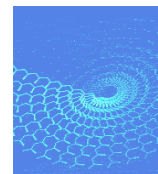




SCHWEIZ. CHEMISCHE GESELLSCHAFT SCG  
SOCIETE SUISSE DE CHIMIE SSC  
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Division Polymere und Kolloide  
Division des Polymères et Colloïdes  
Polymers and Colloids Division

DPK  
DPC  
PCD



# PolyColl 2008

Fribourg, June 13, 2008

- 9.00-9.30**      **Registration and Coffee**
- 9.30-9.35**      **Dr. Bettina Steinmann** *President Polymers and Colloids Division*  
Welcome Note
- 9.35-10.15**    **Prof. Olli Ikkala** *Helsinki University of Technology, Finland*  
Conformational and Self-Assembly Control in Polypeptide Complexes
- 10.15-10.50**   **Dr. Jens Rieger** *BASF, Germany*  
Nanotechnology and Colloids at BASF
- 10.50-11.05**   **Coffee Break**
- 11.05-11.40**   **Prof. Giovanni Dietler** *EPFL, Switzerland*  
Polymer Physics from Single Molecule DNA Images
- 11.40-12.15**   **Prof. Afang Zhang** *ETHZ, Switzerland*  
Helical Dendronized Polymers
- 12.15-13.30**   **Lunch, Poster Session**
- 13.30-14.10**   **Prof. Henk Lekkerkerker** *Utrecht University, Netherlands*  
Gelation versus Liquid Crystal Phase Transitions in Suspensions of Charged Colloidal Platelets
- 14.10-14.45**   **Dr. Laurent Sagalowicz** *Nestlé Research Center, Switzerland*  
Liquid Crystalline Phases in Food: From Basic Science to Applications
- 14.45-15.00**   **Coffee Break**
- 15.00-15.35**   **Dr. Véronique Trappe** *University of Fribourg, Switzerland*  
Relaxations in Squeezed Colloidal Systems
- 15.35-16.10**   **Dr. Andreas Mühlebach** *Ciba Specialty Chemicals, Switzerland*  
Specialty Polymers made by Controlled/Living Radical Polymerization (CRP) and their Use as Dispersants for Pigments
- 16.15-16.45**   **General Assembly of the Polymers and Colloids Division**
1. Annual Report of the President
  2. Financial Report 2007
  3. Release of the Members of the Board
  4. Budget 2008
  5. Activities 2008
  6. Discussion of Proposals of the Members
  7. Various
- 16.40**      **Aperitif**

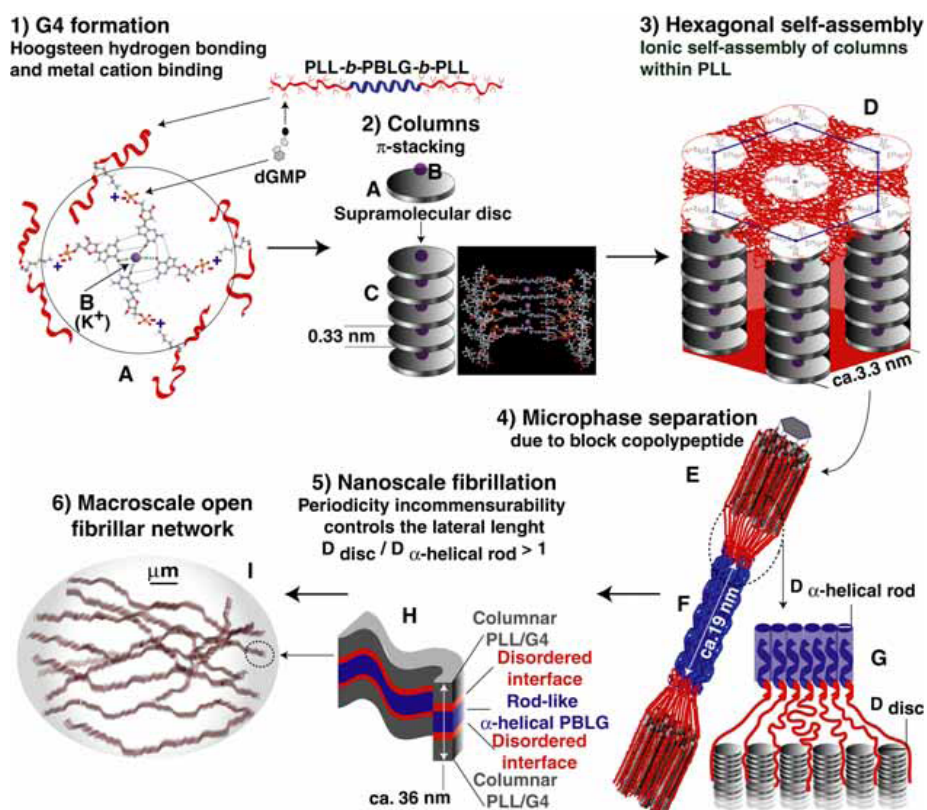
# ABSTRACTS

## LECTURES

### Conformational and Self-Assembly Control in Polypeptide Complexes

Olli Ikkala, Laboratory of Molecular Materials, Department of Engineering Physics and Center for New Materials, Helsinki University of Technology, P.O. Box 5100, FIN-02015 TKK, Espoo, Finland, Olli.Ikkala@tkk.fi

Competing interactions allow self-assembly and combining structural units capable of self-assembly at different length scales leads to hierarchical structures and control of functionalities. A facile route deals supramolecular complexes of polymers or block copolymers and oligomeric surfactants that allow hierarchical self-assemblies at the length scale of polymers and oligomers (see our previous works, eg. *Science* 1998, 2002; *Nature Materials* 2004). Besides self-assembly, complexation of surfactants allows to control the conformation of the polymer backbone. This becomes particularly feasible, taken that the backbone is a polypeptide. We describe the recent work where surfactant complexation in bulk allows to control the secondary structure as well as the self-assembly (*Biomacromolecules* 2006, 2007; *Macromolecules* 2008). A powerful concept for structural multilevel hierarchies is based on complexing DNA monophosphates in block copolypeptides, where we demonstrate six-level hierarchies (*Adv Funct Mat* 2008). We expect that the shown routes open new scenarios to tailor biological polymers even for new nonbiological functional applications.



