Characterisation of Technical Processes in Chemical Engineering

Prof. Dr. Ing. Hermann Nirschl | Institute for Mechanical Process Engineering and Mechanics

The Institute for Mechanical Process Engineering and Mechanics (MVM) is a part of the department of Chemical Process Engineering at the Karlsruhe Institute of Technology (KIT). The section "Process Machines" (short VM) is one of the three chairs of MVM. On the one hand the Nirschl group´s traditional work field is the development of machines for solid-liquid separation processes. On the other hand Nirschl´s group is increasingly working on the development and establishment of characterisation techniques for nanoparticles by SAXS (small angle X-ray scattering) and for soft matter by diverse NMR methods (nuclear magnetic resonance).

Investigation of the formation and growth mechanisms of particles by means of SAXS

Recently, the advantage of short measurement time of SAXS analyses was exploited for a time-resolved investigation on the formation of colloidal SiO_2 multiplets, using a SAXS laboratory camera. By applying the socalled non-isometry ratio Q_{NI}, the SAXS data enabled us to observe the evolution of multiplet structure during the progress of the reaction. In order to control the process regarding desired particle morphology (spheres/multiplets, Figure 1), the current aggregation state and, consequently, the appropriate reaction conditions were determined.

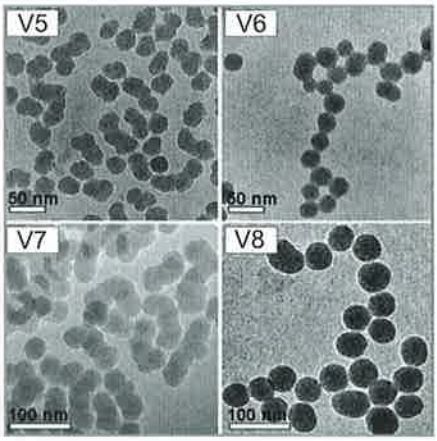

Fig. 1. Multiplet-like (left) and spherical (right) SiO_2 particles

Simultaneous small and wide angle X-ray scattering study of metallic and oxide nanostructured particles

Another project deals with the simultaneous determination of the X-ray scattering phenomena of particulate systems at small and wide angles (SWAXS). This nondestructive characterisation technique allows simultaneous measurements of the primary particle or pore size, surface and mass fractal dimensions, as well as quantitative determination of the crystallite properties, such as phase identification, the coexistence of crystallite components and their fractions. Figure 2 illustrates the SWAXS laboratory camera, developed at MVM.

