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Production Today – Tomorrow: New Technologies in Modern Chemistry

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Production and modern research have already contributed towards development in many complex chemistry process. Our industry is still experiencing increased performance and technology demands. The speakers presented lectures on the latest developments in fractional layer crystallization, high-performance distillation, phosgene processes, front-end engineering, UV and electron beam crosslinking, and enantioselective chromatographic separations.

Keywords: Chromatographic separation · Crystallization · Distillation · Electron beam crosslinking · Front end engineering · Industrial chemistry · Phosgenation

The Contribution of the Chemical Industry and the 'Chemiker FH' to the Swiss Economy

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The chemical-pharmaceutical industry contributes 5% to Switzerland's gross domestic product. Almost 70000 people earn their income in the chemical-pharmaceutical industry; about 1400 of them are 'Chemiker FH', graduates of the Universities of Applied Science, who work mainly in analytical chemistry, development, production, application technology or engineering. These are very typical domains for graduates from the Universities of Applied Science who have cultivated their ability to apply science through apprenticeship in the industry.

An analysis of data from the industry and the Universities of Applied Science show a deficit of approximately 30–40 young 'Chemiker FH' graduates per year. To improve this situation, the following action points are proposed to attract more young people to study chemistry at the Universities of Applied Science:

- Cultivate 'gleichwertig aber andersartig', equal but different, almost as a trademark.
- The ECTS (European Credit Transfer System) must be applicable to studies at the Universities of Applied Science.
- The practical experience and schooling gained during the apprenticeship must be honored appropriately.
- 'Chemiker FH' students must be able to pursue a Bachelor's *and* a Master's degree at the Universities of Applied Science.
- The necessary legislation (FHSG and FHSV) must be implemented as soon as possible and the financial investments have to be approved at federal and cantonal level.

For the full text, see page 77 in this issue of CHIMIA.

Fractional Layer Crystallization for Refining of Organics

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Numerous organics are purified by layer-melt crystallization, which relies upon freezing crystalline layers of a purified component out of molten crude mixtures on cooled surfaces. Subsequently, after drainage of the residual, non-crystallized melt, the crystals are melted down. Crystallization may be performed from a stagnant or an agitated (*e.g.* falling film) melt.

The morphology of the crystal layers generally depends on the impurities content in the crude melt, on the diffusion coefficients of the impurity components and on the layer growth rates. The layers are compact and have rather smooth surfaces if the initial impurity content is low. Falling film crystallization can then be applied which allows faster crystal growth rates. If the impurity content is high the crystal layers tend to strong dendritic growth and exhibit rather lower mechanical stability. In such cases crystallization from a stagnant melt is more suitable. To cover wide ranges of impurity contents for a given product the combination of falling film and stagnant

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melt crystallization has been applied in industrial processes.

For certain applications suitable mechanical devices have been developed which additionally stabilize the crystal layers in a crystallizer and prevent negative effects on the process performance.

High-performance Structured Packing

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Different types of packing are optimal for each specific distillation application. For instance, dumped packing is generally the best choice for applications with higher liquid loads, whereas gauze packing tends to be the best choice for operation at low pressures. Metal sheet and grid-type packings are suitable for a wide range of applications and are particularly suited to applications where the maximal allowable pressure drop, limitation in height *etc.* are of importance.

Recent developments of high-performance distillation internals were presented, internals that provide high throughput and separation performance at lowest pressure drop. In addition some criteria for internals in the Miniplant application were presented in order to guarantee a sufficient data base for proper scale-up. A further outlook was given for areas of possible future improvement.

Safe Phosgene Generation and Phosgenation

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Classical phosgenation chemistry and common production routes require a solvent and a high level of dissolved phosgene, which is defined as a potential hazard. This leads to high investment and operation costs due to solvent recycling and/or destruction of the excess phosgene. Compared to the above Davy Process Technology's (DPT) advanced phosgenation technology allows the reaction to proceed with far less or even no use of a solvent at atmospheric pressure. Furthermore, the required excess phosgene can be kept close to the stoichiometric level, so that the phosgene hold-up and final destruction of the excess amount of phosgene can be minimized or even neglected.