**Figure.** A route towards multilevel hierarchies by complexing 2'-deoxyguanosine 5'-monophosphate and polylysine-*block*-polybenzylglutamate-*block*-polylysine. This leads to formation G-quartet (G4) supramolecular disks. As polybenzylglutamates form helical rod-like mesogens, the combination of discotic and calamitic liquid crystalline moieties, block copolyptide microphase separation, and packing frustration lead to supramolecular fibrillation (Houbenov, Nykänen, Iatrou, Hadjichristidis, Ruokolainen, Faul, Ikkala, *Adv Funct Mat*, 2008, in press). We foresee that they allow functional nanofibers, as different metal cations can be incorporated within the G4 cores.

# Nanotechnology and Colloids at BASF

Jens Rieger

BASF Aktiengesellschaft  
Polymer Physics, Ludwigshafen, Germany and  
BASF Advanced Research Initiative at Harvard, Cambridge, USA

BASF is one of the leading companies in the field of chemical nanotechnology. The company is using nanotechnology to improve the existing product portfolio, to create new products and further develop existing ones. BASF comprises all the expertise necessary to benefit from the full potential of nanotechnology: from special analytical methods and expertise in chemistry, physics and biology and the engineering sciences through to the safe handling of new materials. Examples from various fields of activities will be discussed – with emphasis on their colloidal aspects.

As a specific – and scientifically highly interesting – example for nanotechnology/colloid science we will discuss precursor nano-structures occurring in crystallization processes in more detail: Crystallization of inorganic (and organic) matter often proceeds via intermediate stages – rather than by simple nucleation and growth mechanisms. These precursor stages not only comprise crystal modifications that are less stable than the final one (Ostwald's rule of stages), but also amorphous, hydrated Nan particles and emulsion-like precursors have been observed. Recent data on the structural evolution of precipitating  $\text{CaCO}_3$  obtained by means of X-ray microscopy and quench cryo-transmission electron microscopy will be presented, emphasizing that the respective particle formation processes do not follow classical nucleation and growth mechanisms. It will be outlined how these processes can be controlled by means of polyelectrolytes.

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## Polymer Physics from Single Molecule DNA Images

Giovanni Dietler

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DNA molecules were imaged using Atomic Force Microscopy (AFM). After tracing the trajectory of the molecules, it is possible to calculate different statistical quantities like the radius of gyration, the end-to-end distance, the distribution of the end-to-end distance and the asphericity. These quantities in turn permit to determine the critical exponents describing the divergence of the radius of gyration with the length of the DNA molecule or the distributions. For the first time it was possible to compare the experimental distributions with the theoretical one. Additionally, because of the fact that DNA has a rather large persistence length (50 nm), theories for semi-flexible polymers could be tested. The above experiments could be carried out with topologically constrained DNA molecules: linear, circular, supercoiled and knotted DNA molecules were imaged and analyzed.

# Helical Dendronized Polymers

Afang Zhang

Institute of Polymer, Department of Materials, ETH Zurich. Wolfgang-Pauli-Str. 10, HCI G5252, 8093 Zurich

The first and second generation dendronized polymethacrylates **PG1** and **PG2** carrying chiral 4-aminoproline-based dendrons were obtained on the half-gram scale in high molar masses (**PG1**:  $M_n = 5 \times 10^6$  g/mol, **PG2**:  $M_n = 1 \times 10^6$  g/mol) by spontaneous (radical) polymerization of the corresponding vinyl macromonomers. NMR spectroscopic studies on **PG2** together with its unprecedented high glass transition temperature ( $T_g > 200$  °C, decomposition) and structural parameters provided by atomistic MD simulations showed this polymer to be rather rigid. Optical rotation and CD measurements revealed that **PG2** attains a helical conformation which remains unchanged in a wide temperature and solvents polarity range. Impressingly, it also remained so after the polymer was deprotected (and thus positively charged, *de*-**PG2**) at its terminal amines, by which the mass and steric demand of the dendrons is reduced by roughly 50%. MD simulations on models of **PG2** revealed its helical conformation to be right-handed, irrespective of backbone tacticity, and initial results also indicated that *de*-**PG2** retains the right-handedness. For comparison, the first and second generation dendronized polymethacrylates **PG1** and **PG2** carrying chiral lysine-based dendrons were prepared, and found this less compact **PG2** also adopted helical conformation.

## Gelation versus Liquid Crystal Phase Transitions in Suspensions of Charged Colloidal Platelets

H.N.W. Lekkerkerker, Van 't Hoff Laboratory for Physical and Colloid Chemistry, Debye Institute Utrecht University, Padualaan 8, 3584 CH Utrecht, The Netherlands

The equilibrium and non-equilibrium phases of suspensions of charged gibbsite (Al(OH)<sub>3</sub>) platelets are investigated. We study the competition between liquid crystal formation, gelation/glass formation and sedimentation at different colloid concentrations and salt strengths. By variation of the ionic strength we are able to tune the effective thickness-to-diameter ratio of the platelets in suspension and the balance between attractive and repulsive interactions. We find an isotropic (I) nematic (N) and columnar (C) phase wedged between an attractive gel and a repulsive gel/glass. A model for the repulsive glass is proposed. Results of columnar liquid crystals of gibbsite platelets as templates for the generation of ordered silica structures are presented

## Relaxations in squeezed colloidal systems

David Sessoms, Irmgard Bischofberger, Joaquim Clara Rahola, Véronique Trappe

*Department of Physics, University of Fribourg, Chemin du Musée 3, Fribourg, Switzerland*

Strongly quenched colloidal systems, such as glasses, gels, foams and compressed emulsions exhibit residual dynamics, which are characterized by strong spatial and temporal heterogeneities. We investigate these heterogeneities using space and time resolved correlation schemes aiming to characterize the hallmarks of stress- and temperature-driven restructuring processes. In this talk we will discuss recent results obtained with microgels for which the volume and thus packing state can be conveniently varied via temperature.

