FOCUSING EXPERTISE, PROMOTE COOPERATION!

The Bayreuth Center for Colloids and Interfaces (Bayreuther Zentrum für Kolloide und Grenzflächen, BZKG) is a central research facility of the University of Bayreuth. Supported by a high-tech-initiative of the Free State of Bavaria it was funded in the year 2000. The BZKG supports companies seeking direct cooperation with the University of Bayreuth. It offers small and medium enterprises the use latest technologies, materials, and analytical methods to help sustain and develop their national and international competitiveness.

Colloids and Interfaces represent an interdisciplinary research area comprising physical, chemical, biological, medical and engineering science from basic research to product development.

Colloids are particles with diameters between one nanometer and several micrometers. They play an essential role in color pigments, catalysts, in washing detergents, shampoos, cosmetics, coatings, adhesives, polymers, ceramics, pharmaceuticals and food formulations. Also smallest droplets and bubbles in emulsions, cremes, sprays, foams and porous materials are investigated in this research area. In all such materials surfaces and interfaces play an essential role. The characterization of surface properties such as structure, roughness, wetting, adhesion, hardness and biocompatibility are therefore also an important part of research.

For the characterization of all these systems the BZKG offers outstanding competence, experience and an excellent and long list of equipment, being state-of-the-art on an international level.

The members of the BZKG are outstanding scientists, many having received prestigious national and international awards. Together they take care of research projects, provide materials, analytical methods, advanced training and education, and arrange national and international contacts to colleagues or research laboratories. In this brochure they introduce themselves together with their research expertise.

Please contact us for further information or support for your project.

Prof. Dr. Stephan Förster
Managing Director of Bayreuth Center for Colloids and Interfaces - BZKG

Prof. Dr. Rhett Kempe
“Polymer Supported Nanoparticulate (Nobel) Metal Catalysts”

Prof. Dr. Axel Müller
“Synthesis of nanoscopic polymer systems; analysis and characterization of complex polymer structures”

Prof. Dr. Georg Papastavrou
“Polymers at interfaces – single molecule techniques”

Prof. Dr. Markus Retsch
“Colloidal Self-Assembly and Templating as a Versatile Tool For Functional Materials”

Prof. Dr. Thomas Scheibel
“Understanding, modification and production of biopolymers and technical application thereof”

Prof. Dr. Hans-Werner Schmidt
“From molecule to material – synthesis, characterisation and application”

Prof. Dr. Jürgen Senker
“Ab-initio structure elucidation in amorphous, micro- and semi-crystalline materials, as well as at surfaces and interfaces”

Prof. Dr. Birgit Weber
“Molecular Switches with Memory Effect”

Prof. Dr. Stephan Förster
Director of BZKG

Prof. Dr. Andreas Fey
“Characterization and Modification of Interfaces”

Prof. Dr. Thomas Fischer
“Transport in viscous surroundings”

Prof. Dr. Stephen Gekle
“Simulation of single molecules and coflowcomplex”

Prof. em. Dr. Heinz Hoffmann
“Micelles, Colloids and their Applications”

Prof. Dr. Matthias Karg
“Highly ordered colloidal superstructures with tunable optical properties”

Prof. Dr. Seema Agarwal
“Biorelevant polymers, antimicrobial polymers, smart polymers, nanostructures and composites”

Prof. Dr. Josef Breu
“Synthetic clay minerals for microporous materials and nanocomposites, Solid-State Syntheses, Structure solution of disordered materials, Messtructured Materials, Colloid Chemistry.”

Prof. Dr. Andreas Fery
“Characterization and Modification of Interfaces”

Prof. Dr. Thomas Fischer
“Transport in viscous surroundings”

Prof. Dr. Stephan Gekle
“Simulation of single molecules and coflowcomplex”

Zof. Dr. Andreas Greiner
“Synthesis and speciality processing of polymer materials, electrospinning of nanofibers, metal nanoparticle-polymer conjugates, polymer coatings”

Prof. Dr. Seema Agarwal
“Biorelevant polymers, antimicrobial polymers, smart polymers, nanostructures and composites”

Prof. Dr. Josef Breu
“Synthetic clay minerals for microporous materials and nanocomposites, Solid-State Syntheses, Structure solution of disordered materials, Messtructured Materials, Colloid Chemistry.”

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Polymeric drug carriers

Amphiphilic polymers in solution can spontaneously self-assemble into micelles and vesicles. These are promising carrier systems for diagnostic and therapeutic applications. In micelles hydrophobic substances can be solubilized, whereas in vesicles hydrophobic as well as hydrophilic substances can be encapsulated. At the Chair of Physical Chemistry I we specifically develop micelles and vesicles for diabetes diagnostics and therapy. In this respect we investigate methods to load nanoparticles into the polymeric carriers. Nanoparticles serve to provide contrast for magnetic resonance tomography (MRT).

Self-assembly processes

At the Chair of Physical Chemistry I a major focus is the investigation of self-assembly processes of nanoparticles and polymers. Spherical nanoparticles can be assembled into well defined multiplets (s. Fig. above). Amphiphilic polymers assemble into a large variety of structures. These materials can be used as templates for the production of mesoporous oxides (see Fig. below).
Biodegradable polymers

Polymers with positively charged ammonium moieties form polyelectrolyte complexes with deoxyribonucleic acids, which are of potential interest for gene therapy. The copolymerisation with cyclic ketene acetals makes new positively charged polymers available, which are biodegradable after gene transfection into the cell nucleus. This fulfills an important prerequisite for the application of these polymers in gene therapy.

Antimicrobial polymers

Polymers with positive charged moieties are investigated intensively by the Agarwal team as antimicrobial materials. Here, polyguanidines are in the focus which display typically high and fast antimicrobial efficiency at low toxicity against cells. Another specific characteristic of polyguanidines, is their non-leaching profile. No antimicrobial moieties release from the polymer chain, which is next to scientific question of relevance for technical failure analysis and patent issues. Mechano-responsive polymers are another core competence of the Agarwal team.

Smart polymers

Smart polymers are another focal point of the Agarwal team. Smart polymers change their properties significantly, for example the solubility or the color when an external stimulus is changed, for example a change in temperature, pH-value, concentration, or light. Smart polymers open completely new fields of applications, for example drug release, cell adhesion, or membrane technology.

Polymer nanospheres obtained by electrospinning with complex electrode geometries.

Nanostructures and composites

Nanostructures and composites are another focal point of the Agarwal team. Thin nanofibers in Polyimide films.

Nanocomposites of polyimide nanoparticles and nanofibers in Polyimide.

Bacteria on polymer films with thermo-responsive antimicrobial properties.

