

Chapter 1 : Makromolekulare Chemie I: Arbeitsgruppen

*Porous epoxy thermosetting polymers obtained by a phase separation process in the presence of emulsifiers (pages).
A. Garcia Loera, M. Dumon and J.P. Pascault.*

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Abstract Nanosized PTFE-based core-shell particles can be prepared by emulsifier-free seed emulsion polymerization technique starting from spherical or rod-like PTFE seeds of different size. The shell can be constituted by the relatively high Tg polystyrene and polymethylmethacrylate as well as by low Tg polyacrylic copolymers. A very precise control over the particle size can be exerted by properly adjusting the ratio between the monomers and the PTFE seed. Samples with uniformity ratios suited to build 2D and 3D colloidal crystals are easily prepared. In particular, 2D colloidal crystal of spheres leads to very small 2D nanostructuration, useful for the preparation of masks with a combination of nanosphere lithography and reactive ion etching. A great deal of interest was focused on the resulting polytetrafluoroethylene PTFE nano- and microparticle latexes [1 , 2]. Moreover, by varying the amount and structure of PFPE as well as the quantity and nature of the comonomers, PTFE nanoparticles can be designed with highly differentiated architectures, including size, shape, and chemical composition. Many interesting applications were disclosed for these PTFE nanoparticles including components in PTFE bimodal mixtures, ultralow-K dielectric materials, nanofillers for fluoroelastomers and fire-retardant additives with the reported purpose to inhibiting the dripping of molten particles from the burning polymer [3 - 6]. However, the compatibility and adhesion characteristics of PTFE and the various polymeric matrices are inadequate thus resulting in low dispersion degrees within the matrix and poor mechanical coupling among the various blend components. Moreover, the resulting compositions exhibit a pearl scent opaque appearance that prevents their use in applications where transparency is required. Finally, the poor dispersion of PTFE material adversely affects the efficiency in reducing the heat release. To enhance wettability and compatibility, several PTFE surface modification strategies were developed based on either high power chemical and reactive processing procedures [7 , 8] or high energy treatments [9 , 10]. Reactive extrusion of electron-beam irradiated PTFE and polyamides was demonstrated [11 - 13] to be effective in the preparation of well-dispersed compounds due to the occurrence of transamidation reactions accompanied by the breakdown of the PTFE agglomerates. However, consistent degradation of the basic PTFE structure upon irradiation is unavoidable. An alternative and promising nondestructive approach to produce compounds with a perfect dispersion of PTFE particles is based on the preparation of core-shell particles in which the core is constituted by PTFE and the shell by a conventional polymer. In the early studies, several composite particles consisting of PTFE core and crosslinked polybutadiene shell were described by Okaniwa [14] whereas the preparation of one sample only of core-shell particles in which the core is made up of PTFE and the shell of crosslinked PS was reported by Othegraven [15]. More recently, extensive studies were reported for core-shell nanoparticle systems composed of PTFE core and polyacrylic shell [16 , 17] with various compositions. Two-stage seeded emulsion polymerization is the general method [18 , 19] to prepare latex particles featuring a core-shell morphology. The first stage, the core preparation, can be carried out either separately or in situ and the polymerization process of the second stage is a seeded process in which the second monomer is added continuously or batchwise to the reaction vessel. In most cases, some miscibility between the core polymer and the shell-forming monomer produces a partial swelling thus ultimately leading to an intermixing diffuse region. However, in the present case, the shell-forming monomer is always insoluble in the PTFE and the polymerization occurs exclusively in the shell region of the latex. A perfect degree of dispersion of the PTFE particles within a polymer matrix could be obtained if the shell is constituted by the same polymeric material with which the polymer matrix is made up. The application of this concept leads to a novel class of specific and very efficient PTFE-based additives Scheme 1. PTFE dispersion improvement through the use of core-shell particles. An additional reason of interest for these core-shell nanoparticles is also related to their