# Liquid Crystalline Phases in Food: From Basic Science to Applications

L. Sagalowicz

Nestlé Research Center, Vers-Chez-Les-Blanc, CH-1000 Lausanne 26, Switzerland

E-mail: Laurent.Sagalowicz@rdls.nestle.com

The present challenge of the food industry is to deliver nutrition-health benefits by keeping, and possibly improving the taste and aroma impact. Adding individual molecules is most of the time not possible or not sufficient to achieve the desired goal, due to the fact that many interesting components are only hardly soluble in aqueous systems and macroscopic properties, such as release, depend on the food structure. Therefore, a promising possibility to increase the functionality of nutrients or flavour compounds in foods lays in the systematic control of structure formation. One way to achieve this is to use the intrinsic abilities of food ingredients to form self assembly and liquid crystalline structures. For example, amphiphilic molecules, such as monoglycerides, phospholipids, etc. form spontaneously liquid crystalline phases, when they are present in an aqueous and/or oleic environment (Figure 1). These liquid crystalline structures incorporate (solubilize) spontaneously hydrophilic and/or lipophilic molecules of various sizes, molecular structure and polarity depending on their structural organisation. This makes them ideal carrier systems for mixture of nutrients and flavours [1].

In the present contribution, the parameters which dictate the formation of liquid crystalline phases and recent analytical techniques to study them will be described [2,3]. The functionality of various self-assembly structures will also be compared. In particular, it will be shown how such delivery systems can be used for molecule solubilisation and release[1,4].

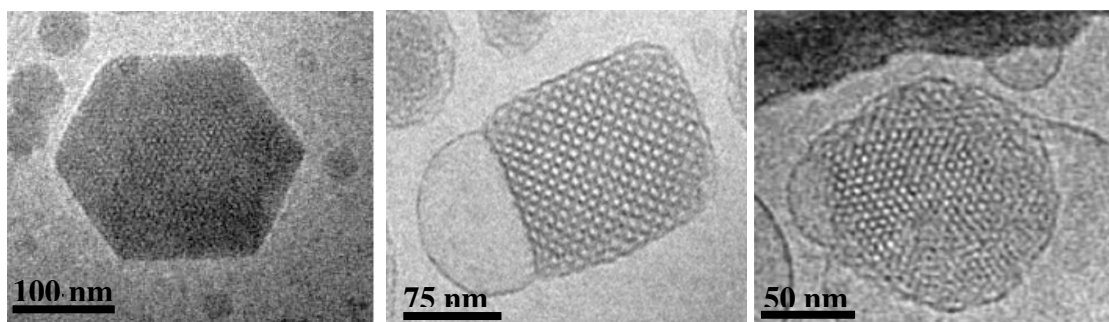


Figure 1: Particles with different internal self-assembly structures, (left) reversed hexagonal, (center) reversed bicontinuous cubic with a space group  $Im\bar{3}m$  and (right) reversed bicontinuous cubic phase with a space group  $Pn\bar{3}m$ .

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# Specialty Polymers made by Controlled/Living Radical Polymerization (CRP) and their Use as Dispersants for Pigments

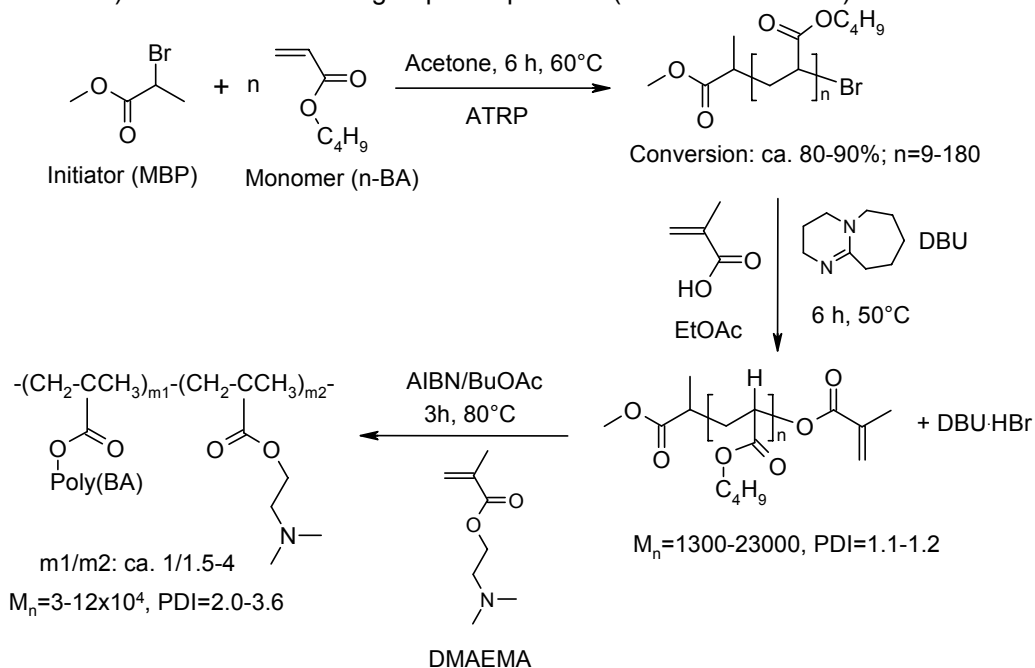
Dr. Andreas Mühlebach

Ciba, Group Research, CH-4002 Basel, Switzerland

Polymeric dispersants are essential for the formulation of high solids and waterborne pigmented coatings. Whereas commercially available dispersants for organic pigments are based on polymer mixtures with broad polydispersity, new technologies for controlled/living radical polymerization (CRP) allow the synthesis of tailor-made, narrow dispersed homo- and block-copolymers of different architecture and therefore play a key role for the future development of improved pigment dispersants.