Biorelevant polymers

Biodegradable polymers are of major importance for numerous applications which are not restricted to medicine, pharmacy, agriculture. Biodegradable polymers gain more and more relevance as packaging and construction materials for ecological reasons. The Agarwal team elaborated by ring-opening copolymerisation of cyclic ketene acetals and classic vinyl monomers a new class of biodegradable polymers with a huge potential for applications. Examples for new applications are biodegradable coatings, biodegradable carriers in gene transfection, biodegradable additives, and biodegradable rubbers and gels. The biodegradability could be efficiently tuned by the variation of comonomers and precision control of the polymer microstructure. Radical ring-opening homo polymerisation of cyclic ketene acetals like 2-methylene-1,3-dioxepane yields amorphous poly-epsilon-caprolacton (PCL). In contrast, PCL obtained by classic ring-opening polymerization is crystalline and therefore non-transparent. Degradation rates and transparency could be efficiently tuned over a wide range by mixtures of amorphous and crystalline PCL, since the biodegradability is ruled in particular by the solid state morphology. This new polymer materials could result in completely new applications for example for packaging applications or for agricultural applications.

Complex polymer materials

- Biodegradable and biocompatible polymers
- Antibacterial polymer materials
- Smart polymers
- Biobased polymers
- Polymer nanocomposites
- Complex Polymer-nanofibers

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Photo polymerisation

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Polymer nanospheres obtained by electrospinning with complex electrode geometries.
In the widest sense, our research focuses on hierarchical structuring of solid matter by programmed self assembly. Structuring spans the length scales from atomic to mesoscopic. To the same extent we succeed in complementing the diversity of chemical compounds by the complex structuring seen in biological systems, novel materials with hitherto unknown functionalities will become available.

“Chemical Tailoring” of Clay Minerals

Clay minerals are formed by weathering of primary rocks and are therefore ubiquitous on the earth’s surface. High adsorption capacity, cation exchange ability, catalytic activity, a very good sealing effect against water, and superb rheological properties make natural clay minerals to one of the most important industrial minerals. We concentrate on the melt synthesis of clay minerals as versatile starting materials for a variety of technical applications. We apply classical methods of solid-state synthesis as well as novel scalable processes (e.g. skull melting) especially when having potential industrial applications in mind.

Microporous Hybide Materials

Porous materials attract considerable interest in chemistry, physics, and material sciences due to their versatile and extraordinary properties giving rise to applications e.g. as storage materials, mole sieves, or (shape) selective catalysts and sensors. Our group turns synthetic layered silicates into microporous hybrid materials. Physiosorption measurements of intercalation compounds of such synthetic smectites with molecular spacers (“Pillared clays”) reveal a monomodal microporosity. The pore size can be fine tuned over a broad range by adjusting the charge of the pillars and the charge density of the host lattice. Moreover, by selecting appropriate pillars a variety of functionalities can be introduced.

Nanocomposites

The first “nanocomposites” have been produced as early as in the middle of the 19th century: “Simple” soot was added to rubber blends since the era of industrialization. By compounding nanosopic fillers into polymer matrices large interface areas are generated and significant effects are seen even with low filler contents. Over the last decades, the range of nanocomposite fillers has been broadened considerably. We focus on the synthesis and modification of classic inorganic fillers: Layered silicates (Clays) and Layered Double Hydroxides, LDHs. We seek to make nanocomposites with improved properties (gas barrier, mechanical, and flame retardancy) without sacrificing processability or optical and electronic properties.

Mesotechnology

Mesotechnology represents the link between the macroscopic world and nanotechnology. Tailor-made nanoparticles allow to do a kind of “chemistry” with them and it is possible to form “crystals” from such colloidal building units. Layered silicates can easily be exfoliated yielding nanoplatelets with high aspect ratios ideally suited to fabricate onedimensional colloidal crystals. Layer-by-layer assembly of cationic polyelectrolytes and negatively charged layered silicates produces highly oriented materials. Following this simple biomimetic approach artificial nanocrude materials with superb mechanical properties are obtained.

Polymer Screening

Patent protection, e.g. of a pharmaceutical drug, normally is related to a certain crystal structure. Therefore, when submitting a patent, a detailed knowledge of all accessible metastable solid forms besides the thermodynamically stable polymorph is essential. To be able to catch metastable Ostwald products in microcrystalline form, it is essential to be able to selectively induce the nucleation of the desired polymorph as well as to suppress nucleation of competing more stable polymorphs. State of the art sensors connected to an automated laboratory reactor (LabMax, Mettler-Toledo) allow to reproducibly control crystallization in some detail and enable monitoring of nucleation almost in real time.

Equipment and Infrastructure

Pore sizes, pore types, and specific surface areas are determined via physis- and chemisorption (Autosorb-1) applying different gases like nitrogen, argon, krypton, or carbon monoxide. Aside of conventional X-ray structure analysis methods we have established in-situ powder X-ray diffraction at defined relative humidity (THC, Anton Paar) as well as at controlled temperature and gas pressure (up to 900°C, up to 10 bar, XK, Anton Paar). We have access to further important analytical methods via collaborations and contacts to different scientific groups in Bayreuth: EDX (IMA, Bayreuth), ICP-OES (BGI, Bayreuth), Microprobe (BGI, Bayreuth), and impedance spectroscopy (FAN, Bayreuth). In cooperation with TU Munich iron-containing layered silicates are studied by Moesbauer spectroscopy.

All instruments are also used for fundamental research projects and are operated by skilled scientists and technicians. Thus, a competent contact person is always available.

OVERVIEW OF EQUIPMENT

• STOE Stadi-Powder Diffractometer (Cu-Radiation)
• Siemens D5000 Powder Diffractometer (Cu-Radiation)
• Panalytical Xpert Pro Powder Diffractometer (Reflection Geometry)
• STOE IPS’S U1 Single Crystal Diffractometer System (Mo-Radiation)
• Linseis DSC L 63
• Linseis DTA/TG L 61
• Quantachrome Autosorb-1 (Physi- and Chemisorption)
• Microfluidics Inc. Microfieleder M10Y
• Particle Metrix Stabilizer Streaming Potential Measuring Device
• Mettler Toledo LabMax (with ReactIR and FBRM-Probe, Lasentec)
• Büchi Stirring Autoklav for Polymerization
• Bruker if66/66IR
• Cary 5 UV/Vis- NIR
• SurfaceNet GmbH Skull-Melting Oven
• Linn HighTherm Graphite oven with rotatable tube
• NT 1900
• Hüttner High Frequency Oven 20 kW TIG 20/300

ACCESS TO VARIOUS POOLED EQUIPMENT:

• Energy Dispersive X-ray Spectroscopy EDX (IMA, Bayreuth)
• Microprobe-Analysis (BGI, Bayreuth)
• Laser Ablation-Inductively Coupled Plasma-Mass Spectroscopy (BGI, Bayreuth)
In Situ combination of AFM force spectroscopy with optical microscopy for characterization of microcapsule deformation.

Interactions and Micromechanics

Force spectroscopy is a recent development in Atomic Force microscopy. In this technique, rather than surface imaging, precise measurements of surface interaction forces or deformation forces is central. Microparticles, fibers or other micro-objects can directly be attached to microscopic cantilevers, as shown in the image on the left hand side. Thus repulsive interactions, adhesion or even friction of these particles with other surfaces can be precisely determined. In a similar fashion mechanical properties of coatings and deformable particles like microcapsules are accessible.