potential use as building blocks for colloidal crystals and other nanostructured materials [20 – 23]. They can self-assemble to generate 2D and 3D ordered structures which represent promising candidates in applications such as catalysis, sensing, optics, molecular separation, molecular reaction dynamics, single-molecule detection, and optoelectronics. Two-dimensional structuration leads to 2D colloidal crystals which are extensively employed in nanosphere lithography [24] NSL , among others. NSL is based on the self-assembly of nanospheres into close-packed monolayers or bilayers, which are then employed as lithographic masks to fabricate nanoparticle arrays. Consequently, NSL is an efficient, inexpensive, inherently parallel, material-specific, and high-output process for nanostructure fabrication process which can systematically produce a 2D array of periodic structures. The use of spherical colloids to obtain highly ordered periodic 3D structures, that is, photonic crystals, opens novel perspectives in organic photonics since the periodic modulation in structure and composition allows to control light propagation and emission [25]. In addition to the building up of conventional opals, once a well-ordered assembly of core-shell particles is obtained, annealing at a temperature higher than the flow transition temperature of the shell-forming polymer allows the shell-forming polymer to soften and form a matrix surrounding 3D periodic array of in which the PTFE particles are arranged in regular registry. Since the characteristic size of the different domains is on the nanometer range, nanoscale thin films and three-dimensional materials can be prepared Scheme 2. For these applications, the particle size control represents the key to produce successful nanostructures. As monodispersed particles in the 30 nm to 1 micron size range are involved in the preparation of nanostructured materials, emulsion polymerization [26] is the most suitable particle forming technique. In this process, the most important parameter, that controls the particle nucleation, is the surfactant concentration. A narrow particle size distribution can be achieved when a low surfactant concentration is employed [27 , 28]. However, SFEP approach affords particles with size generally comprised between and nm whose monodispersity is not always guaranteed. In addition, a certain variability in the final average particle size is observed. A related approach to prepare such nanosized particles [29 , 30] relies on the seeded emulsion technique starting from seeds of a few nanometers. These can be prepared in situ by a self-seeding technique or can derive from a distinct preparation. Considering the latter approach, polytetrafluoroethylene PTFE latexes with particles in the submicrometer size range can be successfully employed as seeds in the emulsifier-free emulsion polymerization of various monomers including styrene [31 , 32], acrylic mixtures [33], or methyl methacrylate [34 – 36] or thus leading to a wide variety of core-shell nanosphere architectures featuring a relatively narrow size distribution. In this paper, we review our most recent results concerning preparation, properties and self-assembly behavior of PTFE-based core-shell particles using polytetrafluoroethylene PTFE latexes with particles in the submicrometer size range, as seeds in the emulsifier-free emulsion polymerization of various monomers thus leading to a wide variety of core-shell nanosphere architectures in which the shell-forming polymers can be designed with glass transition or flow transition temperatures above or below room temperature. Their characteristics, including particle radius and particle shape, the solid content, the particle number concentration, and the conductivity are collected in Table 1. First, the appropriate amount of PTFE latex was introduced into the reactor containing mL of deionized water at room temperature with a stirring rate of rpm. The mixture was purged with nitrogen for 20 min and nitrogen was flushed during the entire polymerization procedure. Then, after additional 15 min of equilibration time, the potassium persulfate aqueous solution 10 mL, 0. The obtained latex was purified from the unreacted monomer by repeated dialyses using a membrane with MWCO of All the latexes were obtained following the above general procedure by varying the initial PTFE latex amount. The series were marked with an acronym comprising the PTFE seed, the type of monomer or comonomeric mixture, and a number which relates to the initial PTFE weight percent with respect to the monomer or comonomeric mixture. As an example, the MDMn series is obtained from the MD latex and methylmethacrylate M whereas styrene and the two acrylic mixtures are indicated as S, C1, and C2, respectively. The experimental details of the already published sample series are described in the corresponding papers as summarized in Table 2. Already published core-shell series and corresponding references. Synthesis details, yield, and solid content of the various samples. Preparation of 2D and 3D Colloidal Crystals 2D colloidal crystals were prepared by the floating technique [37 – 39], on the

air liquid interface and then lifted on the surface of a solid substrate. This gives an accurate control of the floating level of the monolayers on the water surface thus allowing the formation of large 2D crystals screening the surface charge on the sphere by water molecules. The final addition of a surfactant in the water further compacts the 2D crystalline islands. In this phase, parameters like sphere surface charge, ethanol content, and dispersion method are rather critical in the formation of large crystal assemblies. Silicon substrates have been used to lift the floating crystals. Opals were composed of flat domains with the $[100]$ direction of the face-centered cubic lattice of spheres perpendicular to the substrate. In the best samples, domain sizes are 500 nm. However, defects inside these domains, in particular stacking faults, cannot be excluded. Opals are marked with the prefix O before the name of the sample employed to build up the corresponding opal. Each value is the average of five measurements. The instrument was checked with a standard polystyrene latex with a diameter of 100 nm. Electrophoretic mobility was measured with a Malvern Zetasizer HS. A multilayer of nanoparticle is obtained by drop casting and aliquot of water suspension on a disc of freshly cleaved muscovite mica Electron Microscopy Sciences, Hatfield, PA, USA and the solvent is allowed to slowly evaporate. Images are then subjected to flattening using the microscope constructor software. The solid content of latex dispersions was determined by TGA. Samples of about 5 mg were employed. Dry nitrogen was used as purge gas. Transmitted light was collected and driven by another optical fibers to the spectrometer. Normal incidence reflectance was measured on six different 2 mm diameter spots by a Y reflection probe bundle fiber. Light was linearly polarized by using a Glenn-Thompson Halbo Optics polarizer. All details on the optical set-up are reported in [41].

