Process Analytical Technology in Food: Rheo-Raman Monitoring of Milk Coagulation as a Case Study

Leonardo Sibono^{1*}, Stefania Tronci¹, Martin Aage Barsøe Hedegaard², Massimiliano Errico², Massimiliano Grosso^{1,2}

Process Analytical Technology (PAT) is gaining increasing relevance in advanced food manufacturing, where the real-time acquisition of critical process parameters is fundamental to ensuring product consistency and quality, offering rapid, non-destructive feedback to support data-driven decision-making. Numerous unit operations in food processing (e.g., fermentation, and enzymatic coagulation) involve complex physicochemical dynamics, where continuous and precise monitoring is essential. One example is given by traditional cheesemaking, where quality control methods (manual inspections, rheological tests, pH measurements) are intrusive and unsuitable for industrial environments. In this context, non-invasive optical technologies like NIR and Raman spectroscopy offer the potential to develop innovative effective PAT tools to address these limitations. Raman spectroscopy, in particular, provides molecular-level sensitivity and enables simultaneous monitoring of structural and compositional changes in complex food matrices under real-time conditions.

In this study, use of Raman spectroscopy coupled with multivariate statistics has been explored for in-line monitoring during milk renneting to characterize the molecular and rheological transitions of dairy matrices. Spectral data were processed using Principal Component Analysis (PCA), which identified latent structures corresponding to the dominant physicochemical mechanisms. These were subsequently modeled using a pseudo-kinetic approach adapted from Carlson et al. (1987), capturing the effects of time, temperature, and enzyme concentration on the coagulation process. The proposed methodology demonstrates the capability of Raman spectroscopy, integrated with chemometric modeling, to non-invasively predict both rheological evolution (e.g., gelation and viscoelastic property development) and molecular transformations (e.g., casein aggregation and bond formation). This highlights its potential for robust PAT deployment in dairy process control, offering a step change toward real-time quality assurance in enzymatic coagulation processes.

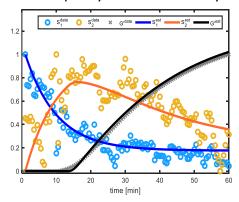


Figure 1: Time-dependent profiles of the first two principal component scores (derived from Raman spectra) and the reconstructed elastic modulus (G'), computed via a compartmental model based on Carlson et al. (1987). Experimentak data points are shown for comparison.

Keywords: PAT, Raman Spectroscopy, Multivariate analysis, Food processing, kinetic modelling **References:** Carlson A, Hill CG, Olson NF (1987) The kinetics of milk coagulation: IV. The kinetics of the gel-firming process. Biotechnol Bioeng 29:612–624. https://doi.org/10.1002/bit.260290510

¹ Dipartimento di Ingegneria Meccanica, Chimica e dei Materiali, Università degli Studi di Cagliari, Via Marengo 2, Cagliari, Italy;

² Department of Green Technology, Southern Denmark University, Campusvej 55, Odense, Denmark E-mail: leonardo.sibono@unica.it