Coating and patterning of surfaces

The chair for Physical Chemistry II have many years of experience especially in the field of polyelectrolyte-multilayers and block copolymer films. In these systems the layer thickness and composition can be controlled at the nanoscale. Also composite coatings of polymers and inorganic nanoparticles can be realized. In addition, homogeneous chemically or topographically patterned coatings can be achieved without requiring elaborate and expensive lithography steps by contact printing methods or controlled wrinkling of surfaces. These building blocks allow the production of (multi-) functional and responsive coatings of different kind.

Customized particles and microcapsules

Particles can be coated with the polyelectrolyte multilayer technique in a very similar way as described above. This fact, makes that their colloidal stability and their interactions can be tailored to the necessary environmental conditions. This approach also allows the encapsulation of drugs and their controlled release.

Microscopy Methods

Scanning force microscopy is ideal for investigation of topography, interaction forces and nanomechanical properties of interfaces. Surface profiles can be determined with Nanometer-resolution in air or under solvents. Environmental conditions like temperature, vapor pressure or pH can be controlled and varied in situ. Advanced Atomic force microscopy imaging modes allow determining chemical information, friction forces and mechanical properties on the nanoscale. Modern optical microscopy methods like confocal laser scanning microscopy, for three-dimensional imaging or fluorescence correlation spectroscopy for investigation of transport processes and diffusion complement the spectrum of methods.

Accepted by other methods such as X-ray and neutron scattering, X-ray
The variety of chemical and biological functionalities of colloidal particle surfaces turns colloidal particles into an important tool in the medical, biochemical and biophysical field. Colloidal particles are being used for novel forms of DNA-sequencing, optical bar-coding, as carriers of biomolecules and proteins in microfluidic channels, and as fluorescent markers in microfluidic experiments of the cytoskeleton. One important type of colloidal particles are (super)paramagnetic colloids, i.e. colloids that contain a superparamagnetic core made of paramagnetic grains surrounded by a polymer shell. These particles can be manipulated using magnetic gradient fields. One prominent use is hyperthermia treatment of cancer, in which magnetic field gradients are first used to collect and enrich these particles in the cancerous part of the human body and then destroy the cancerous tissue using high frequency magnetic fields that heat the particles and the tissue. Important issues such as the compatibility and nontoxicity of these particles in the human body have been addressed extensively and they can be used directly when manipulating the same kind of particles in vitro in microfluidic devices that analyze or detect specific chemical or biological species.

Magnetic manipulation of paramagnetic particles in the human body is limited to fields with spatial variations on the macroscopic scale. These limitations, however, do not exist on a lab-on-a-chip; where magnetic fields can be created that vary on the colloidal scale.

Focus of our team is the development of magnetically, electrically or chemically controlled colloidal interfacial transport mechanisms. Homogeneous external fields are converted into fields that are heterogeneous on a colloidal scale. Such conversion allows the massive parallel manipulation of an ensemble of biochemically functionalized colloidal particles on a lab on the chip.

We feel that the potential that lies in the use of a dynamic magnetic field landscape varying laterally on the colloidal length scale has not been exploited to full extent and therefore needs to be explored.

magnetic colloids

We explore the full power of magnetic manipulation of paramagnetic particles on magnetically heterogeneous surfaces, i.e. surfaces with particularly chosen or designed domain patterns and stray field landscapes. The use of magnetic fields heterogeneous on the colloidal scale will enable simple controlled manipulations of a large assembly of colloidal particles, with individual control over each particle. We explore the multitude of fundamental colloidal transport modes that can be triggered in heterogeneous magnetic fields by using homogeneous time dependent external 3D magnetic field modulations. In contrast to optical tweezers manipulations the magnetic manipulation device can be miniaturized to the nanoscale. Miniaturization allows for simultaneous control of larger assemblies of particles. Developing magnetically controlled transport of paramagnetic particles on surfaces adds a powerful new tool for biochemical and biophysical analysis to the arsenal of microfluidic devices.

interfacial phenomena

Time resolved microscopic observation is the key to understanding of dynamic colloidal processes near interfaces. Transmission, Brewster angle, fluorescence, and polarization microscopy setups are ready to be used in our lab. You can choose between optical tweezers, magnetic squeezers, and magnetic shift registers if you want to transport and manipulate your colloidal assembly. Forces can be measured in the femto to pico Newton regime. Our key competence is the exploitation of complex dynamic hydrodynamic processes for your needs. All instrumentation can be used for fundamental as well as for applied questions and are operated by qualified scientific personnel.

KEY INSTRUMENTATION

- Brewster angle autocorrelation spectroskopy
- Optical tweezers and fluorescence microscopy incorporated into a Langmuir balance
- A magnetic squeezer combined with polarization microscopy
- A magnetic shift register combined with fluorescence microscopy
Molecular structure and dynamics of soft matter

The proper functioning of biological molecules depends not only on their chemical composition but also on their conformation, i.e., the spatial arrangement of the constituting atoms. This includes the intramolecular structure (for example the folding of a protein) as well as the supramolecular arrangement of molecules with respect to each other. A spectacular example of supramolecular ordering is the spontaneous formation of a lipid interface in water due to hydrophobic interaction. These processes can be investigated and understood using Molecular Dynamics simulations such as those employed within our research group.

Example application I: Organic solar cells using perylene dyes

Perylene molecules are strong dyes which are able to absorb sun light very efficiently. Besides that, they are very good electron conductors such that the absorbed energy can be transported rapidly and efficiently through the material towards an outer contact. For this fast charge transport supramolecular ordering, so called “stacking”, of perylene molecules plays a decisive role. With the help of our simulations we could show the existence of a thus far unknown structure (see image below) and thereby explain spectroscopic experiments conducted in the research groups of J. Köhler (Experimental Physics I) and M. Thelakkat (Macromolecular Chemistry I).

Example application II: Absorption of electromagnetic energy in cell membranes

The question whether electromagnetic radiation as used in telecommunication can have undesired effects on the human organism is currently the subject of intense and controversial societal debates. With the help of Molecular Dynamics simulations we recently calculated the absorption spectrum of cell membranes (see figure above) and derived quantitative predictions for the resulting temperature gradients within the cell.

Fluid dynamics of complex liquids

The term complex liquids refers to solutions of particles within a carrier liquid where the individual particles can be solid objects (colloids) such as sand grains, soft objects such as biological cells or even drops of a second, immiscible fluid. Due to this heterogeneous structure complex liquids behave partly as an elastic solid and partly as a viscous liquid: they are “viscoelastic.” Prominent examples include synthetic emulsions such as creams or paint, but also natural sub-stances like milk or blood.

Our research group investigates the fluid dynamics of complex liquids in detail. From the simulation results we can predict macroscopic properties such as the viscosity. At the same time, our simulations allow detailed insight into the microscopic dynamics, for example into the spatial distribution of particles in a channel.

Example application III: Microcapsules in blood flow

Intensive worldwide research efforts are currently being dedicated to possible applications of synthetically produced microcapsules as drug delivery agents. Here, the drug is incorporated into a microcapsule which is injected into the blood stream. The capsule then circulates towards the desired destination (e.g., a tumor) where it is destroyed to release the drug.

With the help of simulations such as those conducted in our group one can investigate the behavior of microcapsules during their circulation. An important question here is which external factors (surface chemistry, shape, deformability) are most influential for the dynamic behavior and whether clustering of particles can occur at certain locations in the microcirculatory system.