DPT demonstrates a clear break-through within the field of dynamic phosgene generation and phosgenation technology and its concept of safety. The technology covers:

- A primarily safe production of phosgene gas, produced on demand in its intrinsically safe generator, without any requirement for liquefaction or storage.
- A primarily safe and efficient phosgenation process, which selectively reacts the generated phosgene within the downstream advanced phosgenation reactor with a minimum excess and hold-up level of phosgene.
- The complementary safety of the entire plant is given by the process absorption and the safety absorption units as well as the containment system and the plant control system with its hard-wired logic, which allows a safe and easy operating process.

UV/EB Crosslinked Polyacrylate Nanocomposites and Their Applications

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Nano-sized silica particles were used as fillers for acrylates. Because of their nanometer size and large active surface area radiation cross-linked polyacrylate nanocomposites exhibit markedly improved properties as compared to pure polymers: increased modulus and heat resistance, improved scratch and abrasion resistance as well as reduced gas permeability. This makes them very promising as coatings for technical applications.

For favorable embedding of the silica particles within the acrylate matrix the surface of the fillers was chemically modified by reaction with organosilanes such as methacryloxypropyl trimethoxysilane or vinyl trimethoxysilane. Acid-catalyzed condensation of the organosilanes yields a polysiloxane shell which is linked to the silica surface *via* reaction with surface OH groups (Fig. 1).

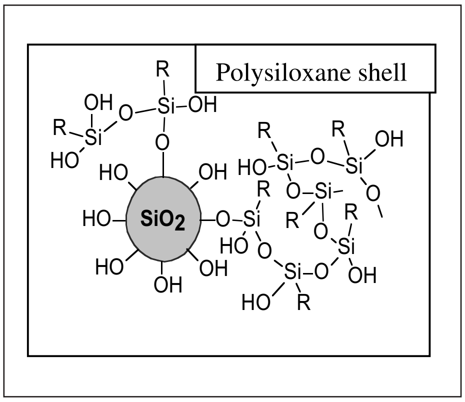

Fig. 1. Polysiloxane shell

The formation of covalent Si-O-Si-R bonds by condensation of silanes and/or by reaction with OH groups on silica was demonstrated by multinuclear MAS NMR, diffuse reflectance FTIR and XPS. From the following tentative substructures of the polysiloxane shell mainly bidental and tridental surface complexes (right) were identified by NMR (Fig. 2). These findings are in accordance with fragmentation pattern of the polysiloxane shell as measured by MALDI-TOF.

The REM image shows a typical surface topology of a polyacrylate nanocomposite. It can be seen that agglomerates are embedded within the organic matrix (Fig. 3). However, coatings with a thickness up to 50 µm remain transparent.

