ESRF	**Experiment title: Nucleation and Growth Phenomena of Oriented HKUST**	**Experiment number**: 26-02-825
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Shifts: 15	**Local contact(s)**: **Daniel Hermida Merino**	*Received at ESRF:*

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Report:

Summary

During our first SAXS beamtime at the DUBBLE, the feasibility of multiple research projects was evaluated. Besides the GISAXS-GIWAXS experiments on *ex-situ* grown oriented HKUST-1 MOF thin films, interesting *in-situ* SAXS-WAXS data is collected on zeolite particle nucleation in the solution phase of while forming ethanol- and TPA-directed ZSM-5 zeolite thin films. In addition, *in-situ* DSC-SAXS measurements were performed to monitor the coke removal in meso- and micropores of Fluid Catalytic Cracking (FCC) particles.

Experiments

Experiments were performed both in GISAXS-GIWAXS configuration as well as in SAXS-WAXS mode. Below, a list can be found of experiments done:

1. Ex-situ GISAXS on oriented HKUST-1 thin films with increasing number of deposition cycles. An increasing number of cycles is expected to induce increasing number of nuclei accompanied by an average size increase. However, the data shows limited sensitity to island formation, likely due to the low electron density of the formed islands.

2. *In-situ* SAXS-WAXS during the nucleation and growth of Co-ZIF-67 with different $Co(NO_3)_2.6H_2O$/2-methylimidazole ratios. While scattering contrast could be obtained for this sample type, a smaller q-range was required since the particles formed appeared not to completely fall within the detected range.
3. *In-situ* SAXS-WAXS during the nucleation and growth of ZSM-5 zeolite thin films. The solution phase on top of the thin film was measured for particle nucleation in the liquid mixture. In Figure 1 (left and right), background subtracted SAXS patterns are plotted at different points in time (collection time per scan is 30 seconds) for ethanol- and TPA-directed zeolite synthesis growth processes. Multiple discrepancies can be substantiated from the scattering evolutions and scattering curves. First the scattering intensities for ethanol-directed growth is an order of magnitude higher compared to TPA-directed growth, implying that more particle nucleation occurs in the formed synthesis method – which will evidently affect the thin film growth process. In addition. The particle sizes for ethanol-directed synthesis appear larger (lower q-range) relative to TPA-directed synthesis particles. In conclusion, ethanol- (resp. TPA-) directed growth induces more (less) smaller (larger) particles. Guinier analysis will be applied to extract quantitative information.
4. *In-situ* DSC-SAXS-WAXS experiments were performed during temperature-programmed heating of coked FCC particles to interrogate in which q-range (and therefore pore range) coke burnout occurs. It was obtained that 10% less scattering is obtained in the micropore regime, indicating that coke after reaction mainly resides in the micropore network of the zeolites. The DSC curve also confirms that the coke has been burned around the expected temperature of 723 K.

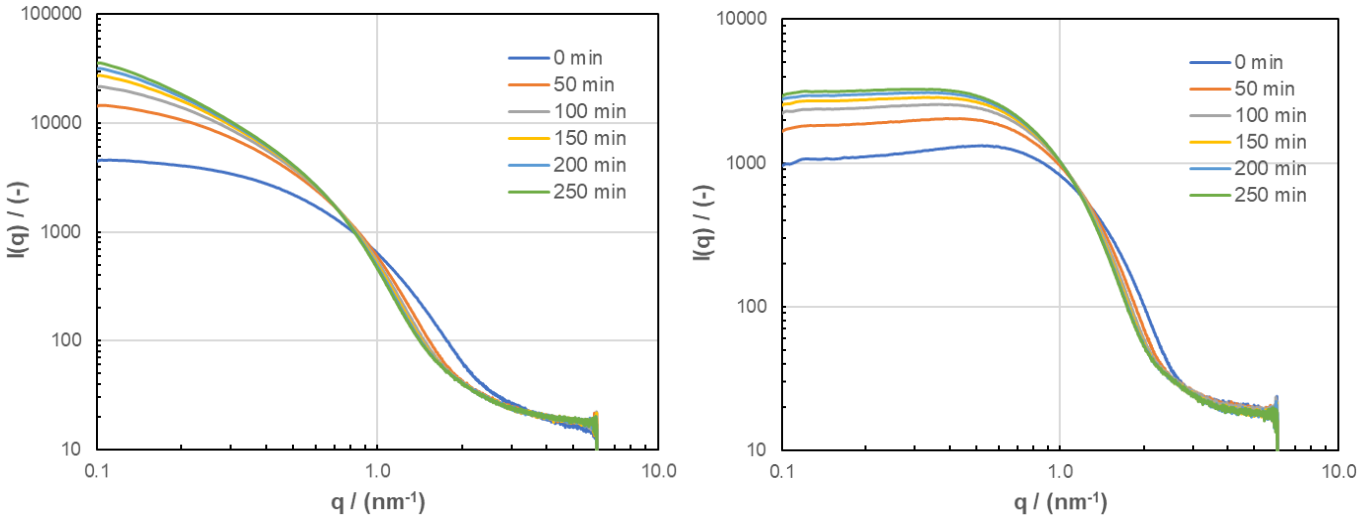

Figure 1: *in-situ* SAXS data measured through the liquid phase of a mixture used for (left) ethanol- and (right) TPA-direct ZSM-5 zeolite thin film growth.