doi:10.2533/chimia.2012.248 Chimia 66 (2012) 248–255

Conference Report

10. Freiburger Symposium 2011 der SCG-Division Industrielle Chemie Technology Progress, Success Key for our Production Sites

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Abstract: This short paper presents the abstracts of the different presentations during 10. Freiburger Symposium 2011 der SCG-Division Industrielle Chemie: Technology Progress, Success key for our production sites held Thursday and Friday, September 29 and 30, 2011 at the Ecole d'ingénieurs et d'architectes de Fribourg (Switzerland).

Keywords: Chemistry · Industry · Production · Technology progress

1. Switzerland – a Leading Country for Innovation – The Contribution of the Swiss Innovation Promotion Agency CTI

Dr. ***Martin Riediker***, President NRP 66

The CTI is the Swiss government's innovation promotion agency. It has been fostering the transfer of knowledge and technology between universities and businesses for over sixty years, bringing together partners in applied research and development projects and supporting the creation of start-ups. Switzerland has one of the world's leading levels of innovation. The CTI has a budget of around CHF 120 million and applies the maxim of 'Science to Market'. Businesses develop new knowledge in collaboration with the universities which can then be commercialized for the market in the form of products and services. CTI plays a unique role in the innovation landscape of Switzerland. All tools available within CTI will be discussed in detail and supported by examples relevant to the audience.

Project Promotion

CTI's objective is to generate more innovative products and services by encouraging higher education institutions and companies to work together on joint R&D projects. Each year, CTI supports several hundred of these joint R&D projects. Companies – especially small- and medium-sized enterprises – are thus able to leverage the R&D resources of higher education institutions to develop their innovative ideas into marketable products and services. Only those who can quickly bring brilliant ideas to market will succeed in global competition. Companies benefit two-fold: from the project results and the growing expertise of young R&D workers trained to conduct research that meets the needs of the market. Support is generally available for R&D projects relating to scientific innovations in all disciplines.

Start-up Promotion and Entrepreneurship

Many innovative ideas are brought to life and put on the market by young entrepreneurs and start-ups. Switzerland urgently needs people with exciting business ideas, who also have the drive to take on the competition and turn their vision into reality. That is why the CTI launched its CTI Start-up initiative in 1996 to promote Swiss science-based start-up companies with high growth potential. The CTI's start-up promotion scheme offers a wide range of training and coaching activities. These activities are modular in structure and enable young entrepreneurs to get the specific support they need. In the framework of CTI Entrepreneurship CTI offers in addition the consulting and training program 'venturelab'. The program is targeted for potential and existing young entrepreneurs and sensitizes since 2004 students primarily from the technological sectors.

Networks

CTI offers Swiss companies swift and easy access to knowledge available at universities and public research organizations in Switzerland as well as direct links to international promotion programs for applied research. CTI is fully aware that Swiss companies act on a global scale and research is in no way limited to national boundaries.

2. Setup Time / Change Over Time Reduction by SMED

Dr. ***Erwin W. Studer***, Profact

Einleitung

Die Auslöser für Anstrengungen zur Reduktion der Rüstzeiten können unterschiedlich begründet sein (Fig. 1). Die controller hat immer die Bestände im Auge, dies aber bei hoher Flexibilität und kurzen Lieferzeiten! Der beste Fall liegt vor, wenn es sich um eine Engpassanlage handelt und dabei die mehr hergestellten Teile oder Einheiten auch am Markt abgesetzt werden können.

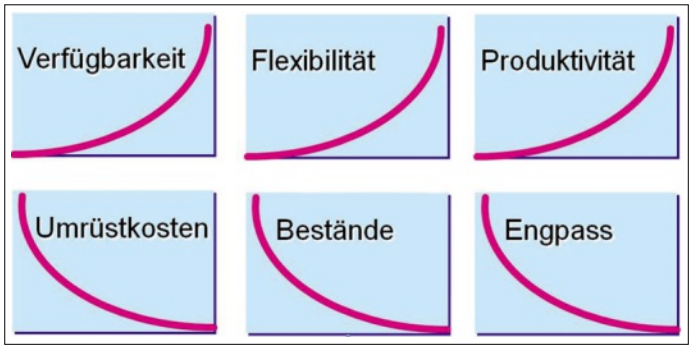


Fig. 1. Warum Rüstzeiten senken?

Das Verfahren 'Single Minute Exchange of Die'

SMED wortwörtlich übersetzt heisst Format- oder Produktwechsel im einstelligen Minutenbereich.

Dies ist eine Methode/Verfahren, wodurch die Rüstzeit einer Produktionsmaschine oder einer Fertigungslinie reduziert wird, wobei die 9 Min. 59 Sekunden in der Prozessindustrie höchst selten erreicht wird! Dennoch hat sich der Begriff SMED zur Methode in der Fertigungsindustrie durchgesetzt.

Definition Rüstzeit (Fig. 2): Zeit vom letzten Gutteil des alten Fertigungsloses bis zum ersten Gutteil des neuen Fertigungsloses. Wobei Gutteil je nach Prozess zu spezifizieren ist. Ebenso ist in der Praxis das Ende der Rüstzeit schwierig zu bezeichnen. D.h. Ab welchem Zeitpunkt läuft die Anlage wieder stabil mit dem Folgeauftrag?