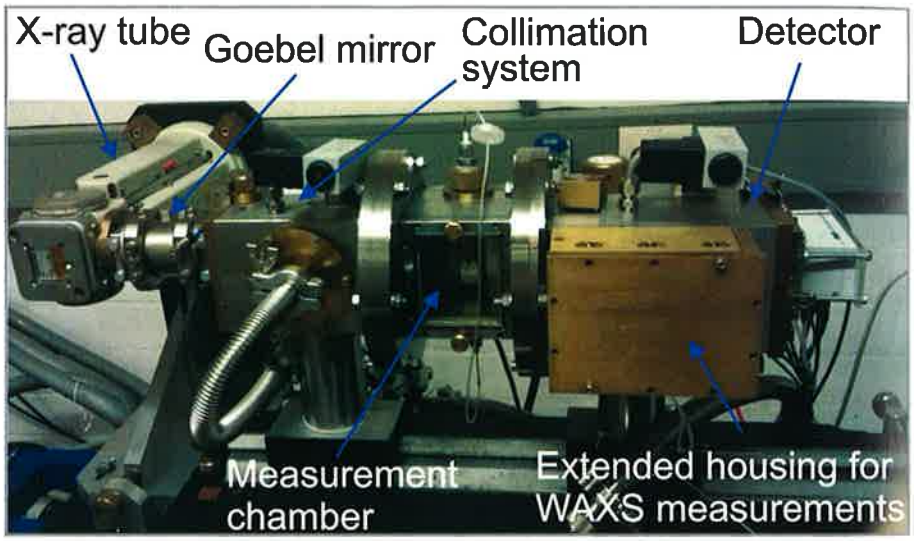

Fig. 2. SWAXS device at MVM laboratory

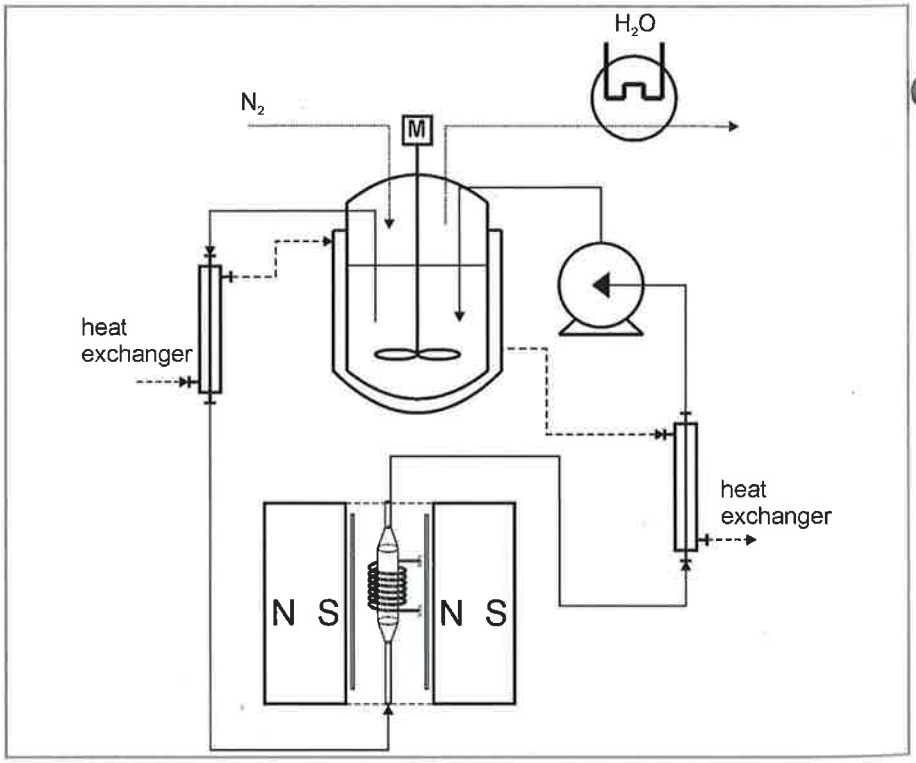

Fig. 3. Principle of the on-line process analytics in a bypass by low-field NMR-spectroscopy

Characterisation of Technical Processes in Chemical Engineering

Prof. Dr. Ing. Hermann Nirschl | Institute for Mechanical Process Engineering and Mechanics

The Institute for Mechanical Process Engineering and Mechanics (MVM) is a part of the department of Chemical Process Engineering at the Karlsruhe Institute of Technology (KIT). The section "Process Machines" (short VM) is one of the three chairs of MVM. On the one hand the Nirschl group´s traditional work field is the development of machines for solid-liquid separation processes. On the other hand Nirschl´s group is increasingly working on the development and establishment of characterisation techniques for nanoparticles by SAXS (small angle X-ray scattering) and for soft matter by diverse NMR methods (nuclear magnetic resonance).

Investigation of the formation and growth mechanisms of particles by means of SAXS

Recently, the advantage of short measurement time of SAXS analyses was exploited for a time-resolved investigation on the formation of colloidal SiO_2 multiplets, using a SAXS laboratory camera. By applying the socalled non-isometry ratio Q_{NI}, the SAXS data enabled us to observe the evolution of multiplet structure during the progress of the reaction. In order to control the process regarding desired particle morphology (spheres/multiplets, Figure 1), the current aggregation state and, consequently, the appropriate reaction conditions were determined.