In contrast to the rather sophisticated "older" living polymerization techniques like anionic polymerization or group transfer polymerization (GTP) with stringent requirements for the purity of the raw materials and limited choice of solvents, the new CRP techniques (nitroxide-mediated controlled radical polymerization (NMP), atom-transfer-radical polymerization (ATRP) and reversible addition-fragmentation-transfer (RAFT)) are performed in a variety of solvents (including water!) and are tolerant to almost any functional groups and impurities.

ATRP is one of the most versatile of the new CRP techniques, because of the broad range of monomers and initiators, the mild reaction conditions and the fast and easily adjustable polymerization rate. In addition, specific transformations of the halogen endgroups, e.g. into alkoxyamines (NOR's), acrylates (macro-monomers) and other functional groups are possible (see scheme below).



The presentation will describe the synthesis of well-defined block-copolymers, macro-monomers and comb-copolymers by NMP and ATRP. In addition, it will be shown how structural parameters like chemical composition, block length and molecular weight influence the dispersant performance.

Block- and comb-copolymers based e.g. on poly(*n*-butyl acrylate) as "steric stabilizer" and poly(*N,N*-dimethyl-aminoethyl-(meth)acrylate) as pigment affinic "anchoring" groups are a new class of highly efficient dispersants for solvent (and now also water) borne systems, recently commercialized by Ciba (EFKA 4300 series). High gloss of the coating combined with low viscosity of the millbase is reached at much lower dispersant concentration than with competitor products.

## POSTERS

### Self-assembly of rod-coil block copolymers from weakly to moderately segregated regimes

**Authors:** Nicolas Sary<sup>1</sup>, Cyril Brochon<sup>2</sup>, Georges Hadziioannou<sup>2</sup>, Raffaele Mezzenga<sup>1</sup>

<sup>1</sup>Department of Physics and Fribourg Center for Nanomaterials, University of Fribourg, Ch. Musée 3, CH-1700, Fribourg, Switzerland

<sup>2</sup>Laboratoire d'Ingénierie des Polymères pour les Hautes Technologies, UMR 7165, Université Louis Pasteur, Ecole Européenne de Chimie, Polymères et Matériaux, 25, Rue Becquerel, 67087 Strasbourg, France

We report on the self-assembly behaviour of two homologue series of rod-coil block copolymers in which, the rod, a p-conjugated polymer, is maintained fixed in size and chemical structure, while the coil is allowed to vary both in molecular weight and chemical nature. This allows maintaining constant the liquid crystalline interactions, expressed by Maier-Saupe interactions,  $w$ , while varying the tendency towards microphase separation, expressed by the product between the Flory-Huggins parameter and the total polymerization degree,  $xN$ . Therefore, the systems presented here allow testing directly some of the theoretical predictions for the self-assembly of rod-coil block copolymers in weakly segregated regime. The two rod-coil block copolymer systems investigated, were poly(DEH-p-phenylenevinylene-b-styrene), whose self-assembly takes place in the very weakly segregated regime, and (DEH-p-phenylenevinylene-b-4vinylpyridine), for which self-assembly behaviour happens under increased tendency towards microphase separation, hereby referred as moderately segregated regime. Experimental results for both systems are compared with predictions based on Landau expansion theories.

### Structure-Properties Relationship in Proton Conductive Sulfonated Polystyrene-Polymethyl Methacrylate Block Copolymers (sPS-PMMA)

Chaoxu Li<sup>1</sup>, Laurent Rubatat<sup>1,2</sup>, Hervé Dietch<sup>1</sup>, Janne Ruokolainen<sup>3</sup> and Raffaele Mezzenga<sup>1,4,\*</sup>

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<sup>3</sup> Helsinki University of Technology, Espoo, Finland

<sup>4</sup> Nestlé Research Center, Vers-Chez-Les-Blanc, 1000 Lausanne 26, Switzerland

We report on the dependence of proton conductivity on the morphologies of sulfonated polystyrene-poly(methyl-methacrylate) (sPS-PMMA) diblock copolymers. Three different diblock copolymers of varying molecular weight and block volume fraction were studied, and for each one several sulfonation rates of the PS block were considered. The investigation of the morphologies of the self-assembled sPS-PMMA diblocks was carried out in both dry samples and samples saturated with water, by means of small angle neutron scattering (SANS), transmission electron microscopy (TEM) and cryoTEM. Depending on molecular weight and sulfonation rates, both isotropic phase (ISO), lamellar phases (LAM) and hexagonally perforated lamellae (HPL) were observed. The lamellar morphologies underwent marked volume expansion upon water pick up, while negligible swelling was detected for the HPL morphologies. Proton conductivity was measured in both the dry and wet states, the latter conditions resulting in an enhancement of conductivity up to three orders of magnitude. In particular it was shown that the conductivity, normalized by the volume fraction of the conductive domains (formed by PS, sPS and water), rises monotonically with the content of sulfonic groups, and that discontinuities in conductivities occur systematically when the structure of the block copolymer changes from ISO to HPL to LAM.

# Ionic complexes based on dendronized polymers: towards ultra-nanoporous organic membranes

Nadia Canilho,<sup>a</sup> Edis Kasëmi,<sup>b</sup> Dieter Schlüter,<sup>b</sup> Raffaele Mezzenga<sup>a,\*</sup>

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<sup>b</sup> Swiss Federal Institute of Technology ETH-Zurich, HCI J 541, CH-8093 Zurich, Switzerland

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Supramolecular chemistry is a biologically-inspired process widely used by more than twenty years to easily tune macromolecules by attaching side chains onto the periphery of the macromolecular template via non-covalent intermolecular forces such as hydrogen bonding<sup>1,2</sup>, metal coordination<sup>3</sup>, or ionic interactions<sup>4,5</sup>.

These routes offer the opportunity to build reversible polymer architectures with stimuli responsive features such as photo-induced isomerization, pH-responsiveness, etc.