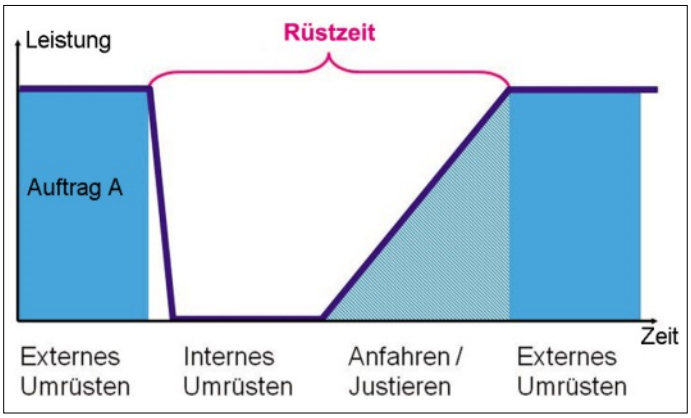


Fig. 2. Leistung in Funktion der Zeit mit der Rüstzeit.

Entwickelt wurde das Verfahren ursprünglich innerhalb des Toyota Produktionssystems (TPS). Die Umsetzung erfolgt in mehreren iterativen Schritten, wobei die Rüstzeit erst durch organisatorische und später durch technische Massnahmen reduziert wird.

Zur Minimierung der Kosten ist es wichtig, dass die Schritte in der vorgegebenen Reihenfolge durchlaufen werden. Die Erfahrung ist, dass jeder Schritt jeweils zu einer Reduzierung der Rüstzeit von 50 bis 60% (in Bezug auf den vorherigen Schritt) führt. Grössere Investitionen werden dadurch, wenn überhaupt, erst zu einem sehr späten Zeitpunkt getätigt.

SMED läuft in vier (fünf) Schritten ab:

• Trennung von internen und externen Rüstvorgängen (Organisation)

• Überführung von internen in externe Rüstvorgänge

• Optimierung und Standardisierung von internen und externen Rüstvorgängen

• Beseitigung von Justierungsvorgängen

• Optimierung der externen Rüsttätigkeiten (Aufwandminimierung)

Wesentliches Element bei der Umsetzung ist es, Rüstvorgänge in interne und mögliche externe Schritte zu unterteilen und die externen Vorgänge zu durchlaufen, solange die Maschine noch produziert oder wieder produziert.

• Interne Schritte können nur bei Stillstand der Maschine durchgeführt werden (z.B. Werkzeugwechsel).

• Externe Schritte können durchgeführt werden, wenn die Maschine noch produziert (z.B. Bereitstellung der Werkzeuge und Vorprodukte).

Zur Optimierung werden verschiedene Techniken angewendet, z.B.:

• Vorbereitung des Produktionswechsels

• Schiebetische statt Kräne

• separates Vorheizen

• Zwischenspannvorrichtungen zur Justierung ausserhalb der Maschine

• Parallelisierung von Rüstvorgängen – gleichzeitig statt nacheinander – d.h. statt einem, mehrere Mitarbeiter einsetzen (wird oft auch als ein fünfter Schritt betrachtet)

• Eliminierung/Reduzierung von Justierungen

• Standardisierung von Rüstaktivitäten (Checklisten!!)

• Standardisierung von Formatsätzen oder Werkzeugabmessungen

• Doppelte Formatsätze

3. Operational Excellence @ BASF Kaisten AG

Dr. ***Wolfgang Baechle***, BASF Kaisten

BASF Kaisten AG is a manufacturer of plastic & lubricant additives (antioxidants) producing mainly for the European and Middle East market. The Kaisten site is part of the BASF business unit 'Plastic Additives' and its global production network.

Over the last ten years pressure from external Asian companies as well as from BASF internal competitors has increased due to gradually commoditization of the product range. In order to protect competitiveness, a new production strategy has been developed and implemented striving for 'Operational Excellence'. Main pillars of this strategy are:

Safety First

Over the past years a lot of effort was expended in the chemical industry to further improve the EHS situation in the sites. Especially in the area of process and occupational safety, a lot of new tools, systems and regulations were elaborated and implemented. Tremendous progress could be achieved, but further improvement of the current level of incidents and accidents needs a different approach. BASF's long term objective strives for further reduction of 'Lost time incidents' (LTI) of about 70% compared to status quo 2010.

It is consensus that we do not need to further boost our regulation system, but we have to implement a cultural change related to safe behavior going through all levels of the hierarchy. The latest initiatives within BASF address the topic 'behavioral safety'. The global 'Safety Champion' program is a good example and shows first positive results.

Fully Automated Production Processes and fully Integrated Material Flow for Standard Products

The first automated production line in Kaisten was commissioned in 1972, the year of the site opening. Already at that time it was recognized that automation is a key element in operating a chemical plant most efficiently. Automation became an important part of Kaisten's philosophy how to develop projects and operate processes. An important milestone was the start up of the new production building for an important intermediate. The process is reliably operating 7 d / 24 h without having a shift organization in place. Beyond the automation of pure production lines, the material flow between tankfarm, production lines, production buildings and even the final product warehouse was streamlined, interlinked and fully automated. The result is a fully integrated production and warehouse set up with no manual interference during regular production.

Differentiation by Offering Customer-specific Products / Systems (make to order principle)

After the expiry of important product patents, a new strategy for managing the business was necessary. In the Antioxidant business one important approach is the provision of customer-specific products and solutions. Up to five different products (BASF-owned and third-party components) are blended according to the recipe of the customer. Finally the product is converted into a nice, granular, easy-to-handle product form. Innovative manufacturing technology, high operational flexibility and short lead times as well as efficient and costly processes are the key success factors in order to successfully differentiate from competitors.

