Applying Diffuse Reflectance Mid & Far IR Spectroscopy to monitor additive adsorption and reactivity for liquid-phase 2D materials

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Diffuse reflectance infrared Fourier transform spectroscopy (DRIFT) has become a widely applied vibrational spectroscopy technique for loosely packed powders, but is rarely used for 2D materials. Starting from 2D material dispersions (here especially MoS_2) we recently developed a freeze-drying protocol that allowed us to show that DRIFT powder-spectral information in the mid-infrared to far-infrared can be used to semi-quantitatively assess physisorbed or chemisorbed organic molecules on MoS_2 nanosheets exfoliated by different liquid phase exfoliation techniques. Having access to information on nanosheet surface purity for example enables monitoring of the removal of excess surfactant in centrifugation protocols or the assessment of purification efficiency, e.g. for typically strongly adsorbing polymers such as Polyvinyl Pyrrolidone (PVP) – a stabilizer commonly used in electrochemical exfoliation.[1]
In addition to the analysis of additive adsorption, we now show that also nanosheet surface related reactions in pure solvents can be traced with this technique: Specifically, N-Methyl-2-pyrrolidone (NMP) – a common well-known solvent in liquid phase exfoliation – is known to degrade and/or polymerize under certain circumstances.[2, 3] Adsorption of the reaction products to MoS_2 surfaces has been reported.[3] However, reaction products were not clearly identified in systems of sonication-based exfoliation until now. Here, these adsorbents are further characterized and sonication conditions identified that avoid the formation of difficult to remove surface adsorbates from exfoliation in NMP. This demonstrates the broad applicability of DRIFT as a powerful, thus far little explored technique in the field of liquid-phase exfoliated 2D materials.

References

[1] Carey T, *et al.* 2023 *ACS Nano*, **17**(3)**:** 2912-2922.
[2] Ou YJ, *et al.* 2017 *IOP Conference Series: Earth and Environmental Science*, **100**(1)**:** 012036.
[3] Sim DM, *et al.* 2017 *ACS Omega*, **2**(8)**:** 4678-4687.

Figures

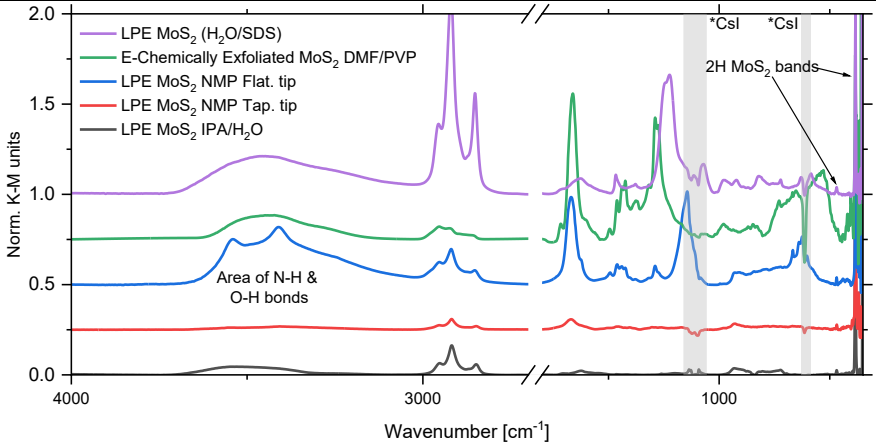

Fig. 1: FIR-MIR Spectrum of differently processed MoS_2 powders from exfoliation in liquids.