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Multivariate PAT Monitoring of Sodium Phosphate Solubility and Crystallization In Alkaline Media

Viviana Cardenas Ocampo

Georgia Institute of Technology

ABSTRACT

Crystallization in alkaline process streams is challenging to monitor because solubility, aqueous speciation, hydrate form, and crystal morphology can evolve simultaneously with temperature, and pH. Motivated by phosphate-bearing alkaline waste streams relevant to nuclear waste processing, this work contributes to the development of real-time analytical and modeling tools for crystallization-prone process systems. We develop a data-rich Process Analytical Technology framework that integrates in situ spectroscopy, particle monitoring, temperature and pH measurements with multivariate calibration to quantify phosphate species and track solubility and crystallization behavior in real time. In situ experiments were conducted to characterize sodium phosphate solubility and crystallization under two chemical regimes: strongly alkaline conditions, using 3 molal NaOH, and unadjusted-pH conditions without added NaOH. These conditions provide access to different phosphate speciation regimes. Online ATR-FTIR spectroscopy was used to monitor solution-phase phosphate concentration, while Raman spectroscopy provided complementary information on phosphate speciation and solid-phase formation. Focused Beam Reflectance Measurement was used to track particle chord-length distributions, and EasyViewer imaging provided direct visualization of crystal morphology during heating and cooling. ATR-FTIR spectra were collected during controlled heat–cool experiments for sodium phosphate systems in water and alkaline media. The 910–1155 cm^{-1} spectral region was selected because it contains the main phosphate vibrational bands, including overlapping contributions from HPO_4^{2-} and PO_4^{3-}. Focused beam reflectance measurement was used to distinguish clear-solution and slurry regions, enabling spectra from fully dissolved conditions to be selected for calibration and validation. Partial least squares regression was then applied to extract concentration information from the full spectral response rather than relying on a single peak height. The resulting multivariate calibration provides a foundation for real-time estimation of phosphate concentration during dissolution and crystallization. The calibrated models were further used to generate temperature-dependent solubility profiles from continuous in situ measurements. These results demonstrate how data-rich PAT experiments and multivariate modeling can support real-time monitoring of solubility, supersaturation, and crystallization risk in complex alkaline process streams.

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