SIMULATION METHODS

- Molecular Dynamics
  - Spatial arrangement of complex molecules
  - Supramolecular structure formation
  - Dynamic conformational changes (folding)
- Lattice-Boltzmann/Boundary-Integral
  - Flows of complex liquids
  - Macroscopic properties: Viscosity
  - Microscopic properties: Density distribution, deformation of individual cells/drops
This leads to stable nanofibers. relatively low glass transition temperatures during electrospinning. Film formation of the dispersion particles made of polymers with water-stable fibers. The trick is here a one dimensional mate disruption technique of pest insects very good success rates. The usage of pheromone loaded nanofibers showed in the example is the application of nanofibers in biotechnical plant protection. The core competence of the Greiner team at the chair of macromolecular chemistry II is the synthesis of polymer materials and their processing to thin films, nanofibers, and nanoparticles by speciality techniques. Particular focus of the team is on electrospinning of nanofibers which display large surface to volume ratio and versatile options for functionalities. This results in numerous applications, for example in filtration, textile, agriculture, catalysis, tissue engineering, drug release, and composites. Characteristic for the accomplishments of the team are material developments which could be utilized in specialty processings under realistic and technical conditions. An example is the application of nanofibers in biotechnical plant protection. The usage of pheromone loaded nanofibers showed in the mate disruption technique of pest insects very good success rates. However, electrospinning on agricultural fields became possible only much less in comparison to the diameter of a human hair. Polymer nanofibers are obtained as nonwovens with diameters of the nanofibers much less in comparison to the diameter of a human hair. A small amount of a water soluble polymer has to be added which does not harm in agricultural applications. These tailor-made systems allow highly innovative approaches to solutions for application relevant technical problems utilizing polymer chemical methods. Electrically charged polymer dispersion particles in water yield polymer nanofibers with surface charges, which can be utilized by simple dipping for further functionalizations. The surface of these versatile system was functionlized selectively by dyes, catalysts, and antibacterial metal nanoparticles for different applications. The surface could be changed like a chameleon changes it color on demand. The electrospinning of dispersions offers numerous other options for the preparation of highly functional polymer nanoparticle nanowovens. Electrically charged polymer dispersion particles in water yield polymer nanofibers with surface charges, which can be utilized by simple dipping for further functionalizations. The surface of these versatile system was functionlized selectively by dyes, catalysts, and antibacterial metal nanoparticles for different applications. The surface could be changed like a chameleon changes it color on demand. The electrospinning of dispersions offers numerous other options for the preparation of highly functional polymer nanoparticle nanowovens. Electrically charged polymer dispersion particles in water yield polymer nanofibers with surface charges, which can be utilized by simple dipping for further functionalizations. The surface of these versatile system was functionlized selectively by dyes, catalysts, and antibacterial metal nanoparticles for different applications. The surface could be changed like a chameleon changes it color on demand. The electrospinning of dispersions offers numerous other options for the preparation of highly functional polymer nanoparticle nanowovens. Electrically charged polymer dispersion particles in water yield polymer nanofibers with surface charges, which can be utilized by simple dipping for further functionalizations. The surface of these versatile system was functionlized selectively by dyes, catalysts, and antibacterial metal nanoparticles for different applications. The surface could be changed like a chameleon changes it color on demand.
BayColl was founded by Prof. em. Heinz Hoffmann in 2003 to follow applied research and to increase consulting for companies. BayColl works in cooperation with members of the BZKG (University of Bayreuth). Inquiries from companies in the local environment and also partners working worldwide are initially fed back from the practical problem of the physicochemical principles. According to this, practical solutions are developed in the laboratories of BayColl and also with partners. In the laboratories, up to six employees are engaged in such projects. The available experimental methods are designed for applications.

Rheology, differential thermal analysis, microscopy and also pure research equipment are included. New materials with cement and silicates as well as patent consultations are typical examples of the activities. Based on surfactant science, the scientific field of BayColl has extended to questions on almost all aspects of colloid chemistry.

Preparation of colloidal systems

The phase behaviour of surfactant still plays a central role in the research field of BayColl. Defined by the selected components, samples can range from water viscosity liquids to high viscosity materials with a yield stress. These types of fluids and phases have special rheological properties and can be used to optimize various formulations, such as in crop protection, metal machining and drilling fluids.

Characterization of colloidal systems

The explanation of macroscopic properties can always be found in the microscopic structures. For this purpose, BayColl has developed a number of methods that provide valuable information about the microstructure of complex systems. Simple methods are used such as polarized-light microscopy and electron microscopy for modern use. For detailed scattering experiments, small angle neutron scattering and small angle X-ray scattering measurements are carried out in cooperation with various research centres. For the investigation of anisometric particles, the electric birefringence method is used. Focus is the detailed analysis of rheological data. This allows to draw conclusions about the microstructure. Often, however, only the combination of different methods give evidence about the actual structure.

Optimization of colloidal systems

Examples are:

- Emulsions and dispersions for cosmetic and agrochemical industry
- Viscoelastic fluids for the petroleum industry
- Gelation of liquids
- Microemulsions for cosmetic applications
- Additives to control properties of cement
- Surfactant solutions and phases for the agrochemical industry
- Proteins as helping components for the cosmetic industry
- Aerogels coatings for various substrates
- Surfactant solutions for flow acceleration
- New hybrid materials for coating of surfaces (lotus and Antifogging effect)

Available equipment

- Rheology
- Flow birefringence
- Electric birefringence
- Scanning Electron Microscopy
- Cryo-TEM/SEM and freeze-fracture in cooperation with members of the BZKG
- Surface tension
- Interfacial tension
- High pressure emulsifier
- Classical and dynamic light scattering in cooperation
- Small-angle X-ray and neutron scattering in cooperation
- For specific methods we have also access to centralized shared facilities and connections to international research groups.

Typical particle size and spacing in colloidal solutions are in the range of the wavelength of visible light and therefore can lead to impressive colours, depending on the viewing angle.
Plasmon resonance coupling and plasmonic structures

Surface plasmon resonances are collective oscillations of the electron cloud of metallic systems. These resonances become energetically localized if the size of the metal object is reduced to the nanoscale. The most popular examples of particles showing such localized surface plasmon resonances (LSPR) are gold and silver colloids. Due to their intense coloration these particles have been used since ancient times. The position of the LSPR directly depends on the particle size and shape but also on the dielectric properties of the surrounding medium. Furthermore, in case of anisotropic particles, a polarization dependence of the resonances is found.

In the PHYSICAL CHEMISTRY – COLLOIDAL SYSTEMS we are especially interested in plasmon resonance coupling effects. If, for example, two individual gold or silver nanoparticles are brought into close vicinity, coupling of the plasmon resonances appears affecting the resonance position, intensity and band width. Hence, methods which allow to control the interparticle distance statically or dynamically will allow the preparation of materials with tunable optical properties. One approach we employ to realize materials with tunable optical properties is the combination of stimuli responsive polymers with gold or silver nanoparticles.