Adoption of the 'Lean Manufacturing' Philosophy as the Basic Culture for Continuous Improvement

Lean Manufacturing is derived from the famous Toyota production system. Between 2006 and 2009 a Lean Manufacturing

project was rolled out over the whole former company (Ciba) in order to become world class and cost leader in operations. An important part is the introduction of lean tools like MIFA, Wrench Time, OEE, SMED, Lean Blitz *etc*. These tools help you in analyzing the current status of a work process and to evaluate alternatives and optimizations.

The whole Lean project delivered a quantum leap with respect to operational efficiency. Reduction of production fix costs of more than 20 % could be achieved within four years.

The Lean project itself is only the start on the way to a sustainable lean manufacturing culture.

The application of some of these Lean tools will exemplarily be demonstrated. An example out of quality control will show how you can successfully leave a valid and proven system and to enter new fields of how doing business on a much higher level of effectiveness and efficiency.

Fostering Involvement of all Employees by Implementing 'Total Productive Management' Tools like '5 S' and 'Idea Management'

The most important asset of a company is its workforce, its people having huge professional potential and great ideas. One of the biggest challenges is to make these potentials available and best use of them.

Our attractive Idea Management System 'Inspirato', which significantly involves the employee in the monetary success, is one possibility to actively involve our people in the continuous improvement process.

In parallel to the Lean Manufacturing initiative, we additionally promoted the 5S program. 5S stands for Sort, Straighten, Shine, Standardize, Sustain.

The content of 5S is the 'My Area Concept' giving each employee a physical area for which he is responsible. This means resonsible for its status and condition, but also for the corresponding improvement process ('should be' is defined together with his boss and its colleagues).

Each employee receives a written contract of his 5S duties and responsibilities and the contract (called 'Göttibrief') is signed by him and the Site Manager. This official procedure has a high impact on the awareness.

Periodic audits guarantee long-term sustainability of the whole 5S system.

All related measures and initiatives lead us to innovative solutions and a quantum leap in productivity. In addition, qualification, motivation and satisfaction of most of our employees have significantly increased.

4. Appropriate Handling of Highly Active Substances – Investments in Containment Technologies at Roche's Technical Development Department

Dr. ***Thomas Osswald***, F. Hoffmann-La Roche

In the past, the indication area and the potency of the substance was often related (*e.g.* oncology products) and the number of potent substances was low. Changed research approaches over the past years have led to more specific, highly active drugs requiring facilities with adequate technical installments to avoid personal protective equipment (PPE).

In his talk, Dr. Thomas Osswald will show how the technical development organization (PTDA) at Roche has transformed their existing assets – he will cover the chain from laboratory over miniplant to pilot plant – into assets meeting the requirements for handling highly active substances with a design exposure level (DEL) of the facilities of 1 µg/m^3, the so called 3A level at Roche. In two examples he will briefly explain two facilities for handling 3B compounds. A DEL of 50 ng/m^3 must be met at these facilities during operation.

In the first part, measures to reach 3A requirements in the laboratory and plant area are described. In the first example, a larger laboratory refurbishment project for synthesis and crystallization development is briefly described. Constraints and how they were overcome are being discussed. In the second part, technical solutions to reach a containment level of 1 mg/m^3 in the plant area (scale 40–4000 L) are presented. Focus is primarily on the charging and the discharging process of substances. New developments in the area of flexible glove-boxes and a successful usage of a downflow booth instead of an isolator are described. Focus is also put on the question how the transfer of the wet cake from the centrifuge to the driers can be accomplished in a closed manner.

In the second part, two facilities for handling substances of the class 3 B with a DEL of 50 ng/m^3 are described. First, a miniplant with two laboratories and a small production unit is briefly discussed and layout schemes are shown. In the second example, a pilot plant facility for handling 3B compounds on the 400–630 L scale is described.

The talk concludes with some general statements and points to consider when running such investment projects from various perspectives.

5. Innovation dans les techniques analytiques online et offline : Des alliées précieuses pour une meilleure maîtrise des procédés en chimie industrielle

Gregor Pfundstein, Syngenta

Depuis plusieurs années maintenant, l'industrie chimique occidentale fait face à de plus en plus de challenges et ressent une constante pression dans un monde où nous parlons activement de mondialisation.

Le jeu devient plus complexe, et les défis ne se situent plus uniquement à trouver de nouvelles molécules et de mettre en place un procédé industriel pour les synthétiser à un prix raisonnable. A n'importe quelle étape du cycle de vie d'un procédé nous attachons une importance croissante à améliorer constamment son efficience, à mieux le maîtriser et à le rendre plus robuste.

Ces maîtres mots que sont «efficience, maîtrise et robustesse» nous permettent et nous permettront de rester compétitifs dans un environnement de plus en plus concurrentiel.

La chimie analytique représente un élément clé dans cette quête d'optimisation constante. Sans elle ce serait un tâtonnement permanent et une compréhension souvent incomplète ou erronée des mécanismes parfois très abstraits régissant notre science.

La dernière décennie a été très riche en innovations analytiques tant au niveau de l'équipement que des méthodologies et le but de cette présentation est de mettre en exergue un certain nombre de cas concerts où une implémentation de moyens analytiques online et offline adéquats a permis de relever les défis exposés ci-dessus.

Nous aborderons ensemble dans les grandes lignes les thèmes suivants :

Présentation du groupe d'analytique spéciale et de procédé de Syngenta Crop Protection Monthey

Introduction à qui nous sommes et à notre mandat que ce soit sur le site de Monthey et également en partenariat avec les sites Syngenta autour du globe.