To this end, we have investigated the phase diagram of supramolecular ionic complexes of sulfate alkyl tail surfactants and cationic dendronized polymers of varying generations (DPG<sub>x</sub> with  $x = 1,2,3$ )<sup>6</sup>. As a consequence of the chemical diversity of polymer backbone and side chain surfactants, microphase segregation takes place leading separate to various LC phases with a well-defined molecular packing. Transmission electron microscopy (TEM) and small angle X-ray scattering (SAXS) techniques were used to demonstrate columnar packing into hexagonal, rectangular and tetragonal lattices or in lamellar arrangement depending on the dendron generation and on the alkyl tail length, with typical lattice parameters ranging between of 3 to 5 nm. In particular, TEM analysis revealed for all the columnar phases a molecular packing consistent with the alkyl tails confined within the cylinders.<sup>7</sup> Given the lattice period and the reversibility of the supramolecular routes used to drive the self-assembly, the present result open news doors to the development of ultra-nanoporous organic materials.

## Self-Organization on Multiple Length Scales in “Hairy Rod”-Coil Block Copolymer Supramolecular Complexes

Matthew R. Hammond, Harm-Anton Klok, Raffaele Mezzenga  
University of Fribourg, Chemin du Musée 3, CH-1700 Fribourg, Switzerland

A peptide-synthetic hybrid block copolymer, poly(ethylene oxide)-block-poly(L-glutamic acid), is shown to form supramolecular complexes with primary alkylamines of varying alkyl chain lengths (8 to 18 methylene units) in organic solvents via acid-base proton transfer and subsequent ionic bonding. The peptidic block being in the  $\alpha$ -helical conformation, these materials behave as coil-“hairy rod” block copolymers, and show hierarchically selforganized nanostructures in the solid state. X-ray scattering measurements show mesomorphic behavior at the length scales of both the overall block copolymer and the polypeptide-alkylammonium complex.

# Synthesis, Self-Assembly Behaviour and Supercritical Carbon Dioxide Polymerization of Dendronized Polymers

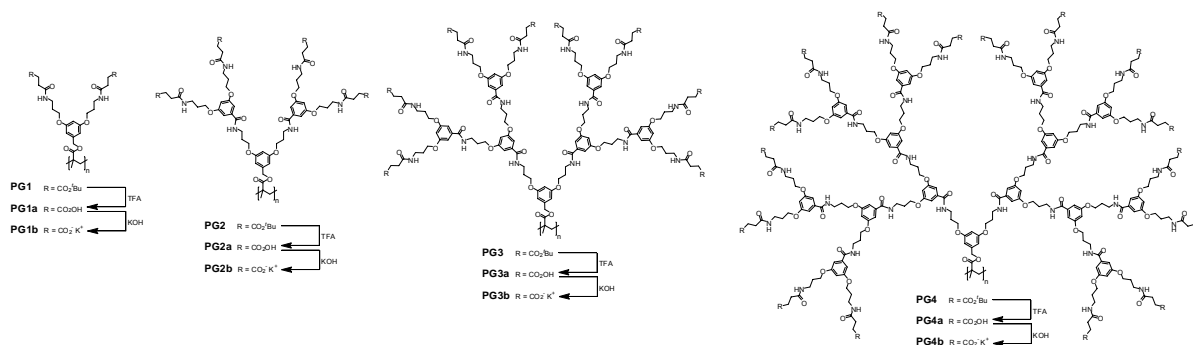
E. Kasëmi<sup>a</sup>, W. Zhuang<sup>b</sup>, I. Costa<sup>c</sup>, G. Storti<sup>c</sup>, M. Morbidelli<sup>c</sup>, J. P. Rabe<sup>b</sup>, P. Walde<sup>a</sup> and A. D. Schlüter<sup>a</sup>

<sup>a</sup>Department of Materials, ETH-Zürich, HCI J541, Wolfgang-Pauli-Str. 10, CH-8093, Switzerland.

<sup>b</sup>Department of Physics, Humboldt University of Berlin, Newtonstrasse 15, D-12489 Berlin, Germany.

<sup>c</sup>Department of Chemistry and Applied Bioscience, ETH-Zürich, HCI F129, CH-8093, Switzerland.

Dendronized polymers (denpols) are hybrids of dendrimers and polymers. They belong to a class of comb polymers in which regularly branched units (dendrons) with different numbers of branch points (generations) are attached to a linear backbone at every repeat unit.<sup>[1]</sup> Due to their shape-anisotropic nature, these polymers exhibit interesting application options, which include hierarchical structure formation through self-assembly in bulk<sup>[2]</sup>, in solution<sup>[3]</sup> and on surfaces<sup>[4]</sup>, as well as molecular constructions on the single molecule level.<sup>[5]</sup>



Here we describe a practical and step-efficient synthesis of **G1-G5** macromonomers and their respective free radical polymerization to anionically chargeable **PG1-PG4** dendronized polymer.<sup>[6]</sup> SFM investigations of individualized denpols both with neutral (**PG2**, **PG3**) and negatively charged peripheral groups (**PG2b**, **PG3b**), respectively, reveal duplex superstructures for the latter based on contour length analysis of these charged and non-charged species.<sup>[7]</sup>

The synthesis of denpols according to the macromonomer route has the intrinsic disadvantage that the average chain lengths drop drastically with increasing dendron generations. In an attempt to overcome this critical limitation, a G3 dendritic macromonomer was polymerized in supercritical carbon dioxide (scCO<sub>2</sub>). Molar mass determinations with GPC show average molecular weights on the order of several millions which are higher by a factor of roughly 5 than under conventional free radical conditions. The denpols obtained were checked by SFM for their structural integrity and mostly linear chains were observed.<sup>[8]</sup> This finding may mark a breakthrough for the synthesis of denpols.