Colloidal self-assembly

To which extend self-assembly can be driven is one of the major questions our research aims to answer. Of particular interest for us is the assembly of nanoscale building blocks such as metal nanoparticles. In order to achieve large scale assemblies in 2D and 3D we employ soft, deformable polymer coatings/ligands, which present a tunable separator between the metal nanoparticles and allow to obtain unusual structural symmetries. The different synthetic protocols we use can be applied to prepare shell thicknesses ranging from a few nanometers up to microns. In addition, we can adjust the surface charge of the soft shell and thus the interaction potential. Due to the softness of the shell, the phase behavior of these particles is significantly different compared to that of simple hard spheres.

We employ scattering methods such as small angle neutron scattering (SANS) and small angle X-ray scattering (SAXS) to determine the morphology and composition of core-shell colloids with different architectures. This is typically done in the dilute regime, where the particle form factor can be analyzed. For colloidal superstructures, these scattering tools are used for the analysis of the structure factor. This allows the determination of the crystal structure and the lattice constants. Furthermore, SANS and SAXS measurements are performed in combination with shear as a convenient approach to induce, align or destroy crystalline assemblies.

Core-shell particles

The coating of inorganic nanoparticles with organic polymer shells provides several interesting features: i) the polymer shell can enhance the colloidal stability; ii) the polymer shell leads to an increase of the effective particle volume. iii) hybrid materials with multifunctional properties can be created.

One interest of our research is the development of polymer coating strategies for different inorganic materials. As core materials we use silica, gold, silver, platinum and magnetite nanoparticles, amongst others. Crucial for a successful incorporation of these inorganic components is their surface functionalization, which is typically done via ligand exchange procedures. Depending on the polymer shell composition multifunctional core-shell colloids can be prepared. If, for example, stimuli responsive polymers are used for the encapsulation of inorganic nanoparticles, hybrid colloids with well-defined volume phase transition properties can be achieved. Consequently the shell thickness of such colloids will be a function of an external stimulus such as temperature, pH or ionic strength.
The core competencies of the Institute of Inorganic Chemistry II are focused on the research area of catalyst design. The research spectrum is distributed to the synthesis of porous systems (MOFs = metal organic frameworks, SiCN precursor ceramics in cooperation with G. Motz [Department of Ceramic Materials Engineering]), their functionalization with metal particles via various methods (MOCVD = metal organic chemical vapor deposition, application of metal modified precursors, etc.) and their catalytic applications.

Formation of porous structures

The Institute of Inorganic Chemistry II generates porous structures of 3D framework analogue coordination polymers (MOFs). Therefore organic poly acids are reacted with metal salts under hydrothermal conditions. Pores in the range between few Ångström to several nanometers are formed, designing the three dimensional structures.

Formation of the pore sizes

It is very easy to control the pore sizes and the geometry of the porous coordination polymers by a variation of the organic poly acids (linker) and the metal salts (SBU = secondary building unit). The high multifunctional character of the material category of MOFs is confirmed by the individual formation of the pores sizes for special applications (e.g. chiral pores). The pore sizes of the precursor ceramics could be regulated by the variation of the additives (ADC, urea, ammonium thiocyanate) or the fillers (various polymers) and by the pyrolysis conditions. Non-functionalized MOFs are applied as hydrogen storage materials and show an absorption capability up to 1 wt % H₂ at room temperature.

Functionalization of porous structures

The described structures could be functionalized by metal nanoparticles. The pores of the MOFs are filled with metals making the use of volatile metal organic compounds (palladium, platinum) under high vacuum (investigation of the species by solid state nmr method) and followed by the reduction with hydrogen. The size of the nanoparticles is directly affected by the size of the pores. The functionalization of the SiCN precursor ceramics occurs in a different manner. Therefore the precursors (polysilazanes) are functionalized by metals via the aminopyridinato metal chemistry, which is one of the leading research areas in the Inorganic Chemistry II. The cross-linking process follows as usual and thereafter the pyrolysis of the green body to the ceramic which ends in the precipitation of the metal particles (copper, platinum, palladium) in the ceramic.

Applications in catalytic reactions

Metal particle functionalized MOFs show a broad field of applications in catalytic reactions. Systems with chiral pores are especially of interest and should be able to catalyze enatioselective reactions. The room temperature hydrogen storage is also conceivable. The advantage of the SiCN ceramics in comparison to the thermally unstable MOFs is their robustness and their broad range of application under extreme conditions (temperature, caustic chemicals). This gives high importance to CH activation reactions and to equilibrium reactions in which the equilibrium is disadvantageous at low temperatures. The detection of the products is conducted by NMR, GPC-, GC- and HPLC instruments. The investigation of the GC products is detected by an online-GC.

EquPMENT OVERVIEW

- STOE IPDS II single crystal diffractometer (Mo radiation)
- Varian Unity Inova 300 MHz NMR
- Varian 400 MHz NMR with solid state NMR
- Bruker ARX 250 MHz NMR
- GCs with autosampler and chiral columns and an online-GC
- GC-MS
- GPC + HT GPC
- HPLC
- React IR
- ChemBET Pulsar
- Nova 2200 e
Synthesis of nanoscopic polymer structures

Using modern “living” polymerization techniques we synthesize complex polymer architectures, e.g. self-organizing amphiphilic block copolymers and nanoparticles. All methods for molecular, structural and thermal characterization as well as for polymer analysis are available.

Synthesis of nanoscopic polymer structures

We utilize “living” polymerization processes (anionic, cationic, and controlled radical polymerizations) for the directed synthesis of complex, self-organizing polymer nanostructures in a gram to kilogram scale. For this purpose, we use lab autoclaves (see image) from 250 ml up to 10 l and flow-tube reactors.

In particular, we have a strong expertise in the synthesis of linear and (randomly, comb- and star-like) branched polymers and block copolymers. In dependence on the architecture different bulk and solution structures are formed that control the mechanical and flow properties. They find widespread application, e.g. for thermoplastic elastomers, compatibilizers in polymer blends, adhesives, carrier for drugs, optical data storage, or as viscosity index improver in motor oils.

“Amphiphilic” block copolymers have hydrophilic and hydrophobic blocks and self-assemble into micelles of various structures in water (see image). They can be used as dispersant, e.g. in emulsion polymerization, for pigments, or in cosmetics. Depending on the structure they can respond to external stimuli (temperature, pH, salts) and form physical (reversible) networks. This is interesting for applications in cosmetics and pharmaceuticals. Moreover, the use of blocks made of natural polymers, like sugar or proteins, opens completely new application fields.

We put a strong focus on novel hybrid structures and nanoparticles. Janus nanoparticles, corresponding to the latter class, consist of two hemispheres with different polarity and form interesting hierarchical superstructures (see image). Amphiphilic core-shell molecular brushes have a hydrophilic core and a hydrophobic shell (or vice versa) and are used as nanoreactors for organic-inorganic hybrid structures.

Organic-inorganic hybrid structures

Using the new method of “self-condensing vinyl polymerization” it is possible to grow hyperbranched polymers from colloidal gold or silica particles. The large number of (almost freely adjustable) functional end groups on the surface add completely new properties to the materials. In an aqueous medium, “polymer trees” can be grown from planar surfaces, e.g. silicon, glass, or gold, too. In this way the physical (e.g. roughness, friction, wetting) and chemical (e.g. sensor technology, attachment of biomaterials) properties of the surface can be varied at will. A preceding patterning of the surface (lines, islands) allows to add the functions on specific places at the surface.