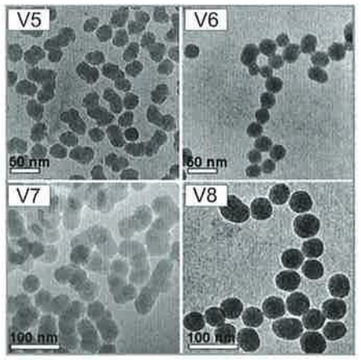

Fig. 1. Multiplet-like (left) and spherical (right) SiO_2 particles

Simultaneous small and wide angle X-ray scattering study of metallic and oxide nanostructured particles

Another project deals with the simultaneous determination of the X-ray scattering phenomena of particulate systems at small and wide angles (SWAXS). This nondestructive characterisation technique allows simultaneous measurements of the primary particle or pore size, surface and mass fractal dimensions, as well as quantitative determination of the crystallite properties, such as phase identification, the coexistence of crystallite components and their fractions. Figure 2 illustrates the SWAXS laboratory camera, developed at MVM.

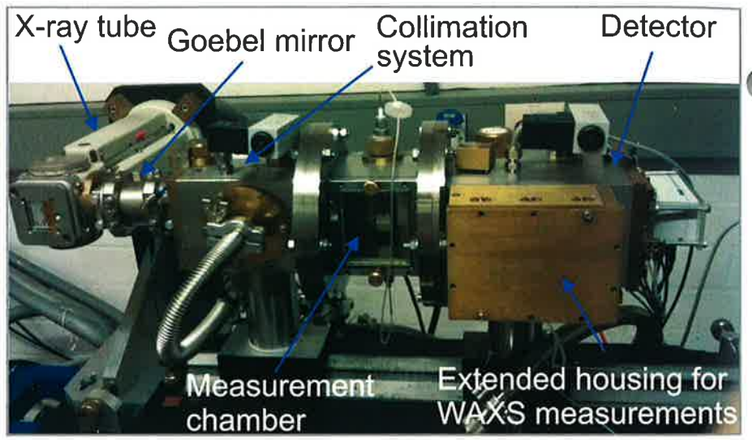

Fig. 2. SWAXS device at MVM laboratory

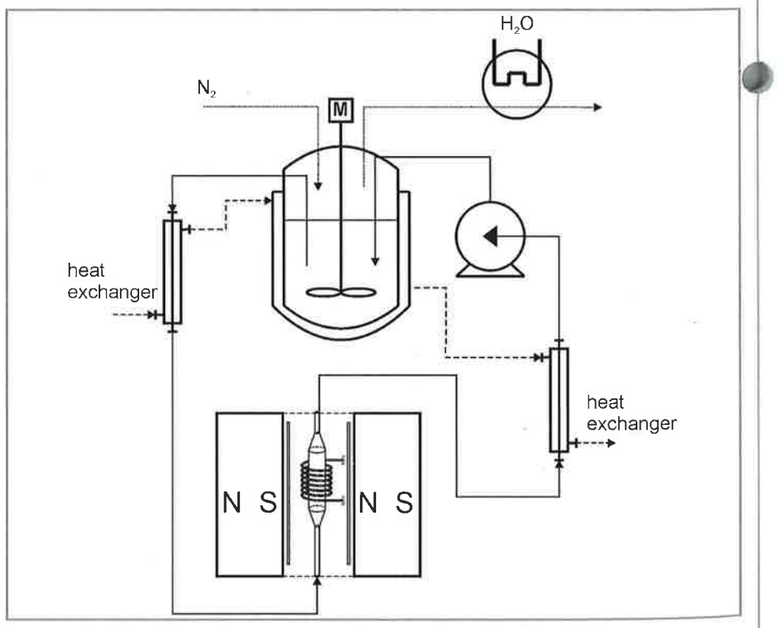

Fig. 3. Principle of the on-line process analytics in a bypass by low-field NMR-spectroscopy

Towards on-line process analyses by low-field NMR spectroscopy

On-line analysis of dynamic processes like chemical reactions by NMR allows detailed insight into mechanisms and kinetics. Especially permanent magnet based, low-field devices (1-85 MHz ^{1}H-frequency) provide a suitable instrumental basis for an application in a rough industrial environment. Technical and NMR developments were made such that the method can now be used for reaction monitoring at elevated temperature and pressure.

Characterisation of liquid dispersed systems by NMR

(Double) emulsions are complex dispersed systems with a high potential for encapsulation of active agents (collaboration with the Food Processing Department, KIT), which requires a detailed characterisation of structure and dynamics. NMR is a versatile tool for revealing product properties such as the droplet size distribution, the concentrations of the dispersed phases by means of NMR diffusometry, relaxometry, and spectroscopy. Most important, however, phase separation and release kinetics can be observed and quantified.

