The Potential of Diffuse Reflectance Far IR Spectroscopy for the Characterization of Liquid-exfoliated Nanomaterials

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Diffuse reflectance infrared Fourier transform spectroscopy (DRIFT) setups available as commercial benchtop equipment has become a widely applied spectroscopy technique for loosely packed powders. Due to the mirror setup, not only specularly reflected light is collected but also diffusely reflected light which stems from multiple absorption and reflection events and thus provides surface-related information. Many vibrational modes of organic compounds can be found in the mid-IR range, which is why standard spectrometers are designed with dedicated mirrors, beam splitters and optical windows optimized for MIR. For inorganic compounds, such as Molybdenum disulfide (MoS_2), the metal-chalcogen IR active vibrations are typically found at lower wavenumbers, i.e., the far-infrared range (FIR)[1], but typically not considered for 2D materials since powders are traditionally not available.
Here, we unravel the potential of DRIFT in the FIR regarding the characterization of layered inorganic materials using MoS_2 as model substance in the form of bulk powder and randomly restacked, size-selected nanosheets. Since various crystal defects and impurities are known to be present in commercial MoS_2 powders, DRIFT spectroscopy is first used to assess the quality from different raw materials. In addition, different material processing within the field of TMD nanomaterials also affects the crystal lattice on the atomic scale. In order to understand this in more detail, the FIR modes of freeze-dried powders of TMD nanosheets after different liquid-exfoliation techniques (ultrasonication, electrochemical exfoliation, microwave treatment), as well as different fractions of size-selected samples from liquid-cascade-centrifugation were analyzed. This gives insights in defect content, polytype and purity before and after processing (e.g., Figure 1). Further, systematic changes with nanosheet layer number can be observed as expected from theory [1].

References

[1] Molina-Sánchez, Hummer, Wirtz, *Surf. Sci. Rep.*, 70,4, (2015) 554-586.

Figures

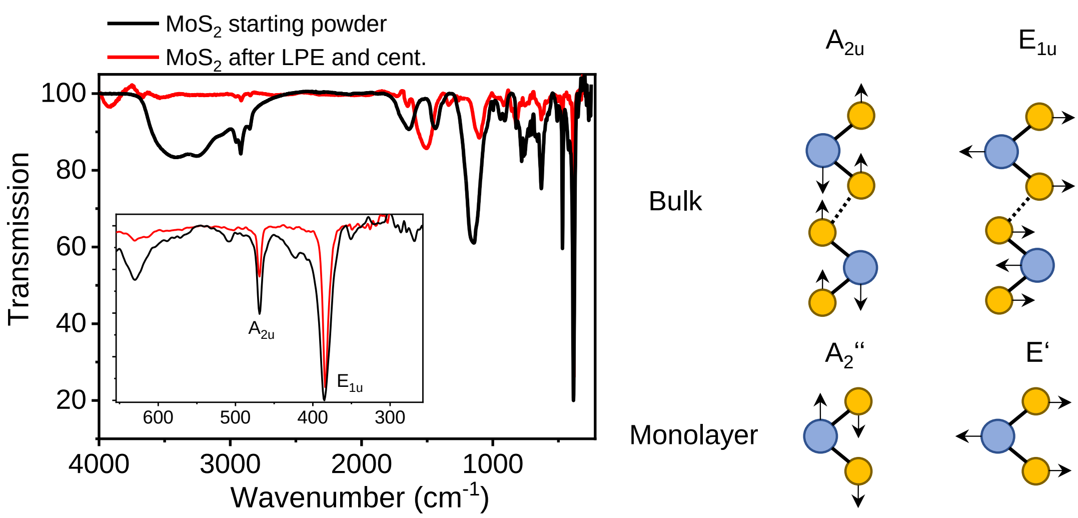

Figure 1: MIR/FIR Spectrum of MoS_2 powders before and after liquid phase exfoliation and calculated FIR modes according to ref [1].