International Workshop on Dispersion Analysis and Materials Testing 2014

Berlin, Germany
January 23 – 24, 2014

Again, the LUM GmbH, specialist for analytical devices for dispersion analysis and particle characterization, hosted their annual International Workshop in Berlin. About 90 participants discussed the production and optimization of nanoparticle suspensions, foams and emulsions as well as separation characteristics of solid-liquid systems. For the first time, the Young Scientist Award had been launched before, and 4 nominated research projects were presented to the audience.

Hermann Nirschl from Karlsruhe Institute of Technology (KIT) works on particle-particle interactions and interactions between particles and rigid walls. In recent time special emphasis was laid on the centrifugal separation of super-magnetic particles, which are functionalized with ion exchanger molecules, from liquids. The superposition of the centrifugal field and a gradient magnetic field is achieved by a wire matrix installed inside the centrifuge chamber. The functionalized particle surface binds target molecules, e.g. proteins within fermentation broths. For rapid analysis the superposition of magnetic and centrifugal forces was realised in a LUMiFuge equipped with newly developed sample holders, where different magnets could be used for attracting magnetic particles on defined substrates. Force load applied simply by centrifugal field upon interacting particles allows for quantifying interaction strength, and contrary to alternative analytical systems this measuring system allows determining a high number of particle interactions with a limited number of experiments.

The Hansen solubility parameters (HSP) are a quantitative measure for the solubility of molecules within different solvents. In detail they describe affinity as combination of the energies from dispersion forces, from dipolar intermolecular force, and from hydrogen bonds between molecules. In the three-dimensional parameter space the HSP form the centre of a sphere with a so-called interaction radius. Molecules are easy to disperse within solvents with similar HSP ("like seeks like"). Shinichi Takeda from Takeda Colloid Techno-Consulting in Japan extended the HSP concept on nano-particles which are to be dispersed in liquids. The HSP parameter spheres were determined by a LUMiReader by measuring the dispersibility of a nanoparticle in different solvents with known HSP (HSPs of solvents are available in a database under www.hansen.solubility.com). The relative sedimentation velocity of the considered particles serves as measure for the HSP calculation.

Different analytical systems measure different particle sizes due to different physical characteristics correlated to the size. This is even more valid with non-spherical structures. Frank Babick, Technical University of Dresden, addressed these and compared the particle size distributions obtained with two systems for dynamic light scattering and sedimentation technique (LUMiSizer) for pyrogenic powders, which build fractal agglomerates. He showed how the characteristic parameters measured with the respective systems (intensity of scattered light and hydrodynamic properties) depend on the fractal dimensions of the aggregates. On the basis of the relationships between agglomerate structure and measured properties together with some assumptions concerning the aggregate structure (monodisperse, spherical primary particles, DLCA-like aggregate structure) the measured particle size distributions were converted from optically weighted to number weighted distributions, which were more similar for both measurement techniques [1, 2].

The RUSNANO Metrology Center in Moscow offers reference nanoparticles from Al_2O_3, TiO_2, SiO_2, and ZnO in form of colloids with certified number-based and intensity-weighted mean particle sizes. Such reference colloids are applied for calibration purposes, filter testing, or for in vitro assessments of the impact of nanomaterials on living systems. As Anna Lizunova described, particle sizes and size distributions are measured by transmission electron microscopy, acoustic spectroscopy, optical centrifugation analysis with the LUMiSizer and dynamic light scattering. The TEM diameter for all colloids was about 30% lower than the values given by acoustic spectroscopy and analytical centrifugation. Dynamic light scattering delivers about twice as high mean diameters. With spherical particles and a normal size distribution, best agreement of data from different measurement systems was achieved. The highest discrepancy of measured values from different systems could be found with ZnO, due to the non-spherical shape of the particles.

Keren Fogel, Perrigo Israel Pharmaceutical Ltd., applied the LUMiSizer as bioequivalence tool in nasal

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screen oil. By adding an organogelator to the oil, jelly-like nanoparticles are formed that are dispersed in water afterwards by using a stabilizing agent. Gellifying the oil turned out to make more stable dispersions than the standard O/W emulsions.

Yogev Dahan from Perrigo Israel Pharmaceutical Ltd. described the production and stability analysis of emulsion foams for external application. The question of stability arises, because the original aerosol is stored under elevated pressure in the can before being applied. The LUMiFuge results (transmission curves and results of separation front tracking) showed that the foam stability depends on the preparation of the oil in water emulsion. The ways of preparation differ from each other in the temperatures of the two phases during mixing and afterwards. As in the competitive area of the pharmaceutical industry development time is an essential factor, applying the LUMiFuge means a clear progress in this field.

Mathieu Balcaen and collegues from Ghent University, Belgium, tested analytical centrifugation for yield determination of double W/O/W (water/oil/water) emulsions. The yield parameter to be analysed is the resulting percentage of aqueous phase remaining within the oil droplets after the final preparation step. As the analytical method that has been applied so far, a low resolution PFG (pulsed field gradient)-NMR-spectroscopy, is costly and time-consuming, a LUMiFuge that offers a more simple and straightforward data analysis was applied. Here oil droplets with internal aqueous phase (cream) are separated from the surrounding water phase. With front tracking the volume of the cream layer can be calculated, and by subtracting the oil volume from this value, the yield parameter is obtained. Good matching of the results obtained from several samples with PFG-NMR-spectroscopy and the LUMiFuge was observed.

