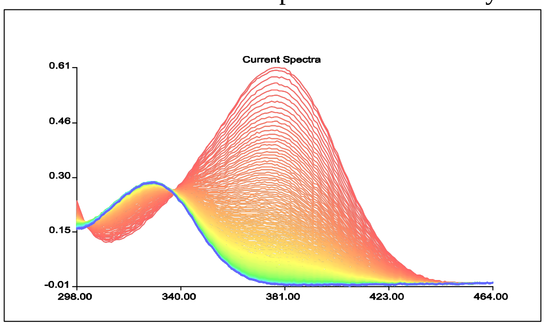
ABSTRACT: The Application of Self-Modeling Curve Resolution to a Batch Reaction Using IntelliFORM and the HEL Automate system.

INTRODUCTION: Self-Modeling Curve Resolution (SMCR) techniques have long been used as a means of analyzing complex data sets that have overlapping absorption bands. These methods work by mathematically decomposing complex data sets into estimates of concentration profiles and pure component spectra that represent the reaction components. The reaction profiles can help characterize the reaction and the technique can be used on multiple reactions to help determine which reaction produce more or less product and which reactions have faster or slower reaction rates.

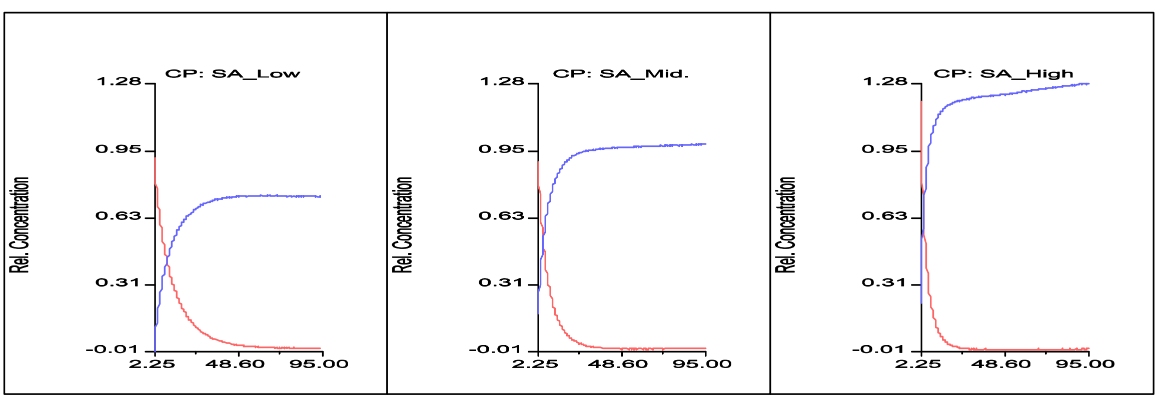
EXPERIMENTAL: The acetylation of salicylic acid with acetic anhydride and phosphoric acid as a catalyst was chosen as a test reaction due to its overlapping absorption bands and its exothermic nature.

RESULTS: The UV/Vis profile for the salicylic acid acetylation reaction is shown below. As can be seen,

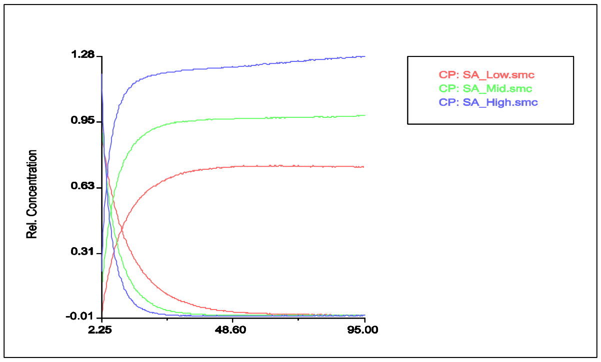


the spectra are complex due to overlapping absorption bands in the UV/Visible range. For this study, three acetylations were performed at different temperatures and different amounts of salicylic acid. This gave a varying set of reactions that could be used to illustrate the abilities of IntelliFORM to show which reactions produced more or less product and which reactions had faster or slower reaction rates. The HEL system was important for these reactions due to the exothermic nature of the acetylation reaction. The HEL system was capable of keeping the reaction at a constant temperature, which helps reduce spectral drift associated with temperature fluctuations in the reaction. The three reactions that were run were labeled SA_Low, SA_Mid, and SA_High. The SA_Low reaction had a reaction temperature of 55°C and had a mole ratio of 0.8 to 1.0 for salicylic acid to acetic anhydride. The SA_Mid reaction had a reaction temperature of 65°C and had a mole ratio of 1.0 to 1.0 for salicylic acid to acetic anhydride. The SA_High reaction had a reaction temperature of 75°C and had a mole ratio of 1.2 to 1.0 for salicylic acid to acetic anhydride.

CONCLUSIONS: With IntelliFORM, the data was preprocessed and appropriate wavelength and time ranges were selected for the different reactions. Additionally, the reactions were baseline corrected and any outlier spectra were removed. Each reaction was analyzed separately and then the three reactions were analyzed using a multi-way approach. The results can be seen below. The blue profile for each reaction



represents product formation for the reactions and the red profile represents reactant use by the reaction. As can be seen from the results, an increase in the amount of reactant increased the amount of product produced in the reaction. The concentration profiles show relative concentrations because the SMCR routines do not require a user to supply concentration values for the reactants. However, the differences in the relative concentration profiles closely match the initial concentration ratios present for the three reactions. The differences in reaction rates are apparent by looking at an overlay plot of the different reaction profiles. For this plot, the reactions are color coded with the SA_Low reaction being red, the SA_Mid reaction being green, and the SA_High reaction being blue. From the plot, it can be seen that the SA_High reaction has the fastest reaction rate and the SA_Low reaction has the lowest reaction rate of the three reactions. This is expected due to the different reaction temperatures of the three reactions. This set of reactions shows the usefulness of Self-Modeling Curve Resolution in determining which batch reactions produce more product and which batch reactions produce product at a faster rate.



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