Analysis and characterization of complex polymer structures

State-of-the-art methods for the molecular characterization of homo- and co-polymers as well as the analysis of unknown polymer samples are available. The chemical composition and molecular weight distribution can be determined by the combination of chromatographic methods (size exclusion and adsorption chromatography, 2D chromatography, field-flow fractionation) with light scattering, viscometry, UV and infrared spectroscopy, as well as MALDI-ToF mass spectrometry.

Superstructures formed by the self-assembly of single molecules in bulk, in the melt and in solution are investigated by scattering methods, like wide- and small-angle X-ray scattering (WAXS, SAXS), static and dynamic light scattering (SLS, DLS), and by imaging techniques, e.g. transmission electron microscopy and scanning electron microscopy (TEM, SEM), scanning force microscopy (SFM). In addition, rheology and thermal methods, like differential scanning calorimetry (DSC), thermogravimetric analysis (TGA) and dynamic mechanical thermal analysis (DMTA), are used.

Transmission electron microscopy for studying the bulk morphology of block copolymers.

The size of nanoparticles is determined by dynamic light scattering (DLS).

Equipment for polymer synthesis:
- 7 lab autoclaves (Büchi) from 250 ml to 10 l for solution and emulsion polymerization
- Flow-tube reactors for extremely fast polymerizations (half-lives down to to 0.01 s)
- Fiber-optics NIR probes for online conversion determination combined with a portable diode array NIR spectrometer (Zenn)
- Glove-Box (M. Braun) and 6 high-vacuum lines
- 2 hydrogenation reactors (Büchi) for pressures up to 100 bar
- Freeze-dryer (Christ)

Equipment for polymer analysis:
- 300 MHz NMR spectrometer (Bruker Avance 300)
- FT-IR and FT-NIR spectrometers (Bruker, Nicolet) with ATR, dichroism setup, flow cell, fiber-optics immersion probes, IR microscope
- UV spectrometer (Perkin Elmer)
- Membrane osmometer
- 5 size exclusion chromatography (SEC) systems for organic solvents and water with UV and RI detectors, partially coupled with online viscosity detector (Wyatt Viscosort), multi-angle light scattering (Wyatt Heleos), FT-IR and MALDI interface; one system is coupled with liquid adsorption chromatography (+ 2D chromatography)
- Asymmetric flow field-flow fractionation (Wyatt Technology Eclipse 2) coupled with multi-angle light scattering detector (Wyatt EQS)
- MALDI-ToF mass spectrometer (Bruker Reflex III)
- Gas chromatograph (Fisons/THERMOquest)
- Static and dynamic light scattering (Sofica, ALV)
- Differential scanning calorimeters for bulk samples (PerkinElmer Pyris 1) and solutions (Setaram LSCII)

In addition, there is access to following key facilities:
- Transmission electron microscopy: Zeiss CEM 902, Zeiss EM922 OMEGA with cryo-TEM option
- Scanning electron microscopes (Zeiss) with cryo-SEM option
- Scanning force microscopes (SFM)
- Small- and wide-angle X-ray scattering (Seifert, Bruker AXS)
- Rheology lab (Rheometric Scientific: ARES, stress rheometer, DMTA, rheo-optic)
The group of Professor Papastavrou studies the properties of polymers at interfaces. Interfaces are of great importance for various properties of solids, like adhesion, friction, or their wettability by liquids. Thin polymer layers allow tuning these properties on purpose. Also the formation of bacterial biofilms on medical tubing or ship hulls represents a phenomenon, which is mediated by extracellular biopolymers. In order to understand polymer interfaces and to tune the aforementioned macroscopic characteristics, it is important to study their behavior on a molecular level. Description on the level of single polymer segments allows then for a rational approach in order to alter surface properties and to adapt them for specific applications.

In our research we study not only the adsorption of polymers to surfaces but also the properties of the polymer-modified interfaces. Additionally, we work on the development of single molecule techniques and the development of stimuli-responsive layers.

Analytical techniques

A very important technique for our research is the atomic force microscope (AFM). With this instrument we can study surfaces with a resolution in the nm-regime. The measurements can be performed in liquids or under controlled atmosphere.

For the measurements in liquids a cell-system is available, which allows controlling and measuring in a defined manner the pH, ionic strength and composition of the liquid phase.

The AFM does not only allow determining the surface topography but also to laterally resolve variations in surface properties such as charge, elasticity or distribution of functional groups. Furthermore, the AFM allows measuring the surface forces. The technique is sufficiently sensitive to even follow the extension of single polymer chains or to determine the interaction force between two single colloidal particles as function of their separation distance. Information on the single molecule level can be combined with techniques, which average over a larger surface area, such as ellipsometry, reflectometry and electrochemical or electrothermical methods.

Adhesion on the single molecule level

As shown schematically in the figure on the top left side, the extension of single molecules, e.g. polyacrylamides, can be followed by AFM. In this manner the stretching response of single polymer segments and its variation due to external parameters (pH, ions, etc.) can be determined directly. Furthermore, it is possible to measure by AFM the desorption of single polymer strands from solid substrates.

In combination with the “colloidal probe” technique one can measure also simultaneously long-ranged forces for a defined interaction geometry. Complicated adhesion events acquired by the colloidal probe technique, which result from simultaneous stretching and desorption of a large number of polymer strands, can be decomposed to the their single molecule contributions.

Imaging of single polymers

In order to study the adsorption of polymers, a number of techniques are available. The conformation of single polymers can be obtained by AFM imaging. Instead, reflectometry or ellipsometry allow determining the adsorbed mass. A number of well defined model substrates, for example on the base of silanes or thiols, can be prepared and characterized in our laboratory. By different approaches these substrates can be also structured chemically on the nanometer level. For the development of stimuli-responsive polymer layers we can combine AFM and electrochemistry.

Measurement of interaction forces on the single particle level

The interaction force between a pair of particles can be determined in the sphere–sphere geometry. For example, we could demonstrate the influence of surface roughness or polymer coverage on the interaction forces. Additionally, a direct comparison with electrophoretic mobility of the colloidal particles is possible.

**Polymers at interfaces – single molecule techniques**

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**SPECIAL ANALYTICAL TECHNIQUES AND COMPETENCES**

- Atomic Force Microscopy
  - Measurements liquids
  - Single molecule imaging
  - Chemical force microscopy
  - Detection of charge distributions
  - Measurement of elastic properties
- Direct Force Measurements
  - Single molecule force spectroscopy
  - Colloidal probe technique
  - Adhesion forces / mapping
  - Combination with electrochemistry
- Characterization of polymer layers
  - Ellipsometry
  - Reflectometry
  - Optical microscopy
  - Contact angle
- Electrophoretic techniques
  - electrophoretic mobility by light scattering
  - electrophoretic mobility by video microscopy
  - Streaming potential (currently in construction)
The “juniorprofessorship” for Polymer Systems focuses on periodic and amorphous functional nanostructures using a wide range of materials. Our predominant route towards well-defined nano- and mesostructured materials starts with the design of colloidal particles, which resemble the fundamental building blocks. These are being assembled into two- and three-dimensional arrays, which not only adds an additional length scale of structure, but also functionality. We have developed simple, yet robust techniques to assemble colloidal spheres into ordered monolayers. Of particular interest are the optical, mechanical, and thermal properties of latex particles, hollow spheres, and plasmonic nanoparticles.