Divers axes d'activité de la chimie analytique au service du processus industriel

Ce point permettra de situer quels secteurs d'activités font actuellement l'objet d'une attention particulière et d'une demande analytique croissante. D'un but simple d'assurer la bonne marche d'un procédé à une analyse pointue de qualité, en passant par les aspects HSE (Hygiène / Sécurité / Environnement), nous en ferons un bref survol afin de comprendre les enjeux.

Rentabilité et amortissement de l'équipement importance de la précision de mesure – mesure primaire et impact statistique sur la variabilité

Chaque personne ayant été confrontée à un exercice de budgétisation a du à un moment ou à un autre donner les arguments persuadant les parties-prenantes du bien-fondé d'un projet. Un argument principal est l'amortissement lors de l'investissement. Ce calcul n'est pas toujours trivial et dépend grandement de la précision et de la variabilité de mesure au final.

Le dilemme; celle-ci n'est pas toujours connue car parfois le recul n'est pas là. Quelles sont les solutions dans un tel cas? Comment une bonne planification des essais peut nous aider à comprendre et prendre en compte ces aspects.

Techniques analytiques les plus fréquemment utilisées au laboratoire et en ligne

La dernière décennie a été très riche en innovation au niveau analytique. La rapidité, la précision des technologies permettent toujours plus de capacités et ouvrent des perspectives jusqu'ici insoupçonnées. Nous allons faire un tour d'horizon, couvrant les techniques de séparation et de chromatographie rapide, techniques spectroscopiques et physico-chimiques. Nous parlerons également d'adaptation d'analyseurs conventionnels du commerce aux conditions de production.

Exemple concrets d'implémentation chez Syngenta

Consolidant les points précédents nous citerons dans cette section un échantillon des principales réalisations de ces derniers 3 ans dans notre entreprise, démontrant ainsi concrètement à quel point une analytique performante peut soutenir la compréhension et la maîtrise des procédés.

Regard sur l'avenir

Dans ce dernier chapitre nous nous pencherons sur:

Quels sont les techniques émergentes, quelles sont les techniques que nous explorons ou allons explorer, quelles sont également les limitations à prendre en compte, quels sont les challenges, les demandes de nous autres industriels pour les prochaines années.

Discussion finale et questions

Mettant un point d'orgue à l'exposé, nous pourrons avoir une approche participative, permettant de poser les questions qui s'imposent, échanger les expériences et avoir un écho général sur la situation dans les divers domaines d'activités et entreprises présentes dans l'auditoire.

En conclusion, le tandem chimie analytique – chimie industrielle n'a jamais été aussi actif et le sera probablement encore bien plus dans le futur afin de satisfaire aux hautes exigences et à une vive compétition. Une bonne compréhension réciproque des divers acteurs est nécessaire pour tisser les synergies positives et être conscient des potentiels et limitations de chaque partie.

6. La pérennité des unités de production de vitamines par l'innovation, exemples pratiques

Dr. ***Olivier Wolfrath***, DSM Nutritional Products

DSM was able to increase its innovation-related sales from 2% in 2006 to 14% in 2010. However, the major part of product portfolio is based on well-established, non-life cycle time restricted products like vitamins and carotenoids. To stay competitive in this business, an ongoing optimization of the production lines is mandatory.

Due to the complex production process there is no single tool for optimization. DSM uses therefore, a large choice of different approaches.

The following approaches have been adopted from 2000 in Sisseln vitamin production and are explained using practical examples:

Process Development

For vitamin E production an optimized synthesis route was designed together with the construction of a new production building. State of the art equipment and process techniques achieved higher chemical yields, better process control and higher product quality.

Six Sigma Methodology and Data Recording

The Six Sigma toolkit is used in all production lines to identify yield variations and losses. This statistical approach increased, for example, the efficiency of various reactions in vitamin A and E using mostly raw production data generally without any lab trials. This is facilitated by the systematic recording of all available production information like flow rates, volumes, temperatures and pH values, for example. Six Sigma benefits from this enormous information volume and helps relate it to current losses.

A first example uses the regular process variations to analyse their effect on the vitamin A chemical yield. It was proven that a batch reaction shows a better yield when working slightly more dilute, decreasing the production costs despite the additional solvent quantities. The concentration set point was redefined and a sustainable improvement was achieved.

Another example is the optimization of a distillation process for acetone recovery. Distillation processes are complex systems and their troubleshooting is a challenge for chemical engineers. However a plant chemist decided to use the Six Sigma approach to improve the recovered acetone quality. By considering the distillation column as a black box and only considering the mass flows in and out, he was able to find relationship with the distillate quality. After implementation the regenerated acetone met 100% of new GMP requirements.

Fixed Costs

The new vitamin E production unit represented a drastic reduction of fixed production costs. The manpower dedicated to the plant was decreased by 70% compared to the old unit while the plant capacity was increased by 200%. Reduced manpower requirements were achieved mostly through highly automated equipment and with online analytics.

Process deviations (quality and safety) are tracked automatically. An automated batch data monitoring system is in place for continuous control. Only in case of a specific deviation are the personnel responsible involved. This is a reliable tool for transparent production and higher GMP compliance.

In general an increase of the automation grade and, even more importantly, making the automation autonomous (called Jidoka in Toyota Production System jargon) is of interest. This concept

delegates the transition operations to the PCS (process control system) and an operator is only required when an error occurs. DSM Sisseln introduces this concept jointly with wireless tablet PCs that any operator can use outside of the control room for more flexibility.