Results and Discussion 3. Synthesis PTFE latexes with different size and shape were prepared by microemulsion polymerization and employed as seeds in the successive emulsifier-free seeded emulsion polymerizations. As the presence of the residual surfactant deriving from the TFE microemulsion polymerization could interfere with the successive emulsifier-free seeded emulsion polymerization, all the PTFE latexes were thoroughly dialyzed. At the end of the reaction, the latexes were purified by repeated dialyses. In all the polymerization reactions, monomer s and potassium persulfate as well as the water content were kept constant, whereas variations were allowed in the amount of PTFE. No gel trace or polymeric gross aggregates were found in all cases. Nearly quantitative monomer conversion and nanosphere yield were obtained. The final latexes are very stable up to two years. Although these samples are prepared starting from PTFE seeds differing in their size and using styrene or methylmethacrylate as the shell-forming monomer s , in all cases they feature a quite narrow size distribution. The composition of all the samples can be easily estimated from the TGA analysis, as reported in Figure 3 for typical samples. In all the thermograms, there are two main losses. The composition of the various samples is in excellent agreement with the one calculated from the amount of the initially added PTFE and monomer or comonomeric mixtures. The described trend is observed in all the sample series prepared starting from the various PTFE seeds and using styrene or methylmethacrylate. To make a homogeneous comparison, the size and U data are plotted as a function of the ratio between the amount of styrene or methylmethacrylate and the number of PTFE particles in the reaction mixture. Figure 7 illustrates the various data. For both monomers, the size and uniformity ratio trends are very similar. The particle size increases as the ratio between the amount of monomer and the number of PTFE particles increases whereas the relevant size distribution decreases. Provided that no pure PMMA and PS or PTFE nanospheres are present at the end of the reaction, and taking into account the yield values, the size of the core-shell nanospheres can be estimated from the amount of the initially added PTFE and the relevant monomer. In all cases, the estimated size is in close agreement with the observed one. The overall picture of these data clearly indicates that a very precise control over the particle size and size distribution can be exerted by properly adjusting the ratio between the monomer and the PTFE seed. During Smith-Ewart Interval II [19], the growing mechanism involves diffusion of monomer from the droplets to the particles and the growing probability appears related to the latex particle surface area. Consequently, bigger particles should display a growing probability higher than smaller particles. However, this mechanism would ultimately lead to an increase in the size distribution, in contrast with the experimental observations. We suggest that the thermodynamic tendency toward a reduction of the surface to volume ratio could produce an increased tendency of the smaller particles to absorb the monomer with respect to the bigger

ones thus ultimately leading to size uniformity, provided that the monomer to seed ratio is sufficiently high.

Chapter 2 : Preparation, Properties, and Self-Assembly Behavior of PTFE-Based Core-Shell Nanospheres

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The use of microwave irradiation for the easy synthesis of graphene-supported transition metal nanoparticles in ionic liquids Carbon. Synthesis and characterization of semicrystalline triblockcopolymers of isotactic polystyrene and polydimethylsiloxane Journal of Polymer Science, Part a: Iron nanoparticles supported on chemically-derived graphene: Catalytic hydrogenation with magnetic catalyst separation Advanced Synthesis and Catalysis. Mesoporous silica supported multiple single-site catalysts and polyethylene reactor blends with tailor-made trimodal and ultra-broad molecular weight distributions. Hydridoboranes as modifiers for single-site organochromium catalysts: Effects of VEGF loading on scaffold-confined vascularization. Bone repair by cell-seeded 3D-bioplotting composite scaffolds made of collagen treated tricalciumphosphate or tricalciumphosphate-chitosan-collagen hydrogel or PLGA in ovine critical-sized calvarial defects. Part B, Applied Biomaterials. Accelerated angiogenic host tissue response to poly L-lactide-co-glycolide scaffolds by vitalization with osteoblast-like cells. Graphene nanocomposites prepared from blends of polymer latex with chemically reduced graphite oxide dispersions Macromolecular Materials and Engineering. Bioinspired macromolecular chemistry - Paying tribute to the pioneering advances of hermann staudinger and helmut ringsdorf Macromolecular Chemistry and Physics. Consequences of seeded cell type on vascularization of tissue engineering constructs in vivo. Palladium nanoparticles on graphite oxide and its functionalized graphene derivatives as highly active catalysts for the Suzuki-Miyaura coupling reaction. Journal of the American Chemical Society. Synthesis, structure, and ethylene polymerization activity Journal of Organometallic Chemistry. Catalysts based upon organoclay with tunable polarity and dispersion behavior: New catalysts for hydrogenation, C-C coupling reactions and fluoros biphase catalysis Catalysis Letters. Material model in the simulation of curing behavior of light-hardening resins: Formgenauigkeit in der Stereolithographie Konstruktion. Improvement of vascularization of PLGA scaffolds by inosculation of in situ-preformed functional blood vessels with the host microvasculature. Bacterial and Candida albicans

