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Raman spectroscopy for in-situ characterization of interzeolite conversion during synthesis

Interzeolite conversion (IZC) is a common phenomenon occurring during zeolite synthesis that can lead to undesirable byproducts [1]. Commercial production of metastable zeolites relies on ex-situ X-ray diffraction (XRD) for synthesis monitoring. XRD allows to quantify the fraction of different zeolite structures [2]. However, this requires sample extraction, preparation, and analysis by qualified operators, which is inconvenient, time consuming and prevents from stopping the reaction at the optimal moment. Here we propose Raman spectroscopy as alternative, in-line, process control technology to speed up sample analysis and simultaneously probe solid and liquid phases. Chemometric analysis of the Raman spectra allow to establish a direct quantitative relation between in-situ Raman and ex-situ XRD data, and therefore obtain similar information. Furthermore, we show this use case to demonstrate harmonization between different Raman spectroscopy systems.

References

[1] Mallette et al., JACS Au, 2, 10 (2022), 2295–2306
[2] Beato et al., Springer Handbook of Advance Catalyst Characterization. Springer Handbooks. Pp 541-563

Figures

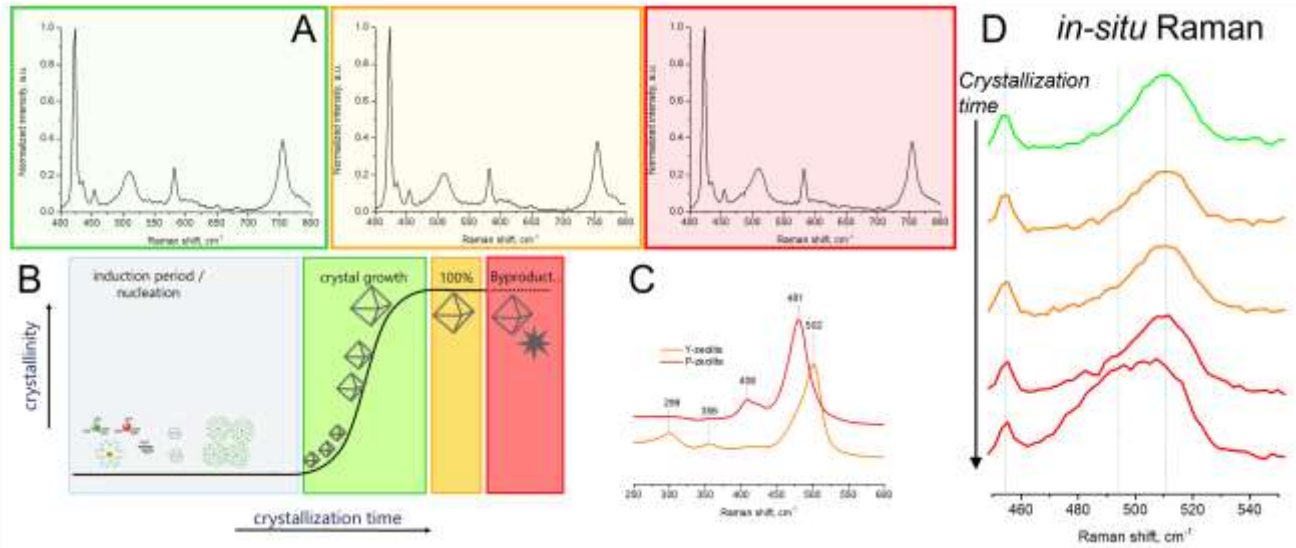

Figure 1: Raman spectroscopy spectra during different crystallization stages. The color code (green = early stage; orange = desired product; red = product of IZC) is maintained for the different figure panels. *In-situ* Raman spectra during different crystallization stages (A); schematic overview of crystallization process (B); *ex-situ* Raman spectra of Y zeolite (desired product) and P-zeolite (IZC product) C); *in-situ* Raman spectra zoom into zeolite relevant spectral area (D).