Operational Excellence

Not only the creation of new tools helps to increase production efficiency; all current workflows have to be challenged. They contain waste such as equipment failure, product reworking, overprocessing, waiting times, *etc*. They cost a lot of effort. Even if a chemical production line is not an assembly line, we use many of the Lean Manufacturing tools to eliminate this waste. Value Stream Mapping, Day in Life of, Overall Equipment Efficiency (OEE) are typical examples. DSM Sisseln has introduced a bottom up process, directly involving the operators, who face these issues daily. Kaizen events (small teams which identify waste and eliminate it quickly) are one pillar of the DSM Sisseln Operational Excellence program, which creates a culture of continuous improvement. Only a few weeks after launching the program many waste issues were identified. This waste represents a lot of unnecessary effort. Typical waste issues are: non value adding reporting/documents, some manual work, double maintenance tasks, low automation grade, non suitable packages, equipment failure, *etc*.

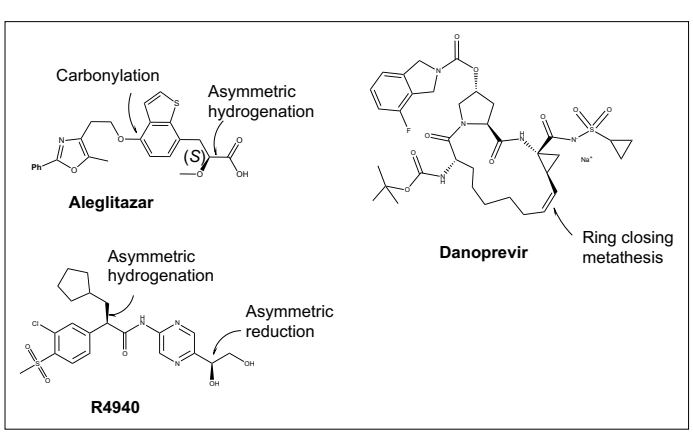
In conclusion, simplification and improvement of vitamin production processes and workflows create value. There is no time to slow down and continuous improvement keeps going.

7. Fortschritte/Trends in Katalyse (Metall und Bio)

Dr. ***M. Scalone***, F. Hoffmann-La Roche

In recent years, a clear tendency towards increasing structural and stereochemical complexity of new drug candidates is observed. This factor, together with the limited amount of time and resources available to invent and develop more efficient syntheses, often leads to a critical issue regarding cost of the active pharmaceutical ingredient (API).

This presentation will illustrate how the early integration of catalysis in the synthesis plan of three new development compounds has not only been the condition to guarantee the timely supply of the amounts required for the planned preclinical and clinical investigations, but has also set the base for scalable and economical synthetic processes. Four reactions will be discussed as an example, three based on metal catalysis (carbonylation, asymmetric hydrogenation, ring-closing metathesis – Scheme 1) and one on enzymatic catalysis (asymmetric keto reduction).



Scheme 1. Metal catalysis in the synthesis of active pharmaceutical ingredients.

8. QbD (Quality by Design)

Dr. ***Thierry Schlama***, Novartis

Quality Risk Analysis – QRA – is both a systematic approach to access the criticality of process parameters and a key document at the interface of Chemical and Analytical Development and Chemical Operations.

Chemical Operations in fulfilling the marked needs has to guarantee a consistently high quality of the drug substance making at the same time economic use of resources.

Chemical Development, besides screening and inventing different routes for a given drug substance, has to overcome the scale up hurdles of a finally chosen route and to establish a robust process, pinpointing the ranges of critical parameters but also justifying the flexibility of operation to allow necessary adjustments in a plant environment.

The quality concept in the QRA refers to (a) the analytical profile of the product which needs to meet the predetermined specifications and (b) the process performance itself. Product and process quality depend on the adjustment of process parameters which are laid out in pilot or manufacturing procedures. A detailed Quality Risk Analysis is based upon a particular manufacturing procedure and follows the sequence of process steps. Development and plant chemist should consider any possible outcome for each individual process step if a parameter is varied ("what happens if ..."). This knowledge is gathered from past laboratory and pilot plant experience and from future experiments which must appreciate the features of a large scale plant scenario (scale down).

The lecture will highlight a number of examples for the influence of parameter levels on process performance and product quality. Furthermore the experimental and management process of gaining this knowledge, assessing the limits, getting a common understanding of process research, chemical operations and quality assurance will be discussed.

Based on the drawbacks still apparent in this approach, the next QbD methodological generation (Fishbone, FMEA, DoE, Design Space) will be discussed.

9. Energiemanagement in der chemischen Produktion

Urs Keller, DSM Nutritional Products

Bei DSM ist das Thema Energien und Energiesparen seit langem ein Thema. Im Jahr 2006 wurde die neue Energiesparinitiative mit der Erwartung gestartet, bis 2010 10% des spezifischen Energieverbrauchs zu senken. Dieses Ziel sollte mit „NoLow Cost"-Massnahmen und -Projekten erreicht werden, die sowohl bei der Erzeugung der Energien wie auch bei den Verbrauchern ansetzten. Das Werk Sisseln braucht grossen Mengen an Dampf als Prozesswärme und Strom als Primärenergie für die Antriebe in den Prozessen und die Bereitstellung der verschiedenen Kühlmedien. Beim Reporting wurden ausschliesslich diese Energien berücksichtigt. Mit 12% konnte das Ziel der Energiesparinitiative erfreulicherweise deutlich übertroffen werden.