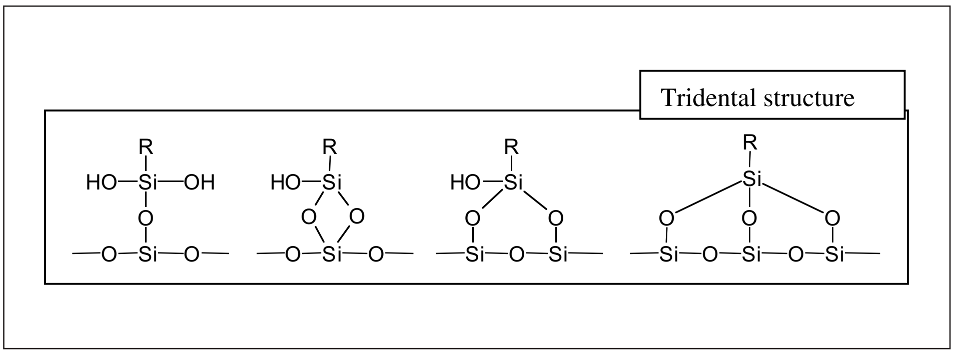

Fig. 2. Tridental structure

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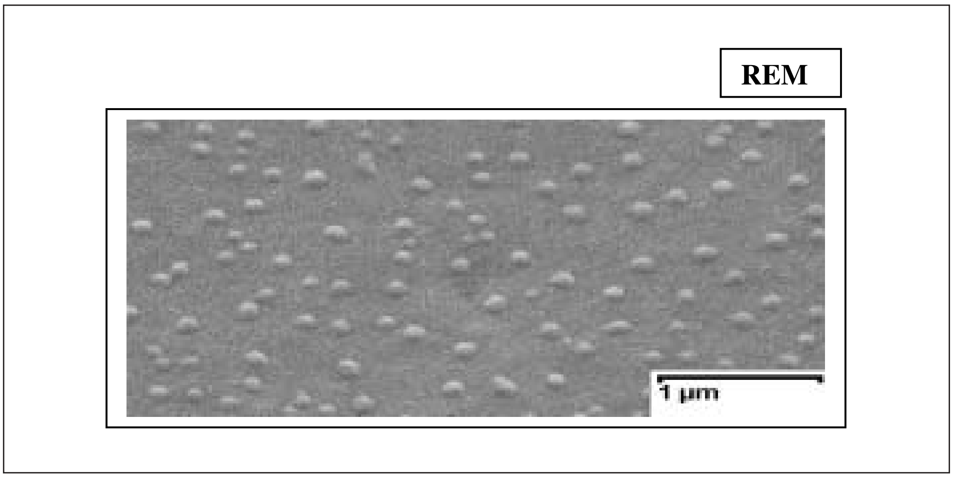

Fig 3. REM

Front-end Engineering for Pharmaceutical & Biotech Industry

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Linde-KCA has developed a front-end engineering approach for the pharmaceutical and biotech industry to allow our clients to make an informed early investment decision, and to minimize the overall project schedule.

Key issues that effect the client's investment decision are the total investment cost (TIC) and uncertainties and risks in process and project development. To address these issues the principal deliverables of Linde-KCA's front-end engineering approach are the technology concept review and the block layout development, with a subsequent investment cost estimate, and a process risk analysis. Using benchmarking factors will ensure the speed and accuracy of the development of the cost estimate. The process risk analysis will be performed in conjunction with the client and includes a review of the R&D work underway and an evaluation of the risks associated with scale-up and implementation of new technologies. In this way critical process issues can be identified at an early point in the schedule to allow for a faster and technically superior process configuration.

With the block layout development and the process risk analysis completed, a first project execution plan can be generated. A coordinated and synchronized engineering approach by all disciplines then ensures the fastest possible conceptual and basic design.

Enantioselective Chromatographic Separations in the Pharmaceutical Industry – Problems, Solutions, and Surprises

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Enantioselective chromatography has become a *sine qua non* for the development of a new chiral active pharmaceutical ingredient (API) in the pharmaceutical industry on analytical, preparative and production scale [1]. Liquid chromatography in particular has shown a tremendous potential for quick and reliable up-scaling using simulated moving bed chromatography [2]. SMB, as the process is known, is a large-scale version of traditional high-performance liquid chromatography used by countless laboratories to purify or separate mixtures of compounds.

Unlike HPLC, however, SMB is a continuous process in which a solvent and the compounds to be separated are injected into and withdrawn from a ring of chromatographic columns at rotating points between the columns. This technique simulates movement of the chromatographic packing material, or bed, against the solvent stream and allows for continuous recovery of the desired compound. The data obtained at the analytical level, *i.e.* retention times, selectivity, *etc.*, can be directly used to scale-up the separation at the preparative or production scale [3] under full compliance with regulatory authorities, *e.g.* the FDA. And, since a racemate synthesis is typically easier, cheaper and less time consuming than a stereoselective one, whose enantioselectivity is often less than 100%, a cheap and simple process to produce both enantiomers of an API, with high enantiomeric excess and high recovery, is obtained when racemate synthesis is coupled with racemate resolution using a SMB unit.

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