Phase transition detection of CO_2 through near infrared spectroscopy

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Near infrared (NIR) spectroscopy is a widely used analytical method for online monitoring of chemical processes. Often, combination with chemometric methods is required to capture actual changes that elude inspection by eye during processes, such as chemical reactions and phase transitions, or to quantify components within complex spectra. Temperature changes may result in shifts in position and changes in the intensity of absorption bands. It is, therefore, possible to analyze these spectral changes and correlate them with actual chemo-physical properties, such as variations in the state of aggregation, thus to phase transitions.

In this work, we studied the behaviour of carbon dioxide at high temperature and pressure s in an autoclave. The low critical point for CO_2 allowed us to span from vapor phase to supercritical phase. An immersion probe was installed in the autoclave and connected to the spectrometer through optical fibers. Spectra were acquired continuously every 30 s in temperature ramp. The spectra in Figure 1a collected during heating were analyzed with Principal Component Analysis (PCA) without data pre-processing. The score plot of PC1 vs. PC2 (explaining 99.8% of the total variance, Figure 1b) revealed the separation of the data into three different clusters attributable to different states of aggregation. The changes in the score plot are associated with changes in temperature and in peak shape and area. In particular, the three clusters identified by PCA characterize the different phases of CO_2 during heating from liquid phase to supercritical phase.

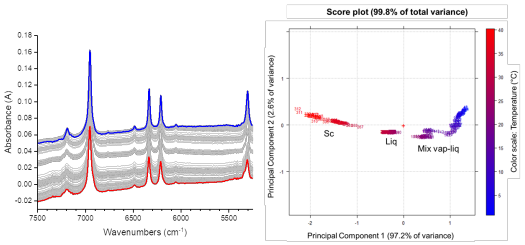

Fig 1: a) NIR spectra obtained during heating liquid CO_2 from cold (blue) to high temperature (red). b) Score plot of the entire dataset identifying three main data clusters.