Im Referat wird neben der Organisation auch das Reporting mit den Erfahrungen und seinen Grenzen vorgestellt. Es wird auf das Messwesen mit seinen gewachsenen Strukturen und Eigenheiten eingegangen, welches die Grundlage für das Reporting bildet.

Es werden ausgewählte Projekte, die in unterschiedlicher Weise zur Zielerreichung beigetragen haben, vorgestellt. Im Projekt „Online-Bilanzierung" bei der Dampfherstellung wird aufgezeigt, wie mit der Darstellung von Prozesswerten der Wirkungsgrad eines Kessels optimiert werden kann. Beim Projekt „Abbau

von Biofouling in Leitungen und Wärmetauschern“ werden die Methodik und der Erfolg des Systems mit den Unterschieden zwischen Sommer und Winter beleuchtet. Aus dem technischen Bereich wird aufgezeigt, wie die Dampfverluste in einem Produktionsbetrieb unterbunden werden konnten. Ein weiteres technisches Projekt zeigt auf, wie sich der Stromverbrauch in der Kläranlage durch den Ersatz von Belüftungsdüsen erheblich reduzieren liess. Im spezifischen Anwendungsbereich lassen sich mit Einzelprojekten häufig deutliche Energieeinsparungen im zweistelligen Prozentbereich erzielen. Im konsolidierten Werksverbrauch entspricht dies Reduktionen im tiefen einstelligen %-Bereich. Die Energiesparprojekte rechnen sich auch finanziell. Die Energierechnung des DSM-Werks Sisseln konnte in den letzten Jahren um mehrere Hunderttausend Franken entlastet werden.

Das neue Energiesparziel von DSM sieht vor, den spezifischen Energieverbrauch auf der Basis 2008 bis 2020 um weitere 20% zu senken. Dies ist eine neuerliche Herausforderung für unser Werk. Es werden die Grundlagen, die möglichen Potenziale und deren Umsetzungspläne aufgezeigt.

10. Solar2fuel – Photocatalytic Reduction of CO_2

Dr. ***Korinna Dormann***, BASF

Drehte sich die öffentliche Diskussion bisher vor allem um die unterirdische Lagerung von Kohlendioxid, zielt das Projekt „Solar2fuel“ auf die direkte Verwertung von Kohlendioxid. Dabei soll der Kohlenstoff im CO_2 mit Hilfe von Sonnenlicht als Energieträger in klimaneutralen Kraftstoff (z.B. Methanol) für Verbrennungsmotoren oder Brennstoffzellen umgewandelt werden.

Innovativ ist dabei nicht nur der technologische Ansatz – speziell funktionalisierte Halbleiter-Nanoteilchen als Katalysatoren – sondern auch das Ziel, industrielle Abgasströme als mögliche Quelle von Wertprodukten zu nutzen. Die stoffliche Verwertung von CO_2 aus stationären Quellen könnte damit neue Wege in Richtung umweltfreundliche Verkehrstechnologien aufzeigen und zugleich durch Vermeidung von klimaschädlichen CO_2-Emissionen eine Alternative zu den bestehenden Plänen der CO_2-Speicherung sein.

Dazu werden in einem Verbundprojekt [Partner: EnBW Energie Baden-Württemberg AG Karlsruhe; BASF SE Ludwigshafen, Universität Heidelberg, Karlsruher Institut für Technologie (KIT)] Ansätze aus der Nanotechnologie und der Materialforschung mit katalytischen Prozessen kombiniert.

BASF entwickelt neuartige photoaktive Materialien mit starker Absorption im sichtbaren Bereich des Sonnenlichts, die zusätzlich die Fähigkeit besitzen, Halbleitermaterialien zu belegen und effizient zu sensibilisieren. Gleichzeitig werden Halbleitermaterialien entwickelt, die die CO_2-Reduktion katalytisch unterstützen. Die Tauglichkeit der Materialien als zentralen Baustein überprüft sie in Zusammenarbeit mit der LMU München in einem eigens dafür entwickelten Testaufbau hinsichtlich Umsatz und Selektivität.

Die Wissenschaftler der ***Universität Heidelberg*** arbeiten gemeinsam mit der BASF an einer luft- und lichtstabilen Kombination von Farbstoffen und funktionalisierten Halbleiterteilchen in Nano-Grösse. Diese ist Voraussetzung dafür, dass das Sonnenlicht mit Hilfe von organischen Farbstoffen im optimalen Bereich absorbiert werden kann und damit Energie zur Umwandlung von CO_2 liefert. Die physikochemische Untersuchung der kritischen Schritte im Mechanismus der photokatalytischen CO_2-Reduktion sowie die Charakterisierung möglicher Katalysator-Degradationswege sind dabei ein wichtiges Hilfsmittel. Mit der ingenieurtechnischen Realisierung von „Solar2fuel“ beschäftigen sich Wissenschaftler des ***KIT***. Sie untersuchen die physikalisch-chemischen und verfahrenstechnischen Aspekte im Gesamtprozess sowohl in der Simulation als auch experimentell.

EnBW untersucht die Energie-, Emissions- und Kostenbilanzen im Gesamtprozess – vom Kraftwerksabgas über die eigentliche Photokatalyse bis hin zur Nutzung der Produkte. Analysiert werden auch die Kosten für die Bereitstellung von Kohlendioxid aus Kraftwerken und direkt aus der Luft. EnBW will damit ermitteln, unter welchen Bedingungen solche Verfahren wirtschaftlich tragfähig sein können.