adhesion on rapid prototyping-produced 3D-scaffolds manufactured as bone replacement materials. Vascularization and biocompatibility of scaffolds consisting of different calcium phosphate compounds. Incorporation of growth factor containing Matrigel promotes vascularization of porous PLGA scaffolds. Ethylene polymerization, on-line particle growth monitoring, and in situ nanocomposite formation using catalysts supported on arylsulfonic acid-modified boehmites Macromolecules. Molecular weight and end group control of isotactic polystyrene using olefins and nonconjugated diolefins as chain transfer agents Macromolecules. Boehmite-based polyethylene nanocomposites prepared by in-situ polymerization Polymer. Novel polyolefins containing crystallizable isotactic polystyrene side chains Macromolecular Rapid Communications. Acrylic nanocomposite resins for use in stereolithography and structural light modulation based rapid prototyping and rapid manufacturing technologies Advanced Functional Materials. Novel organic-inorganic nanohybrids based on poly N,N-dimethyl acrylamide polyisobutylene amphiphilic polymer conetworks Technical Proceedings of the Nsti Nanotechnology Conference and Trade Show, Nsti-Nanotech, Nanotechnology New nitrile-and amine-functionalized polyolefin nanoparticles via tandem catalysis in aqueous miniemulsion American Chemical Society, Polymer Preprints, Division of Polymer Chemistry. Comparative in vitro study of the cell proliferation of ovine and human osteoblast-like cells on conventionally and rapid prototyping produced scaffolds tailored for application as potential bone replacement material.

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Lee, Solubility and diffusion coefficient of gaseous ethylene and?? Masuoka, Solubility of propylene in semicrystalline polypropylene, Sorption of Olefins in High Impact Polypropylene, pp. Kiparissides, Ethylene, and Propylene? Ethylene mixtures in Polypropylene 18 Prediction of Solubility of? Lacombe equation of state, Fluid Phase Equilibria, vol. Reichert, Sorption studies of propylene in polypropylene. Diffusivity in polymer particles formed by different polymerization processes, Journal of Applied Polymer Science, vol. Morbidelli, Measurement of diffusivity and solubility of olefins in polypropylene by gas chromatography, Journal of Applied Polymer Science, vol. Kanellopoulos, Hybrid model for the diffusion of simple and complex penetrants in polymers, Journal of Applied Polymer Science, vol. Yang, Diffusion measurements of isopentane, 1-hexene, cyclohexane in polyethylene particles by the intelligent gravimetric analyzer, Journal of Applied Polymer Science, vol. Smith, Permeation of ethane??? Ward, Single and mixed gas diffusion through polyethylene films, Polymer, vol. Alizadeh, Study of sorption, heat and mass transfer during condensed mode operation of gas phase ethylene polymerization on supported catalyst. Crank, The Mathematics of Diffusion, Frisch, Diffusion in polymers, 4. Doshev, Heterophasic copolymers of polypropylene: Development, design principles, and future challenges, Journal of Applied Polymer Science, vol. Mckenna, Study of the kinetics, mass transfer, and particle morphology in the production of high-impact polypropylene, Journal of Applied Polymer Science, vol. Kwong, On the Thermodynamics of Solutions. An Equation of State. Fugacities of Gaseous Solutions. Gahleitner, Structure-property relations of heterophasic ethylene-propylene copolymers based on a single-site catalyst, Express Polymer Letters, vol. Arnal, Thermal fractionation of polymers, Progress in Polymer Science, vol. Chen, Controlled properties of high-impact polypropylene in-reactor alloys by tailoring chemical structure and morphology, Journal of Polymer Research, vol. Wunderlich, Addendum to the thermal properties of polypropylene Toporov, Effect of contact deformations on the adhesion of particles, Journal of Colloid and Interface Science, vol. Bouzid, Interaction between the polymerisation process and particle morphology, Nestelberger, Melt viscosity effects in ethylene??? Doshev, Phase interactions and structure evolution of heterophasic ethylene??? Some new aspects about morphology and fracture, Journal of Applied Polymer Science, vol. Zhou, Probing into the pristine basic morphology of high impact polypropylene particles, Polymer, vol. Pasch, Defining the distribution of ethylene-propylene copolymer phases in heterophasic ethylene-propylene copolymers by a sequential xylene extraction method: Chemical and morphological analysis, Polymer, vol.

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N. BELHANECHÉ- BENSEMRA, A. BEDDA, B. BELAABED, Â«Study of the properties of rigid and plasticized PVC/PMMA blendsÂ», Macromolecular Symposia, Vol. , pp ,