Interface mediated self-assembly of monolayers

Monodisperse colloidal particles readily assemble into highly ordered nano- and mesostructured arrays – given the right assembly conditions. Colloidal monolayers are of particular interest since they provide a versatile source for defined polymer films on substrates, which can not be spin-coated. They can be used to modify the underlying substrate with sub 100 nm nanostructures and also adjust the optical properties of the support far instance with respect to its reflectivity.

We developed a simple, scalable technique, which is capable to produce highly ordered or amorphous colloidal monolayers on a cm² scale, which allows us to coat target substrates of almost any surface functionality and topography. This has been demonstrated for polymeric nanoparticles from 90 nm up to 1200 nm, but can also be transferred to ligand functionalized nanoparticles like magnetite or gold.

We further advanced this technique such that arbitrary patterns of different colloidal particles can be assembled and transferred without the need for any lithographic equipment. This is achieved by a combination of inkjet printing and interfacial assembly.

Colloidal Templating

Periodic colloidal arrays are often used as sacrificial template to gain access to nanostructures, which would otherwise be only accessible with expensive and sophisticated lithographic techniques. This methodology has been used in two- and three-dimensional systems in our group over the last years. For two-dimensional systems, we took advantage of our facile monolayer fabrication protocol, which allowed us to access large areas. Here, nanoparticle lithography was used to generate ordered arrays of silver or gold nanoparticles. Depending on the processing conditions, nanoparticles, dots, holes, or crescents could be obtained. Such structures can find application as sensors.

For three-dimensional templates a convective assembly method using a dip-coater was employed. The free space between the spheres can be filled with inorganic nanoparticles or with a precursor sol-gel solution. This leaves ample degrees of freedom to incorporate inorganic materials and to add a further length scale of structuring. Combustion of the polymeric components exposes porous metaloxide skeletons, which feature a hierarchy of characteristic length scale. These can be used to study diffusion processes within confined geometries or serve as matrix for future membranes or heterogeneous catalysts.

Hollow Spheres

Colloidal particles cannot only be used to template nanostructured two- or three-dimensional arrays. Each individual particle can serve as a scaffold itself. This for example leads to well defined hollow particles, with a shell consisting of silica or titania.

Such simple, yet versatile particles have been in our focus for the past years. On the one hand, they feature unique optical properties as can be seen in the figure below. The color impression originates directly from Mie resonance of the individual spheres due to suppressed multiple scattering in such low refractive index and amorphous powders.

On the other hand, the low effective density of such hollow objects renders them attractive candidates for future light weight composite materials, which could be fibers for clothing or structures for blast protection. Using our expertise in self-assembly, well-defined mono-, bi-, and tri-layer films of such hollow particles were formed and the collective behavior under compression was investigated. For this we employed nanoindentation. Since all structural parameters have been controlled from bottom up, theory and computer simulations could be used to describe our system.

Thermal Conductivity

The future focus of our research efforts will be laid on thermal transport properties in nano- and mesostructured materials. Well-defined colloidal assembly structures will be used as model platform to investigate the influence of interfaces and structural hierarchy on thermal conductivity.

**EQUIPMENT**

- Miniflecto PlasmaCleaner with integrated IR thermometer and Ar/O₂ gas connection
- Nabertherm L5/11/P330 high temperature furnace
- Bioforce Primo, Heraeus, benchtop centrifuge
- Dip coater
- XFA 500, Linseis, thermal conductivity measurements -50°C – 300°C, vacuum, inert gas, atmosphere, liquid cell and in-plane adapter
- Fully equipped wet-chemical laboratory

**FIELDS OF EXPERTISE AND EXPERIENCE**

- Monodisperse particle synthesis: latex, silica, core-shell
- Colloidal self-assembly: monolayers, colloidal crystals, hierarchical materials
- Plasma etching: surface modification, size reduction
- Microscopic methods: SEM, TEM, AFM
- Single particle spectroscopy
- Nanoindentation
Proteins mimicking the natural sequences. The recombinant genetic engineering to generate a set-up of recombinant spider silk. In our lab we apply biomolecular methods and the elastic flagelliform silk of the Golden Orb Web Spider (Nephila clavipes) and the tough and ductile biopolymers (toughness and ductility), biostatic properties and biocompatibility (no inflammatory or allergic responses). Our model systems are promising candidates for cellular matrix systems in 2D and 3D cell culture and Tissue Engineering, whereas films and non-woven fabrics can be used to functionalize surfaces or as stable membranes or filter systems. Microcapsules made of spider silk proteins are well-suited for encapsulation of macromolecules and can be used as carrier devices. Proteinaceous materials in general have a high potential as biomaterials due to the ease of processing into various morphologies, where the range of application can be easily widened by user-defined functionalizations.

Core competences

The core expertise of the Chair of Biomaterials is in the field of biopolymer research, in particular of structural proteins. Structural proteins can be modified to meet technically relevant product requirements. We have established biotechnological production routes for several structural proteins (including spider silk) as well as their processing into innovative technical and medical materials. Natural materials often exhibit optimized properties that outperform synthetic products. A major aim of our research is to gain insights into structure-function relationships of fibrous proteins by detailed characterization of molecular interactions and assembling mechanisms (including studies on self-assembly, influence of environmental factors, post-treatment, mechanical properties, etc.).

Production and characterization of recombinant silk proteins and processing into various materials

Human knowledge of spider silk for hunting, fishing and wound healing for long time due to its favorable mechanical properties (toughness and ductility), biostatic properties and biocompatibility (no inflammatory or allergic responses). Our model systems are the dragline silk of the European Garden Spider (Araneus diadematus) and the elastic flagelliform silk of the Golden Orb Web Spider (Nephila clavipes). In our lab we apply biomolecular methods and genetic engineering to generate a set-up of recombinant spider silk proteins mimicking the natural sequences. The recombinant production is performed in various suitable host systems like bacteria or yeast. The recombinant proteins are characterized regarding their physicochemical properties and assembly behavior to get insights into the natural assembly mechanism of spider silk. In addition, our group develops processing technologies to form non-natural morphologies such as films, hydrogels, non-woven fabrics, foams, particles and capsules for innovative new materials in technical and medical applications.