Der innovative technische Ansatz ist die Verwendung neuartiger photoaktiver Materialien, bestehend aus funktionalisierten Halbleiter-Nanoteilchen. Gelingt eine geeignete Funktionalisierung, so absorbieren die Materialien im sichtbaren Bereich des Sonnenlichts – die Voraussetzung dafür, dass die Sonne als regenerative Energiequelle „angezapft“ und Sonnenenergie in chemischer Form gespeichert werden könnte. Die angestrebte Technologie zeichnet sich durch den Einsatz von günstigen Materialien aus. Zusammen mit einer angepassten CO_2-Bereitstellung aus Abgasströmen, soll dies dabei helfen, CO_2-Vermeidungskosten zu senken.

Das Verbundvorhaben „Solar2fuel“ gehört zum Spitzencluster „Forum Organic Electronics“ und wird vom BMBF gefördert.

11. Handling of Unstable Reaction Intermediates: Scale-up of Ozonation Reactions

Dr. ***Markus Nobis***, Lonza

Summary

With regard to safe scale-up of the ozonation reactions, it is essential to evaluate the feasibility of the reaction on a technical scale and also to define precisely the set of parameters which is required for the safe handling of the unstable intermediates and their work up. This includes besides the generation of the safety data (*e.g.* calorimetric measurements, DSC) also a risk assessment in an interdisciplinary team, which results in the safe and predictable handling of ozonolysis on a technical scale.

General

Based on its high reactivity, ozone is a well-known oxidation agent for various applications.[1,2] Ozonolysis is in most cases the conversion of unsaturated organic compounds under generation of the partially unstable ozonides and peroxides as reactive primary intermediary products. The ozonides are known as high

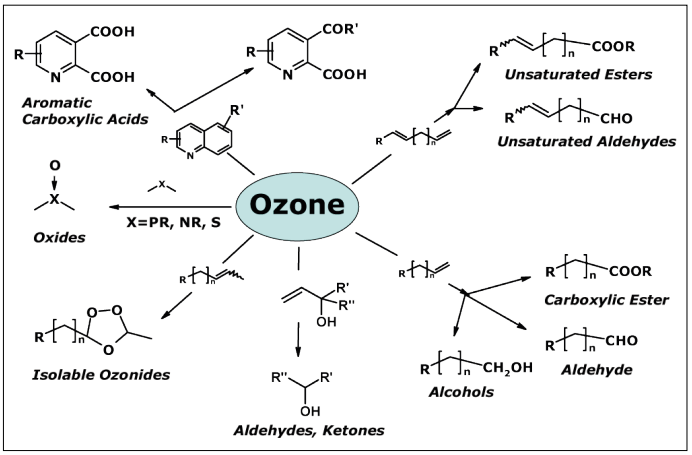


Fig. 3. Reactions with ozone.

energetic, unstable primary reaction products. On the other side, the high reactivity of the intermediates allows the conversion of the materials under very mild conditions to the desired final products[3] (Fig. 3).

In general, the scale-up of chemical processes for their commercial use is strongly influenced by physical and chemical parameters. These parameters (*e.g.* mass transfer and heat transfer) impact directly the robustness of the processes with regard to safety and quality aspects.

Approach for Development and Technical Implementation

The safe implementation of ozonolysis reactions (Fig. 4) at technical scale requires the perfect control of the gas–liquid mass transfer process. Knowledge of the process conditions at laboratory scale is the key to generate most of the criteria for later handling at technical scale. A crucial point which has to be taken into consideration for the fast, safe and successful development and technical implementation is the necessity to mimic the later technical set-up. At Lonza, this is achieved on a small scale by using a gas–liquid loop reactor integrated with a micro venturi injector.

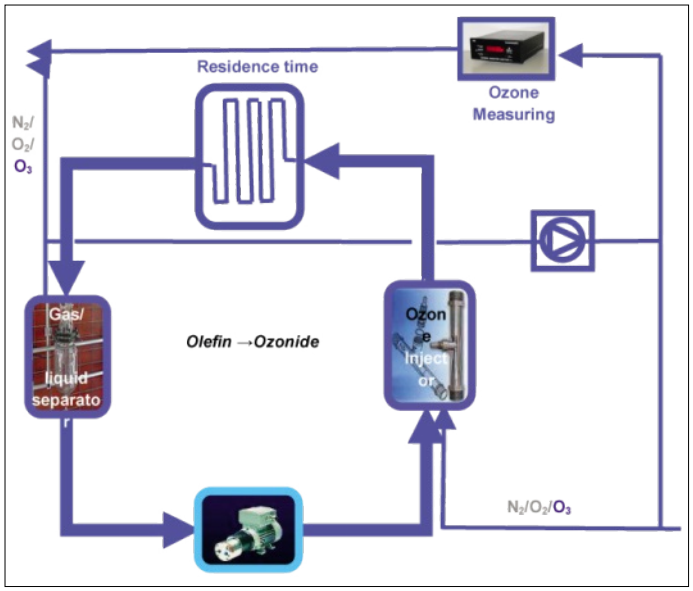


Fig. 4. Safe implementation of ozonolysis reactions.

This approach helps to overcome issues which derive from the physicochemical differences between the 'classical' laboratory setup to the technical plant scale.

An important part of the safe handling of the unstable intermediates in chemical conversions on production scale is the evaluation of the process safety. Lonza has implemented systems on lab scale which enable to generate the required data for the safe, efficient and robust scale up of ozonolysis reactions (*e.g.* calorimetric measurements) and the necessary criteria and parameters which are required for the development of reliable procedures and protocols.