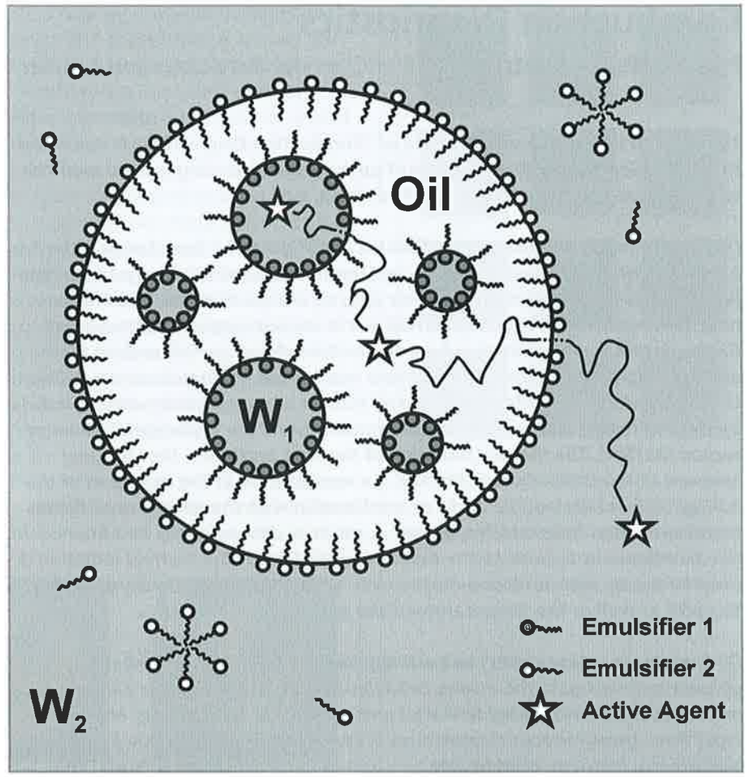

Figure 4: Typical structure of a double emulsion: The release kinetics of active agents is mainly described by diffusion through the hetero-phases.

Selected Publications

[1] Goertz V., Dingenouts N., Nirschl H.: Comparison of Nanometric Particle Size Distributions as Determined by SAXS, TEM and Analytical Ultracentrifuge Part. Part. Syst. Charact. 26, 17-24 (2009)

[2] Goertz V., Gutsche A., Dingenouts N., Nirschl H.: Small-Angle X-ray Study of the Formation of Colloidal SiO_2 Stöber Multiplets J. Phys. Chem. C 116, 2693826946 (2012)

[3] Guthausen G., von Garnier A., Reimert R.: Investigation of hydrogenation of toluene to methylcyclohexane in a trickle bed reactor by low-field Nuclear Magnetic Resonance Spectroscopy, Appl. Spectr. 63, 1121-1127 (2009)

[4] Dalitz F., Cudaj M., Maiwald M., Guthausen G.: Process and reaction monitoring by low-field NMR spectroscopy Prog. NMR Spectr. 60, 52-70 (2012)

[5] Dalitz, F. Steiwand A., Raffelt K., Nirschl H., Guthausen G.: ^{1}H NMR Techniques for Characterization of Water Content and Viscosity of Fast Pyrolysis Oils Energy Fuels 26, 5274-5280 (2012)

[6] Bernewitz R., Guthausen G., Schuchmann H.P.: NMR on emulsions: Characterisation of liquid dispersed systems Magn. Reson. Chem. 49, S93-S104 (2011)

Group Leader:
Prof. Dr. Ing. Hermann Nirschl

Since 2002: Professor at KIT, Chair of the MVM and Head of the section „Process Machines"

Working Fields:
mechanical process engineering, fluid process engineering, solidliquidseparation, threedimensional mixing processes, grinding technology, nanoscale particle structures, numerical simulation

Acknowledgements:
We acknowledge the financial support from the German Research Foundation (DFG No. Ni 414/13-1, SCHU1417/4-1, and GU1123/2-1) and the European´s Seventh Framework Programme under Grant Agreement No. 280765 (BUONAPART-E).