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# Polymers Going Laterally Infinite and Periodically Networked: Challenges to 2D Polymer Synthesis

P. Kissel, J. van Heijst, O. Lukin, J. Sakamoto and A. D. Schlüter  
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ETH Zürich, HCI J 541, Wolfgang-Pauli-Str. 10, CH-8093, Switzerland  
(Contact: [schlueter@mat.ethz.ch](mailto:schlueter@mat.ethz.ch); [sakamoto@mat.ethz.ch](mailto:sakamoto@mat.ethz.ch))

Organic synthesis of 2D polymers remains an uncharted territory in science with great potential. We are currently trying to tackle this challenge [1,2]. "2D polymer" in this context means a covalent macromolecular network being not only laterally infinite and one monomer unit thick, but also ideally possessing a periodical long-range order as seen in graphene, which is a well-known representative from Nature. Our main strategy is based on rational monomer design to allow the monomers to grow only in lateral directions concurrently forming a periodical network. Several anthracene-based cyclic monomers have been designed accordingly, where the anthrylene units are embedded in the molecular skeleton at defined positions and in fixed orientations [3]. By exploiting photo-induced [4+4] cycloaddition among the anthrylene units, these monomers are expected to polymerize in a "programmed" manner towards 2D polymers (Figure 1). The concept of this "programmed" 2D polymerization, some preliminary results from monomer synthesis as well as those from model compounds will be presented.

**Figure 1.** "Programmed" 2D Polymerization

[1] J. Sakamoto; J. van Heijst; O. Lukin; A. D. Schlüter, review submitted. [2] A. Zhang; J. Sakamoto; A. D. Schlüter, *Chimia*, accepted. [3] P. Kissel; J. Sakamoto; A. D. Schlüter, manuscript in preparation.

## Polymerization of lactic acid

*Fabio Codari, Davide Moscatelli, Giuseppe Storti, and Massimo Morbidelli*  
*Institute for Chemical and Bioengineering, ETH Zurich, 8093 Zurich, Switzerland*

**Abstract** High molecular weight poly(lactic acid) [PLA], one of the emerging biodegradable polymers, is commercially produced by the ring opening polymerization of lactide, the cyclic dimer of lactic acid. However, due to the complexity involved in the process, PLA is not yet able to serve as a commodity plastic. On the other hand, the simpler direct polycondensation of lactic acid usually results in low molecular weight products due to difficulties in the removal of the by-product (water), which introduces reversibility in the reaction. Though many experimental works are reported in the literature for the polycondensation of lactic acid, there are no efforts provided for the mathematical modeling of the process.

In the present work, we develop a model based on very simple kinetic scheme, standard for reversible polycondensation, and accounting for water removal by diffusion. Several reaction experiments have been carried out under different sets of reaction conditions, with and without catalyst, in open and closed system. In the first case, water and polymer remain in the reactor until complete equilibration. In the second one, large inert flow and/or depressurization are applied to remove water. Such experimental results are used for model validation and parameter evaluation. The developed model is able to correctly predict the time evolution of average polymer properties. However, the agreement between model and experiments is less satisfactory when examining the complete molecular weight distribution of the polymer, in particular at large temperature. The experimental work is currently going on aimed to deepen the analysis of possible side reactions.

Finally, parametric simulations are carried out to analyze the effects of operating conditions such as reaction temperature, catalyst amount and type, and reaction pressure, aimed to design improved reaction paths for high molecular weight materials.

# A STUDY OF THE EFFECT OF COUNTERION ASSOCIATIONS ON COLLOIDAL STABILITY

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The methodology of a generalized model [1] for colloidal stability has been validated against experimentally measured values of the stability ratio and critical coagulation concentrations (CCC), for electrolytes with mono or divalent cation, i.e., potassium chloride and magnesium chloride, respectively. Besides the classical DLVO theory the generalized model accounts for the interplay between colloidal interactions and the association of cations with the particles surface charge groups. The model parameters are obtained or estimated purely based on information available in the literature. For the monovalent salt the predictions agree well with the experimental data from literature [2], forecasting both the CCC values and stability ratios quantitatively. For the divalent salt the predictions for large values of the stability ratio tend to deviate from the experimental results, but it is noted that the onset of stability, i.e., the CCC, and small stability ratios are correctly forecast. Moreover, a comparison of the above results with those neglecting the effect of counterion association with the particles surface charge groups indicates that the latter substantially overestimates stability ratios in the presence of high salt concentrations, in the case of the monovalent salt, and leads to unrealistic large values of the CCC for the divalent salt. Including the association of cations with the particles surface charge groups can explain the relative low values of experimental CCC for divalent salts compared to the theoretical predictions by the classical DLVO theory neglecting ion association, which is a point of interest in solid-liquid separation processes.

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## Determination of Diffusion Coefficients of Migrants in Polymer Packaging Materials

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Mass flow curves of linear hydrocarbon molecules and aroma molecules (limonene and menthone) across polymer membranes (LDPE, HDPE, PP) have been measured as a function of time by mass spectrometry. A special cell has been build for this purpose. Fitting the normalized mass flow data with the appropriate solution of the diffusion equation allows for the determination of the diffusion coefficient. They are in the order of magnitude of  $10^{-8} \text{cm}^2 \text{s}^{-1}$  and do correspond to values found in the literature. The method permits the simultaneous determination of the diffusion coefficients of various migrants.

# Scattering properties of dense clusters of nanoparticles

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## Abstract

In sheared induced coagulation processes of colloidal nanoparticles, which are customarily used in most industrial processes, dense clusters of particles are usually formed. The characterization of their size, structure and distribution is a key factor for the quality of the final product. Light scattering or laser diffraction techniques are still the most commonly used techniques to perform this characterization, because of their speed, excellent statistics and great simplicity. However, a major obstacle in the effective utilization of this technique is the lack of realistic structural models for dense clusters of spheres which allow one to extract reliable information from scattering data. In addition, the commonly used Rayleigh-Debye-Gans (RDG) theory fails in the case of dense structures and particles with a size comparable to that of the wavelength of the incident radiation. These conditions are the rule rather than the exception in many coagulation processes.

