CCMX ADVANCED COURSE: Inorganic Particle Synthesis by Precipitation

OBJECTIVES

- Introduction to the basics behind precipitation of inorganic powders in theory and in practice
- Discussion of the fundamental concepts of supersaturation, nucleation, growth and aggregation
- Brief introduction to some basic methods used for powder characterisation
- Demonstration of real sample characterisation and correct interpretation of the results collected
- Presentation of precipitation reactors from batch to continuous and in-situ monitoring
- Presentation of "sol-gel" routes in both aqueous and non-aqueous environments
- Thermodynamic and kinetic modelling towards understanding growth mechanisms

TOPICS

- Precipitation basics — supersaturation, nucleation, growth and aggregation
- Powder characterisation — particle size, surface area, morphology (microscopy), X-ray powder diffraction, zeta potential and thermogravimetric analysis
- Precipitation in practice — reactor engineering — from batch to continuous reactors
- "Sol-gel" routes in both aqueous and non-aqueous environments and polyol routes
- Modelling — solution thermodynamics, kinetics (population balance), aggregation, self-assembly
- Case studies — Superparamagnetic iron oxides, towards Good Manufacturing Practice (GMP) for biomedical applications

Please note that while the theory presented in this course applies to many types of materials, the practical examples concentrate on inorganic materials. Also due to limited time only a brief overview of the theory can be given although key references for more advanced analysis are provided throughout.

LECTURERS

Prof. Paul Bowen obtained his PhD in Physical Chemistry in the field of catalysis from the University of Cambridge, UK, in 1982. He then worked at the BP Research Centre, Sunbury, UK, for four years in applied surface sciences before moving to Switzerland and EPFL in 1987. He has been at the Powder Technology Laboratory, in the Materials Institute since its conception in 1988. His main research interests are powder synthesis, powder characterisation, colloidal processing and sintering of ceramics, and atomistic modelling of surfaces and interfaces.

Prof. Markus Niederberger is Chair of the Laboratory for Multifunctional Materials at ETH Zurich. He studied chemistry at ETH Zurich, where he also received his PhD. After a postdoc at the University of California at Santa Barbara, he became group leader at the Max Planck Institute of Colloids and Interfaces in Potsdam. In 2007 he was appointed Assistant Professor in the Department of Materials at ETH Zurich and promoted to Associate Professor in 2012. His research focuses on the development of liquid-phase synthesis and assembly routes to inorganic functional materials over all length sclaes.

Dr. Andrea Testino received his PhD in Material Science (2004) from the University of Genoa and did post-doctoral work at EPFL and at University College London before joining the University of Milan (Italy) as a permanent researcher working on nanoparticle synthesis and characterisation in 2005. In 2007 he joined TDK-EPC in Austria as Senior Development Engineer before moving to the Paul Scherrer Institut as Senior Scientist in 2011, establishing the Advanced Nanopowders Synthesis Laboratory. He is now focused on the continuous synthesis of nanoparticles and thermodynamic and kinetic modeling of precipitation processes.

MONDAY, 19 OCTOBER

9:00 Welcome + Introduction to the course (P. Bowen)

9:15 Precipitation basics I — growth and aggregation (A. Testino)
- Precipitation generalities
- Supersaturation and nucleation
- Solution thermodynamics
- Particle growth — crystallisation

10:30 Coffee Break

11:00 Precipitation Basics II (A. Testino)
- Modelling: Thermodynamic speciation (real supersaturation)
- Precipitation kinetics and population balance methods
- Non-classical growth & pre-nucleation species

12:30 Lunch together

14:00 Example of kinetic modelling - calcium silicate hydrate (A. Testino)

14:30 Test case - kinetics of nucleation and growth - amorphous calcium carbonate (A. Testino)

- Surface area and porosity measurement — nitrogen adsorption/desorption
- Morphology (microscopy)

15:30 Coffee Break

16:00 Preciptiation in practice - reactor engineering - from batch to continuous reactors (P. Bowen)
- Batch reactor and mixing consideration
- Continuous reactors
- Practical case studies

Discussion on the topics of the day

17:30 End of first day

TUESDAY, 20 OCTOBER

9:00 Powder Characterisation I (P. Bowen)
- Particle size measurement
- Surface area and porosity measurement - nitrogen adsorption/desorption
- Morphology (microscopy)

10:30 Coffee Break

11:00 Powder Characterisation II (P. Bowen)
- X-ray powder diffraction
- Zeta potential
- Thermogravimetric analysis and examples

12:30 Lunch together

14:00 Practical demonstrations (P. Bowen, A. Testino)
i) Powder characterisation (PSD, SSA, porosity)
ii) Powder synthesis in practice, mini-batch, baffled batch and continuous reactors and mixers

15:30 Coffee Break

16:00 Shape control and atomistic modelling (P. Bowen)

Discussion on the topics of the day

17:30 End of second day

WEDNESDAY, 21 OCTOBER

9:00 "Sol-gel" routes in both aqueous and non-aqueous environments I (M. Niederberger)
- Aqueous routes
- Non-aqueous routes
- Polyol routes

10:30 Coffee Break

11:00 "Sol-gel" routes in both aqueous and non-aqueous environments II (M. Niederberger)
- In-situ monitoring — towards precipitation mechanisms
- Examples, case studies

Discussion on the topics of the morning

12:30 Lunch together

14:00 Examples and case studies - towards Good Manufacturing Practice (P. Bowen)
- Super paramagnetic iron oxides (SPIONS) for biomedical applications

Discussion of course participants' specific concerns

15:30 Concluding remarks

15:45 End of course

16:00–17:00 Examination for doctoral school credits