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Crystal screening by second order nonlinear optical imaging of chiral crystals (SONICC)

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The recent emergence of stable, handheld ultrafast (~100 fs) laser sources has opened practical access to fundamentally new classes of nonlinear light-matter interactions for characterization crystallization phenomena. In this work, second order non-linear optical imaging of chiral crystals (SONICC) is investigated as a rapid (up to video rate), selective, and ultra-sensitive all-optical method for high-throughputs studies of active pharmaceutical ingredient (API) crystallization phenomena. SONICC relies on second harmonic generation (SHG), or the frequency doubling of light, which is forbidden in unordered media and most achiral salts, but allowed for the overwhelming majority of chiral crystals. This high specificity to chiral crystals combined with an insensitivity to optical scattering and an enormous (~8 decade) dynamic range of linear quantification open new opportunities for high-throughput screenings.

Three proof-of-concept applications of SONICC will be demonstrated for characterization of API formulations; i) polymorph screening from the polarization-dependence of the SHG response, which provides up to 18 unique observables specific to a particular crystal compared to 3 for linear optics, ii) crystallization screening with reliable quantification of trace API crystallinity with sub-parts per million to parts per billion detection limits in powdered APIs and in amorphous polymer formulations (i.e.,4-7 orders of magnitude improvement over most current routine methods for powder analysis), and iii) direct determination of nucleation kinetics within powders and turbid matrices without significant loss of image quality from optical scattering (since only the unscattered incident light contributes to the detected signal). Quantitation and polarization analysis can be performed rapidly with high signal to noise on single crystals less than ~1 μm^3 in size, opening opportunities for performing screenings of hundreds or thousands of conditions with just a few milligrams of purified API when coupled to emerging ultrahigh-throughput microfluidic screening platforms. Furthermore, SONICC is directly compatible with most existing high-throughput screening plates that allow conventional optical imaging without modification. The absence of a background response from disordered media allows the development of simple image analysis algorithms for automated quantification of nucleation rates, crystal growth rates, and activation energies for nucleation.

An overview of the methods and analysis will be presented, along with and a critical discussion of the strengths and limitations of SONICC for practical high-throughput screening and a summary of some of the challenges associated with the measurements. In addition, a description will be provided of the basic instrumentation required for SONICC, which is anticipated to be commercially available in the near future through partnership with Formulatrix.

Keywords: polymorphism, screening, high-throughput

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Prospects and challenges in the synthesis of zeotype materials

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This presentation will cover the challenges in the preparation of discrete zeo-type molecular sieves with emphasis on their complex crystallization mechanism under hydrothermal synthesis conditions. As zeo-type materials, nanosized aluminophosphates (AlPO-5, -11, -18, -41), silicoaluminophosphate (SAPO-5, -11, -18, -41) and titanosilicates (TS-1 and ETS-4) will be presented. The control of precursor suspensions on the nanometric scale allows the preparation of zeo-type nanosized crystals with desired crystalline structures, porosity and particle size.

The focus of this presentation will be on the preparation of nanosized materials from transparent precursor suspensions containing single or mixed templates under controlled microwave or conventional heating. Optimization of the synthesis parameters and detail characterization of the crystallization process provide information on the important design rules for such materials.

Emphasis on green synthesis approaches involving multi-step microwave or conventional heating *via* reusing of non-reacted compounds from precursor suspensions with essential chemical compensation will be presented. The microwave syntheses for nanocrystalline AlPO-*n* and SAPO-*n* have shown almost complete consumption of the organic templates and no disposal of detrimental reagents to the environments. On the other side the preparation of nanocrystals with desired properties and reasonable yield is achieved. The use of microwave instead of conventional heating directs selectively the synthesis of one versus other types of porous materials for extremely short time, thus decreasing the energy consumption and making possible the chemical process to be environmentally benign.

Further the stabilization of the nanoparticles in suspensions using different solvents and surface modifiers together with possible assembling of the zeo-type nanosized materials in thin-to-thick films will be presented.

Keywords: synthesis, nanoparticles, zeo-type materials

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Crystallography, physical properties and applications of sulfosalts

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Sulfosalt minerals are characterized by complex crystal structures with low symmetry [1], [2]. From the total of more than 260 known phases of the sulfosalt family, about 80% crystallize in the orthorhombic and monoclinic system. Therefore, physical properties can be strongly anisotropic and must be described by tensors. Due to important applications in thermoelectric and photovoltaic energy conversion, anisotropic transport properties, like electrical or thermal conductivity, are of basic importance.

The anisotropic electrical conductivity of typical sulfosalts will be investigated and correlated to their crystal structures using in situ methods on a SEM including EDXS, EBSD, and micro-conductivity measurements. Different single crystals of natural grown sulfosalts were oriented, cut, and polished. The electrical conductivities in different directions of the polished and cleaved planes were measured by a 2point and a 4point probe technique. Results were correlated to the crystal orientations and tensors were estimated.

[1] Y. Moëlo, E. Makovicky, N.N. Mozgova, J.L. Jambor, N. Cook, A. Pring, W. Paar, E.H. Nickel, S. Graeser, S. Karup-Møller, T. Balić-Žunić, W.G. Mumme, F. Vurro, D. Topa, L. Bindi, K. Bente, M. Shimizu, *Journal of Mineralogy*,