This risk assessment is carried out based on the set of information which derived from the laboratory investigations regarding the ozonolysis, together with the generated physicochemical data for both the reaction and the generated intermediary products (RC-1 data/DSC data). The risk assessment as a core part for the design of a safe process is carried out in an interdisciplinary team, which defines the measurements for the handling of the process on the technical scale.

This novel approach combines laboratory feasibility studies and technical transfer – including hazard evaluations and safety assessments for the technical implementation.

Conclusion

The handling of the unstable intermediates on production scale can be carried out safely. A key requirement for this is the efficient generation of the necessary set of information about the process to guarantee the safe and predictable scale-up of the chemical process. Regarding the ozonolysis, Lonza has implemented systems and designs which enables to approach process development and data generation for safety assessments in the same time.

[1] A. F. Hollemann, E. Wiberg, 101. Aufl., 'Lehrbuch der anorganischen Chemie', de Gruyter, Berlin, New York, **1995**, 514ff.
[2] 'CRC handbook of Chemistry and Physics', 87 ed. CRC press, Boca Raton, **2006**, 8–23.
[3] a) P. S. Bailey, 'Ozonation in Organic Chemistry', Academic Press, New York, **1978**; b) J. A. Souza, A. L. Bluhm, *J. Org. Chem.* **1960**, *25*, 108; c) D. G. M. Diaper, D. L. Mitchell, *Can. J. Chem.* **1960**, *38*, 1976; d) P. S. Bailey, A. G. Lane, *J. Am. Chem. Soc.* **1967**, *89*, 4473.

12. Industrielle Kontrollierte Radikalpolymerisation

Dr. ***Peter Nesvadba***, BASF, Sandmeyer-Preisträger 2011

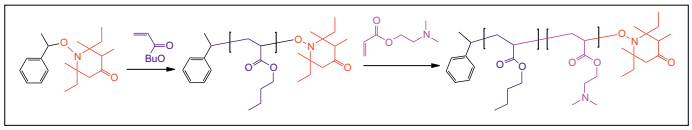
Radical polymerization (RP) is one of the most important industrial polymerization techniques. However, prior to the mid-90s, classical RP did not allow the synthesis of polymers with precisely designed molecular architectures. This situation changed dramatically with the invention of controlled radical polymerization (CRP), one of most important developments in polymer chemistry of the last two decades. CRP allows the design of block-, comb-, and star-copolymers as well as polymers with defined terminal functional groups or with predetermined and narrow molecular weight distributions.

The most important CRP techniques[1] are nitroxide-mediated radical polymerization (NMP), atom-transfer radical polymerization (ATRP) and reversible addition-fragmentation chain-transfer polymerization (RAFT).

Unfortunately, not all CRP techniques are equally well suited for upscaling from small laboratory experiments into large scale industrial processes. Important factors include the cost efficiency and ecological profile of the controlling agent. For example, contamination of the polymer with residues of transition metals (Cu) is the main problem of ATRP whereas the color and odor caused by the end groups (*e.g.* dithioesters) is a drawback of RAFT. For these reasons, NMP is the industrially preferred method; the controlling agent is an unproblematic metal- and halogen-free N-alkoxyamine. However, the initially used 2,2,6,6-tetramethylpiperidine-N-oxyl (TEMPO) based N-alkoxyamines are only suitable for the CRP of styrene but not of other industrially important monomers such as acrylates.

To overcome this limitation Ciba, (now part of BASF) developed a range of proprietary and upscalable nitroxides and related N-alkoxyamines which are suitable for CRP of both styrene and acrylates.

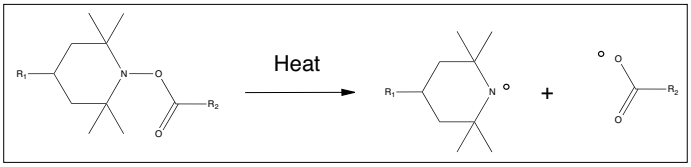
Using such N-alkoxyamine controlling agents, Ciba was the first company worldwide to make CRP an industrial reality. The novel polymer specialties, in particular the block copolymers, manufactured by this new technology set new standards in the coatings and plastics industry in applications such as dispersing additives[2] for pigments in the coatings industry (Scheme 2).



Scheme 2. Synthesis of an amphiphilic block-copolymer by NMP.

In addition, the technology can find use in electronics materials, lubricants and technical adhesives.

During the work on N-alkoxyamines for NMP, it was discovered that the related N-acyloxyamines are efficient radical initiators[3] (Scheme 3) and serve as safe (*e.g.* not explosive or flammable) alternatives to state of the art peroxides. Development and commercialization of a first novel N-acyloxyamine radical initiator for use in controlled rheology of polypropylene will be described in the second part of the presentation.



Scheme 3. Acyloxyamine radical generator (peroxide replacement), simplified mechanism.

This work was rewarded with the "Sandmeyer Award 2011" of the Swiss Chemical Society.

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[2] F. Pirrung, C. Auschra, Polymeric dispersants, in 'Macromolecular Engineering', Vol. 4, Eds. K. Matyjaszewski, Y. Gnanou, L. Leibler, Wiley-VCH, Weinheim **2007**, p. 2135–2180.

[3] M. Roth, R. Pfaendner, P. Nesvadba, M.-O. Zink, WO2001/090113 A1, Ciba SC, **2001**.

Received: March 9, 2012