In this work, a methodology is presented that aims at filling this gap. First of all, a tunable fractal dimension Monte-Carlo algorithm is used to generate dense clusters with a desired fractal dimension. Since it is well known that the tunable fractal dimension algorithm cannot generate clusters with fractal dimension larger than 2.5, a new procedure has been developed to create cluster with fractal dimension up to 3. This procedure starts from clusters with a fractal dimension equal to 2.5 and uses a Voronoi tassellation of the space occupied by the cluster, which is made progressively denser by moving particles initially located on its surface to its empty holes. In this manner, the entire range of cluster fractal dimension encountered in typical aggregation processes, ranging from 1.8 to 3, is covered. The cluster structure is then characterized by means of its pair correlation function. The pair correlation function is finally used to compute scattering properties of cluster used a mean-field version of the T-Matrix theory, proposed by Botet et al., which can provide reliable scattering behavior of dense clusters with arbitrary primary particle size. The predictions of the mean field T-matrix theory are compared to RDG theory predictions, to show the difference between these two approaches. In addition, the application of these results to the analysis of shear-induced coagulation experiments of polymer colloids is discussed.

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# Antifreeze Coatings for Rotor Blades of Wind Turbines

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The sun is our largest energy source. About 2% of the energy of the solar radiation is transformed into wind. Theoretically, wind energy could therefore more than satisfy world's energy needs. In order to fully utilize the existing wind potential in Europe, wind turbines have to be erected at sites that are endangered to icing. Yet, icing is fatal for wind turbines, as ice accumulation at the leading edge of the rotor blades alters their profile and finally impedes energy production altogether. An elegant solution for the prevention of ice accumulation would be a coating that allows a freezing-point depression. We are currently investigating the freezing-point depression of various synthetic polymers on glass substrates. We finally succeeded in preparing such a surface which shows a freezing point depression of about 10°C, compared to uncoated glass. Because the melting point of water is not affected, the above surface displays "thermal hysteresis", an effect known from antifreeze proteins of arctic fish.

## Aerogels as Thermal Insulators

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Aerogels have an extremely high porosity of up to 99%. Their thermal conductivity, which is in the range of 16-21mW/(m·K), is the lowest known for solid materials at ambient pressure and temperature. This makes aerogels desirable for thermal insulation. There is a large potential for the use of aerogel materials in building renovation. The first step of aerogel preparation is the formation of a three dimensional SiO<sub>2</sub>-network in a solvent. For extracting the pore fluid at ambient conditions the gel needs to be made hydrophobic. After the removal of the pore fluid a SiO<sub>2</sub>-skeleton remains, the aerogel. The aerogels were characterised by Archimedes Principle giving information about their bulk density, which is about 0.14g/cc. Helium pycnometry was used to measure the skeleton densities of about 1.77g/cc. The porosity could then be calculated yielding 92%. The thermal conductivity was measured by EMPA giving 16-18mW/(m·K). We studied the crucial process parameters for the preparation of aerogels of high porosity and low thermal conductivity. We developed a new ambient pressure drying process, which allows to work at basic conditions and with it the reinforcement with acid sensitive fibres.

## Coatings with Lime Repellent Properties

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Calcification, or scaling, is a problem known to everyone. Thereby, sparingly soluble calcium carbonate precipitates and adheres to surfaces, forming crusts of lime that are difficult to remove. Scaling can be avoided by water softening, and the latter can be achieved by, for example, ion exchange. A different approach to avoid scaling is to inhibit the adhesion of lime at surfaces. Since little is known about how lime binds to a material, we have been investigating various substrates with respect to their ability to repel calcification – with no success. It appeared to us that a more promising approach could be the development of hydrophilic and water soluble polymer coatings. A promising candidate to obtain hydrophilic coatings is poly(ethylene glycol) (PEG). After usual sol-gel reactions, a coating of PEG-silane on glass can be realized. Such coatings efficiently inhibit the adhesion of lime at surfaces.

# NUCLEOTIDE-BASED AMPHIPHILIC DIBLOCK COPOLYMERS: HYBRIDIZATION STUDIES ONTO SURFACES

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Shared expectations in gene therapy to cure genetically acquired diseases hold the attention and drives major research efforts to establish the human genomic map for instance.

One of the greatest tool-box developed towards this end is the DNA microarray or DNA-chip. This technology is on the market for the last decade. However there is still a need of understanding the process of hybridization onto surfaces, which currently limits the design, efficacy and analysis of DNA microarrays. A theoretical background has been addressed very recently<sup>1</sup>. Nevertheless experimental investigations are still missing.

We report here on the experimental study of hybridization onto surfaces using both conventional oligonucleotide probes and a newly designed polymer-modified oligonucleotide.

Major hindrances to hybridization onto surfaces are indeed the effect of grafting onto a surface and the subsequent interactions between the immobilized probes.

We thus covalently attached a hydrophobic, long and flexible polymer chain to the oligonucleotide probes. The presence of the polymer enables to space away the probe from the surface and control the grafting density, i. e. probe-probe interactions via Langmuir Blodgett transfer onto a solid support from a monolayer at the air-water interface of the amphiphilic polymer-modified oligonucleotide.

The hybridization process is investigated in label-free conditions with surface-techniques such as the quartz-crystal microbalance and impedance spectroscopy. These studies enable the quantification of the immobilized probes and subsequent kinetics and efficiency of hybridization, comparing the hybridization of the targets with polymer-modified oligonucleotides and their precursor nucleotide sequence.

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Evanescent wave illumination selectively illuminates particles and structures near a surface, this makes it ideal for the investigation of particle/surface and protein/surface interactions. We show a variety of measurement methods that are possible on one single setup.

# Characterization of an adsorbed polyelectrolyte mono-layer on oppositely charged colloidal particles

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An adsorbed mono-layer of a cationic polyelectrolyte, poly(diallyldimethyl- ammonium) (PDADMAC) on negatively charged colloidal latex particles was investigated by small angle neutron scattering (SANS) and dynamic light scattering (DLS). SANS gives a layer thickness of 1 nm which is in perfect agreement with the DLS result. This indicates that the polyelectrolyte chains are adsorbed flat on the surface. From SANS we further find that the layer contains a polymer volume fraction of 0.23, which corresponds to an adsorbed amount of 0.28 mg/m<sup>2</sup>. This value is equal, within the experimental error, to that obtained from the onset of the plateau in the measured electrophoretic mobility curve. The experiments thus clearly indicate that the adsorbed layer of a highly charged polyelectrolyte adsorbed on an oppositely charged colloidal particle is very thin.