The colloidal interactions through sedimentation and the compressive yield stress data were in the focus of Simon Biggs from University of Leeds, UK. These features are of major interest when a colloidal dispersion settles out, especially with respect to filtration, pumping or and/or re-suspension issures. As applications the optimization of printing inks, flocculation processes and experiments with colloidal dispersions that serve as models for highly active nuclear waste [4] were mentioned. Reprocessing of spent nuclear fuel produces a highly active liquor (HAL) waste stream. These liquids are stored in tanks and continuously cooled. Over the time particles are known to precipitate, which cannot be removed from the enclosed system. One has to take care that the particles remain dispersed in order to prevent incrustation of heat transfer areas.

At Melbourne University Shane Usher examines colloidal interactions through sedimentation with focus on the dewaterability of sludges. Depending on the volume concentrations, sedimentation was examined with batch settling, pressure filtration and centrifugation. Centrifugation was realized with a LUMiFuge [5] at intermediate concentrations of solids. Equilibrium centrifugation data were used to predict the yield stress P_y as function of the sediment porosity for concentrations below the gel point. With step-wise lowering the rotational speed re-expansion was observed. With rebound yield stresses and the yield stress at maximum solids volume fraction within the sediment the elastic rebound factor was determined. This factor was between 2 and 4 for all materials tested so far. It was assumed that it is correlated to a natural physical constant as the Trouton ratio.

Sedimentation characteristics with focus on the filterability of solid-liquid systems are examined at Laboratory of Agro-Industrial Technologies at University of Compiègne in France. Maksym Loginov explained the measuring procedure with a small filter element mounted into the cuvette of the LUMiSizer. Twelve samples can be analysed simultaneously and allow a high throughput. With this equipment the filtration kinetics for different juices and extracts, suspensions and sludges were determined. From experimental data the filter resistance, permeability and specific resistance of the filter cake can be calculated.

Young Scientists Award – contributions

Suzanna Azoubel from Hebrew University in Israel produces carbon nanotube dispersions and characterizes them by analytical centrifugation. Flexible CNT films are intended to serve as alternative for indium tin oxide as transparent conductive material, which is applied in touch screens. CNT are hardly soluble and tend to aggregate, so the dispersions were homogenized under high pressure. Inter alia the dispersibility of CNT was increased by functionalizing through carbonisation. The LUMiFuge was applied for the rapid quality controlling of the CNT dispersions.

Lalel Solhi from Iran Polymer and Petrochemical Institute focussed on the stabilisation of Montmorillonite (Na-MMT) nanoclays in dilute dental adhesives against unwanted segregation. Aim of the investigations is the improvement of the physic-mechanical properties of dental adhesives by adding poly(acrylic acid) grafted nanoclay platelets. Separation Analyser LUMiReader was applied for the determination of the sedimentation behaviour of these newly prepared dental adhesives in comparison to formulations with pristine Na-MMT nanoclay structures [6].

"Probing the Stability of Sterically Stabilized Polystyrene Particles by Centrifugal Sedimentation" quali-

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fied Huai Nyin Yow from the University of Leeds, UK, for the final round of YSA candidates. In her work the attention was put on colloidal dispersion stability. The capability of the LUMiSizer was documented for the characterization of rheological properties of suspensions with polymer decorated "smart" particles.

The prize was awarded to M.Sc student Shir R. Liber from Bar-Ilan University in Israel for her contribution "Dense colloidal fluids form denser sediments". In her research the LUMiFuge was applied to relate the volume concentration of colloidal dispersions with the nature of their randomly packed solid sediments. The experimental data were reproduced by computer simulations [7].

The next International Workshop on Dispersion Analysis and Materials Testing will take place on 22 - 23 January 2015 in Berlin. On this occasion the Young Scientist Award 2015 will be launched.

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User Seminar of 2D and 3D Rheology and Stability of Disperse Systems

Potsdam, Germany
May 12 – 14, 2014

In May 2014 the 5th User Seminar on "2D and 3D Rheology and Stability of Disperse Systems" was organized in Potsdam. Anton Paar Germany GmbH (www.anton-paar.com), LUM GmbH (www.lum-gmbh.de), and SINTERFACE Technologies (www.sinterface.com) organized this event in a joint venture. Again the conference hotel "Am Templiner See" was used as it is well suited for workshops with about 50 participants. The workshop contained lectures on the fundamentals of surface science, colloidal systems, bulk rheology, and on the available experimental tools, which were given by representatives of the organizing companies. Invited speakers from research groups at universities and in industry reported on various subjects relevant for the topics of the workshop. In addition, the participants took actively part in case studies, where they worked on practical situations engineers encounter in their daily work. As in the earlier workshops of this series the venue turned out to be a perfect location with all required services. All lectures were given in German because the participants were exclusively from Germany, Austria, and Switzerland.

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