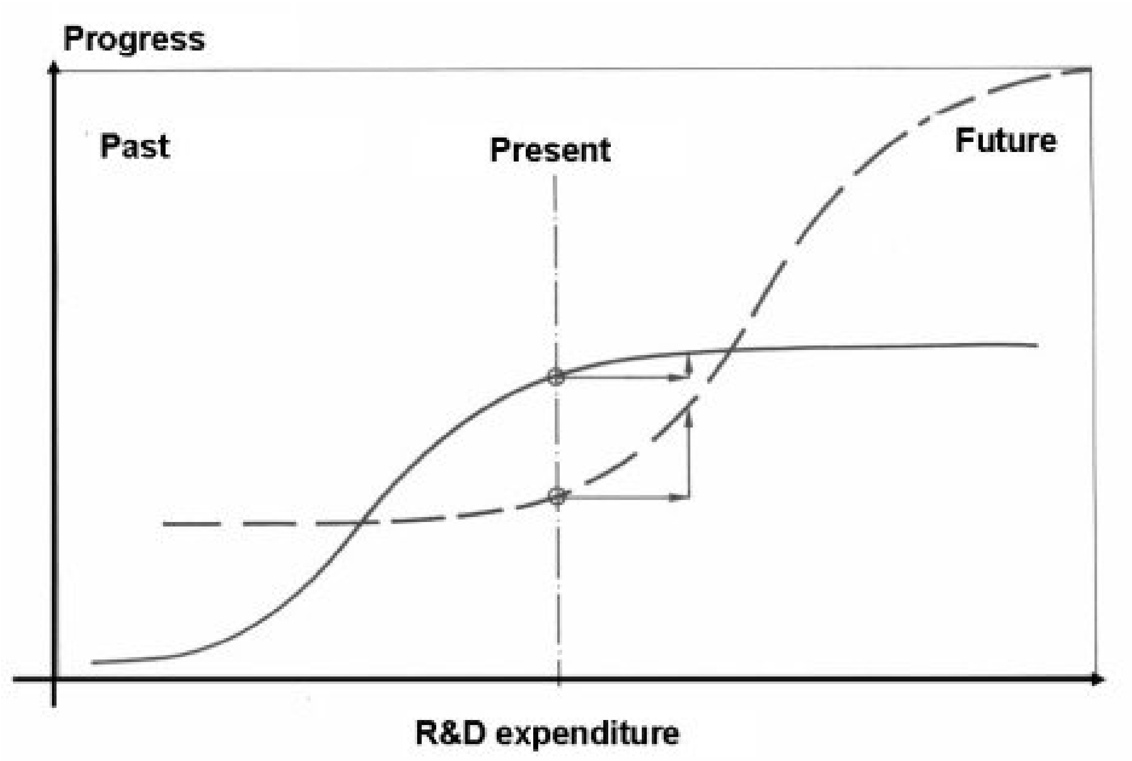


Figure 4. Technology-S-curve: Productivity of R&D expenditure increases considerably on switching from basic technology (____) to a new trend setting technology (-----).

The curve shows that as the research and development expenditure on a given technology increases, the productivity of this expenditure decreases with time. If enterprises are approaching the limits of a given technology, they must accept disproportionately high research and development expenditure, with the result that the contribution made by these efforts to the research objectives of cheaper and /or better becomes increasingly small, thereby always giving the competitor the opportunity of catching up on the technical advantage. On the other hand, it is difficult for a newcomer to penetrate an established market. But, as Korean or Chinese companies have shown in the past, it is not impossible.

Once an enterprise has reached the upper region of the product or technology-S-curve, the question arises whether it is necessary to switch from the standard technology to a new pace-setting technology in order to gain a new and sufficient competitive advantage. Figure 3 depicts this switch to a new technology schematically and shows that on switching from a basic technology to a new pace-setting technology, the productivity of the research and development sector increases appreciably, and substantial competitive advantages can thus be achieved.

The potential of old technologies for the development of cheaper and/or better are only small, whereas new technology have major potential for achieving competitive advantages. It is precisely on this innovative activity that the prosperity of highly developed countries with limited raw material sources such as the European union and

Japan is based, since research represents an investment in the future with calculable risks, whereas capital investments in the present are based on existing technology.

To assess whether a research and development strategy of better and/or cheaper is still acceptable in the long term for a given product or production process, the R&D management must develop an early warning system that determines the optimum time for switching to a new product or a new technology. Here it is decisive to have as much up-to-date information on competitors as possible. This information can be obtained not only from the patent literature but also from external lectures, conferences, company publications, and publicly accessible documents submitted to the authorities by competitors. Since industrial research is very expensive, instruments for controlling the research budget are required, for example:

- A cost/benefit analysis for the particular product area, whereby the benefit is determined by the corresponding user company sector.
- A portfolio analysis to answer the questions:
  - Where are we now?
  - Where do we want to be in 12 years?
  - What do we have to do to now to get there?
- An ABC analysis for controlling the R&D resources, based on the rule of thumb that
  - 20 % of all products account for 80 % of turnover, or
  - 20 % of all new developments account for 80 % of development costs.

It is therefore important to recognize which 20 % these are in order to set the appropriate priorities (A = important, profitable, high chance of success; B = low profitability; C = less important tasks with low profitability).
The ways in which chemical companies organize their research varies and depend on the product portfolio. Mostly it involves a mixture of the two extremes: pure centralized research on the one hand, and decentralized research on the other.

The task of process development is to extrapolate a chemical reaction discovered and researched in the laboratory to an industrial scale, taking into consideration the economic, safety, ecological, and juristic boundary conditions. The starting point is the laboratory apparatus, and the outcome of development is the production plant; in between, process development is required [Dittmeyer 2003, Storhas 2003, Ullmann 2002, Vogel 2002, 2005].

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Biographical Sketch

Herbert Vogel was born in *1951* near Frankfurt/Main, Germany, and served an apprenticeship at Röhm&Haas before going on to study chemical engineering at Darmstadt Polytechnic and chemistry at Technische Hochschule Darmstadt, Germany, where he received his PhD under Alarich Weiss in physical chemistry. Between *1982* and *1993* he was employed at BASF Aktiengesellschaft in Ludwigshafen, Germany, working on the development, planning, construction and installation of petrochemical production plants. In *1993*, he succeeded Fritz Fetting as professor for Technical Chemistry at the Technische Universität Darmstadt. His research interests are heterogeneous catalysis, chemistry in supercritical fluids and renewable primary products.