Characterization of Synthetic Latexes – Physical, Chemical, Colloidal and Morphological Properties

<u>Day 1</u>

<u>AM</u>

- What does it mean to "fully" characterize a latex?
- Basics of creating synthetic latices
 - 1. Particle nucleation and growth
 - 2. Control of particle size distribution
 - Control of copolymer composition, MW, gel content
 - 4. Functional additives (esp. vinyl acids) and neutralization
 - 5. Residual monomer reduction
- Latex applications
 - 1. Coatings (architectural, paper, adhesive, textile)
 - 2. Thermoplastic impact modifiers
 - 3. Printing inks
 - 4. Cement and asphalt modifiers
- Physical and colloidal properties of latices
 - 1. Particle size distributions techniques, comparisons, limitations. Homo- and copolymers. Multi-phase particles. Mono- and bimodal latices
 - 2. Problem solving

PM

- Physical and colloidal properties of latices
 - 1. Particle shape SEM, TEM, AFM
 - 2. Particle density monomer type, water content, composite particles
 - 3. Surfactant adsorption and coverage, CMC
 - 4. DLVO theory charge interactions, nonionic surfactants, titration procedures
 - 5. Competitive adsorption between ionic and non-ionic surfactants
 - 5. Electrophoresis Zeta potential
 - 6. Colloidal stability (CCC, Maron, Waring blender)
 - 7. Establishing surfactant adsorption isotherms

<u>Day 2</u>

AM

Glass transitions, Tg

- 1. Dynamic mechanical analysis (DMA)
- 2. Differential scanning calorimetry (DSC)
- 3. Homo- and copolymers characteristic shapes of transitions through the glass point
- 4. Flory-Fox, Ponchon, Gordon-Taylor equations
- 5. "Wet" T_g. Measurements and contrast with "dry"T_g. Water and organics as plasticizers.
- Minimum film formation temperature (MFFT)
 - 1. Instrumentation and operating conditions
 - 2. Reading the boundary
 - 3. Relationships between MFFT and T_g .
- Latex viscosity
 - 1. Rheometers simple, sophisticated
 - 2. Real and "apparent" viscosities
 - 3. Newtonian and shear thinning viscosity
 - 4. Which viscosity is important?

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- Chemical Properties
 - 1. Overall polymer chemistry
 - a.) Composition average and distribution, NMR, FTIR
 - b.) MW average and distribution, viscometry, GPC
 - c.) Orthogonal chromatography for CCD and MWD
 - 2. "Regional" chemistry
 - a.) Surface chemistry surfactant adsorption, initiator end groups
 - b.) Functional monomers (e.g. AA– distribution in serum and in/on particles (titration techniques)

3. Branching and crosslinking

- a.) Sol solvent extractions, characterization of sol polymer
- b.) Gel solvent swelling index NMR (solid state) for branch points
- c.) DMA of films
- 4. Grafting
 - a.) Selective extraction, grafting efficiency, number of graft sites
 - b.) Solid state NMR
 - c.) MWD of sol (GPC),
- 5. Residual monomer content GC, HPLC

• Serum phase characterization

- a.) Separation of serum phase by centrifugation or filtration
- b.) Serum replacement techniques
- c.) Water soluble polymer (overall overall MW)
- d.) Individual chain identification liquid chromatography, mass spectroscopy

<u>Day 3</u>

<u>AM</u>

• Structured Particles – Morphology

- 1.) General considerations what details are we looking for?
- 2.) External shape SEM, AFM, soft and hard particles, surface chemistry
- 3.) Internal structure
 - a.) Phase separation within particle thermodynamics, kinetics
 - b.) Extent of phase separation quantitative assessment via DSC, DMA – qualitative assessment via TEM

- c.) Polymer-polymer interfaces within particles
- d.) Comparisons between "as is" and thermally annealed samples
- e.) Polymer-polymer de-mixing within particles
- 4.) Problem solving

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• Structured Particles - Morphology

- 5.) Location of the polymer phases within the particle
 - a.) AFM (height and phase modes)
 - b.) TEM (*whole particle*) staining for phase contrast, soft particles, particle edge effects, false positives
 - c,) TEM (*sectioned*) embedding resins (epoxy, latex), sectioning, chemical staining (e.g. Os, Ru, PTA)
 - d.) Minimum domain sizes, interfacial regions, phase ratio constraints, false positives
- 6.) TEM EELS techniques
- 7.) STXM X-ray microscopy
- 8.) The need for "complementary" data sets
- 9.) Problem solving
- Review of important concepts
- Open discussion of questions from participants