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## Reversible and irreversible adsorption of poly(amido amine) dendrimers on Silica

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The adsorption of molecules such as polymers and proteins at the liquid-solid interface is a very common but complex phenomenon of great importance. Poly(amido amine) (PAMAM) dendrimers are hyperbranched globular polymers that are known to strongly adsorb to solid surfaces. Due to their monodispersity, nanoscale dimensions and precisely controlled structure, these dendrimers make for excellent model molecules for adsorption studies. They are also used as globular protein mimics [1]. Previous studies have shown that higher molecular weight dendrimers bind irreversibly to surfaces and form a correlated liquid-like layer [2]. The adsorption was initially understood in terms of the classical random sequential adsorption model but the importance of a three-body electrostatic interaction between two adsorbing dendrimers and the substrate was later discussed in [3]. In this work, the reversibility of PAMAM dendrimer adsorption was studied by optical reflectometry. Reversibility for a given generation depends on the solution pH. This is related to the silica surface charge density. Similarly, reversibility depends on generation for a fixed solution pH. The strength of the adsorption is related to the number of charged terminal amine groups of the polymer. A reversibility map in terms of pH and generation was constructed and can be used to predict the adsorption behavior of the system for given experimental conditions.

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# A combined theoretical and experimental investigation of silica: formation and reversal of charge

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In a recent short review [1] on the charge reversal (CR), i.e. when there is more counter-ions than charges on the surface, Lyklema pointed out the lack of a detailed study, where both the surface charge and the electrokinetic potential of colloids in multivalent salt are characterized. This would allow to unambiguously discriminate between the pure electrostatic and specific adsorption origins of CR. Traditionally, CR is interpreted in the framework of the classical Stern model, solving the mean field Poisson-Boltzmann (PB) equation and using a surface complexation approach, as the result of the specific adsorption of counter-ions. On the other hand, Monte-Carlo simulations and accurate theories clearly demonstrate that the charge fluctuations, the so-called charge-charge correlations ignored in the PB equation, can induce CR. However, in most of these works the surface charge is treated as a fitting parameter independent of the pH, salt composition and concentration.

In the present work, we investigate, combining experimental and theoretical techniques, the validity of a pure electrostatic approach applied to a real colloidal system which shows CR. Our model system is a colloidal suspension of Stöber-type silica particles, characterized in [2]. Using a potentiometric titration method at constant ionic strength we measured the surface charge density of silica in two different electrolyte solutions: potassium and calcium chloride. Calcium ions are shown to strongly promote the negative charge at the silica surface. Simultaneously, the electrophoretic mobility of the silica dispersion in both potassium and calcium salt was characterized. Theoretically, we used a recently developed Monte-Carlo method [3,4], that allows both the description of the charging process and the electrical potential profile (and thus an estimation of the electrokinetic potential) in the double layer as a function of the activity of the protons and other charged species. In presence of KCl, absolute value of particle surface charge increases monotonically with increasing pH. On contrary, in solution of calcium salt at ionic strength of 100 mM and at pH above 7, the absolute value of apparent charge of Stöber-type silica particles is decreasing. In addition, surface charge density measured by potentiometric titration compares well with measurements by Dove et al [5] on Aerosil 380 silica particles. On overall, an excellent agreement between the experimental data and simulation predictions is obtained. Furthermore, the same 3 model parameters were used for both potassium and calcium counter-ions, namely, the surface density of the silanol groups, their intrinsic ionization constant and the minimum separation between the ions and the silanol groups. The latter were set to 4.8 silanol groups/nm<sup>2</sup>, pK<sub>0</sub> = 7.7 and 1.5 Å, respectively. For the system studied, we conclude that i) ion specific adsorption is negligible and ii) CR is mostly induced by charge-charge correlations. More generally, our work clearly illustrates that the classical Stern model and associated surface complexation approach cannot be reasonably used to distinguish between pure electrostatic and specific adsorption effect when dealing with titratable surfaces in presence of multivalent electrolytes.

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## Colloidal monolayer trapped near a charged wall

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Using synchrotron x-ray diffraction from microfluidic channel arrays we have determined concentration profiles of charge-stabilized silica colloids confined between two likecharged silicon walls. For the fluid with low ionic strength, a depletion region forms near the confining walls. A complete rearrangement of the structure and the formation of a colloidal monolayer near the confining wall are observed upon increasing the ionic strength to 0.2 mM. The ionic strength of the colloidal fluid is tuned by adding LiCl. The results can be explained by an effective position-dependent Debye screening length which is much smaller near the walls than in the center of the cavity. Microfluidic array phase profiling (MAPP) technique is employed to obtain spatially resolved colloid concentrations across the confining channels [1].

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## Preparation of Stable Latex Suspensions in Miniaturized Tank-Reactors

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Latexes find widespread use in coatings, textiles and adhesives industries. Functional groups can easily be incorporated into the polymer particles and/or the polymer particle surface. Microemulsions are thermodynamically stable dispersions of droplets less than 30-40 nm in diameter. They are formed spontaneously from mixtures of water and monomer containing large amounts of surfactant. Nanolatexes are characterized by a smaller particle size (diameter usually  $\leq 30$  nm), resulting in a significantly higher specific surface area and thus a higher functionality which increases the number of applications of the products.

A number of parameters such as temperature, concentration of surfactant and initiator and stirring speed influence the outcome of such polymerization reactions and determine the particle size (see below).



**Particle Size**  
~ 60 nm @ 450 rpm

**Particle Size**  
~ 32 nm @ 150 rpm

The Chemspeed MiniPlant technology allows for fully automated screening of these parameters in miniaturized tank reactors and fast transfer of these parameters to the pilot plant scale.

The methodology for the synthesis of a stable nanolatex suspensions, using the Chemspeed AutoPlant robotic platform is described and the effect of parameters such as temperature and shear force (stirring speed) on the particle size is rationalized.

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