Modification and structural analysis of proteins

Our group has established various routes to modify proteins for customized needs, either by genetic engineering or by chemical approaches, e.g. site-specific ligation. Our technology allows tailoring specific properties such as biocompatibility, reactivity, coupling efficiency, and solubility to meet user-defined requirements. Moreover, we have expertise in generation and characterization of small peptides and hybrid biopolymers. Each material is analyzed regarding its chemical composition, purity and structural properties with state-of-the-art spectroscopic methods (CD, FT-IR, UV-Vis, Fluorescence) and microscopy (SEM, TEM, AFM) among others. Structural simulation and homology searches of unknown sequences further assist the characterization. Thus, we can identify molecular interactions between certain target domains of individual proteins, as well as their folding behavior and assembly processes.
The research activities within our group involve the study of nucleation and crystal growth phenomena of molecular systems from solutions and melts as well as early stages of phase transitions between different condensed phases. The synthesis and characterization of functionalized porous frameworks is of particular interest, with special emphasis on specific guest-host interactions – the foundation for exploiting substance specific recognition in gas separation processes or sensor design. Part of our research also includes the development of analytical strategies with the expressed purpose of a chemical differentiation of different guest molecules should be possible.

### Polymorphism and Nucleation

Many systems exist in numerous phases with identical chemical composition while maintaining individual structural arrangement as function of temperature and pressure. The individual polymorphs often exhibit markedly different physical and rheological properties as well as changes for the bioavailability. Being able to control the nucleation process in order to form the polymorph with the desired properties is a detailed understanding of the underlying mechanism is essential.

Many different subcritical nuclei with different structural arrangements can exist in the early stage of crystallisation. The size of such clusters will be small (on the order of a few nanometer); only a few subcritical nuclei survive, resulting in a limited amount of polymorphs which can be synthesised experimentally. However, stabilizing alternative nuclei would make it possible to prepare new polymorphs. As such, detailed information about the subcritical nuclei is mandatory in order to be able to control nucleation processes.

### Carbon Nitrides

2D carbon nitride frameworks exhibit interesting analogies to graphene based materials. However, the partial exchange of carbon by nitrogen leads to severe changes of their macroscopic properties. Layers of graphene are electrical conductors while CN frameworks form semiconductors with band gaps in the optical region. They are promising candidates for applications in photocatalytic reactions and optoelectronic devices. Nearly no structural information is available for this important class of materials due to the poor crystallinity of such systems.

### Porous Materials

Porous materials are ideally suited for applications as gas storage and separation. Metal-organic (MOF) and covalent organic (COP) frameworks allow one to tailor the size and shape of the pores as well as the wall interactions. This ensures a certain shape selectivity. We also work on introducing selectivity into the adsorption process by utilising chemical functionalization of the linker molecules either during the synthesis or by postsynthetic modification. By relying on supramolecular principles like the lock-key-interaction we believe a chemical differentiation of different guest molecules should be possible.

### Solid-State NMR Spectroscopy

Solid-state NMR spectroscopy allows for the detection of dynamical processes and structural properties for any NMR-active nuclei with the benefits of not having to destroy the sample. Relaxation techniques in conjunction with line shape analysis and 2D methods cover a dynamical range of 14 decades. High-resolution experiments enable the identification of characteristic building units, connectivities, and orientation correlations, allowing for experiments up to a few nanometer to be determined for both crystalline and amorphous materials. As such, solid-state NMR is ideally suited for the investigation of intermediate structure motives as well as the processes leading to their formation. The implementation of hyperpolarization techniques will further our understanding of surface specific properties and interactions – the accuracy of solid-state NMR on intermediate length scales is without alternatives.

### Carbon Nitrides

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A system with hysteresis is used, the unit is not only sensor and display but due to the pressure sensors with an easy optical readout with the naked eye. If a spin crossover why such compounds are considered for potential applications as temperature and/or time sensors. An external perturbation (e.g. change of temperature, pressure or light irradiation) initiates a spin transition from a high-spin to a low-spin state that is associated with change in the properties of the material (colour, magnetism, volume). For technical applications of such materials, it is essential to test their suitability for composite particle-matrix interactions can also influence the properties of the final composite material. This gives us the unique possibility to find a new approach for the control of the spin transition temperature – an important parameter that is difficult to control otherwise. For future applications the control of the transition temperature is of utmost importance.

Spin Crossover

Spin crossover (SCO) materials are a class of switchable molecules with a wide application potential in information technologies as sensors. An external perturbation (e.g. change of temperature, pressure or light irradiation) initiates a spin transition form a high-spin to a low-spin state that is associated with change in the properties of the material (colour, magnetism, volume). For technical applications of such materials, it is essential to test their suitability for composite materials and miniaturisation. That is why we investigate several strategies to achieve a mesostructuration of SCO nanoparticles. The aim of this work is the synthesis of nanoparticles that exhibit the same desired spin transition properties as the bulk material (e.g. hysteresis = memory effect or step wise spin transition). There exist different concepts to explain the appearance of hysteresis or steps during the spin transition. It is important to investigate, which of those concepts is suitable to explain such effects in small particles.

The spin crossover is often accompanied by a characteristic colour change. That is why such compounds are considered for potential applications as temperature and/or pressure sensors with an easy optical readout with the naked eye. If a spin crossover system with hysteresis is used, the unit is not only sensor and display but due to the memory effect a single incident can be memorised.

The research areas of the Weber group are focussed on the synthesis, characterisation and nanostructuration of molecule-based switches.

Synthesis of spin crossover nanoparticles

One possibility for the preparation of spin crossover nanoparticles is the inverse-micelle technique (water in oil emulsion). The micelles are the nanoreactors for the synthesis of the SCO compound and the final particle size can be controlled by the size of the micelle.

Our interest lies not only in the influence of particle size and morphology on the spin crossover properties. Particle-particle and particle-matrix interactions can also influence the properties of the final composite material. This gives us the unique possibility to find a new approach for the control of the spin transition temperature – an important parameter that is difficult to control otherwise. For future applications the control of the transition temperature is of utmost importance.

Self-assembly of amphiphilic complexes

The synthesis of amphiphilic compounds opens new possibilities for the mesostructuration of complexes. The formation of different superlattices (lamellar or micellar) is possible. In the research group Weber we did develop amphiphilic ligands that are suitable for the synthesis of spin crossover complexes. With the help of additionally introduced alkyl chains the self-assembly of the complexes in the solid can be influenced and in part even dominated. The main structure motive observed so far are lipid layer-like structures.

The formation of lamellar or micellar structures in solution is imaginable and it should be possible to control the structure by variation of the alkyl chains. Experiments with copper complexes of this ligand type show that self-assembly processes in solution take place. In some cases the formation of thixotropic gels was observed. For the characterisation of the magnetic properties of the complexes and their nanoparticles we use a SQUID magnetometer. The sensitivity of this device is high enough to analyse extremely small sample amounts or, with respect to the magnetic properties, strongly diluted samples. The nanoparticles and the self-assembled structures of the amphiphilic complexes are characterised by transmission electron microscopy and light scattering.

Syntheses and Characterisations

- Synthesis of responsive spin crossover complexes
- Amphiphilic responsive complexes
- Work with air sensitive materials
- Ligand design
- New ligands for spin crossover complexes
- Ligands for multifunctional complexes
- Inorganic Nanoparticles
- Inverse-micelle technique
- Introduction of complexes into polymers
- Characterisation
  - Magnetism (SQUID magnetometer)
  - Mössbauer spectroscopy
  - NMR spectroscopy of complexes
  - X-ray structure analysis
  - UV-vis spectroscopy

Exertises

- Magnetism
- Coordination Chemistry
- Molecular Switches
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