How to optimize Reactor Efficiency with Inline FT-NIR

Example: Pesticide Production

Application Note N529

Minimizing waste and optimizing efficiency are increasingly important in the chemical industry, where raw materials can account for 80-90% of the variable cost of production[1,2]. As a result, interest in implementing more effective process controls continues to increase. FT-NIR can be a powerful tool for process optimization in many chemical reaction processes, helping control costs and improve product quality.

Methods of chemical production vary widely across industries. From the manufacture of artificial sweeteners to synthetic polymers and industrial pesticides, manufacturing processes are as varied as the chemicals themselves. One commonality among all manufacturers is a desire to reduce costs and improve operational efficiency. This can be a challenge in complicated multi-step process syntheses where reaction kinetics can be difficult to control. Manufacturers must often resort to using excess reactants or longer run times to ensure the complete formation of final products. This can result in lost time, wasted raw materials, and reduction in product quality.

The use of inline FT-NIR analyzers (Figure 1) provides manufacturers an accurate and reliable method for monitoring the entire reaction process.

Potential Applications:
In addition to pesticide production, there are numerous additional applications where FT-NIR can be used for process reaction control.
Some of these include:

- Polymer synthesis
- Resin production
- Powder and liquid blends
- Pharmaceutical and botanical blending
- Bio-Fuels and Fermentation
- Petrochemical processes

Figure 1: MATRIX-F II FT-NIR Process Spectrometer for checking important process parameters immediately for reaction monitoring and control.

Results

The results below are from a feasibility study of a batch reactor process for pesticide production. While specific mechanistic details are not disclosed here, the essential reaction proceeds when two feedstock chemicals (compound A and B) are added together in a solvent mixture. An electrophilic reactant is then added to initiate formation of the final product (compound C).

Figure 2 depicts the results of a conformity measurement for determining the reaction endpoint. The results were obtained using a Bruker MATRIX-F II process analyzer with an IN271 transflectance probe. Upon reactant addition, the reaction progressed towards its endpoint, with the conformity values decreasing until they fell below the maximum conformity index; the threshold for determining whether a sample "passes" or "fails". The sensitivity of the measurement is clearly illustrated by the data points representing measurements 45 through 55. This is the point where the reactant feed was stopped, halting the formation of final product. When reintroduced, the reaction continued to completion.

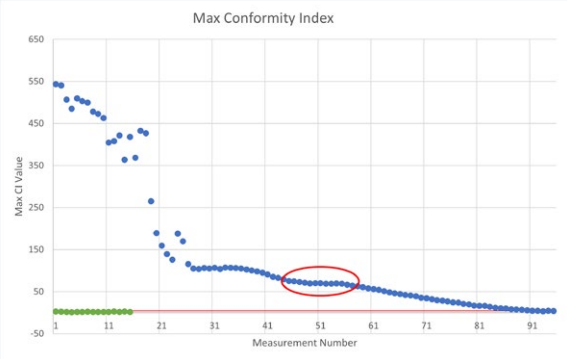

Figure 2: Graph of conformity for the reaction. The green dots represent the reference measurements of the known reaction endpoint and are used to establish the conformity index (red line) for the final product. The blue dots represent the measurements of the process as the reaction proceeds.

In addition to conformity, feasibility studies were conducted to determine the suitably of FT-NIR for quantifying the principal reactants involved. The results in Figure 3 suggest a high degree of calibration accuracy for monitoring the reduction of compounds A and B, and formation of compound C in percent concentration. The statistical results for each individual calibration can be found in Table 1.

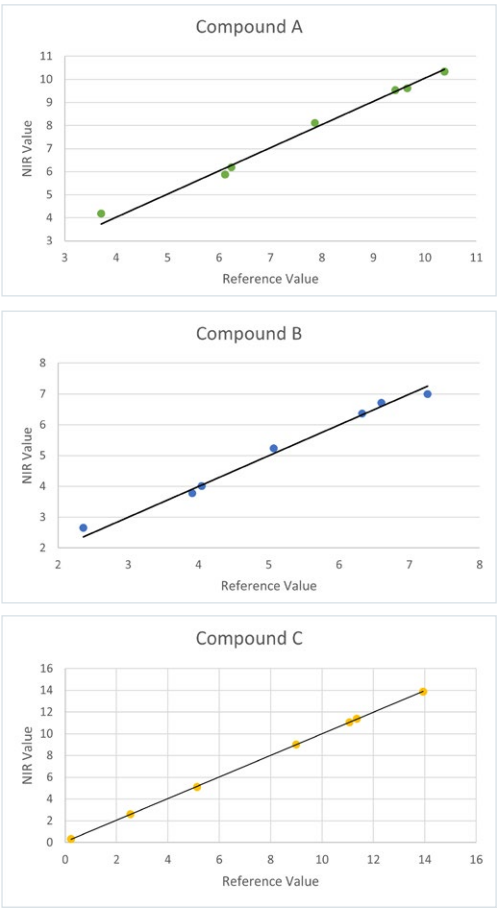

Figure 3: Feasibility calibrations for predicting raw percentages of feedstock material (A and B) and final product (C) during the reaction.

Compound	RMSECV	R^2
A	0.228	98.95
B	0.176	98.20
C	0.049	99.99

Table 1: The RMSECV (error) and R^2 values for the calibration models of compounds A, B, and C, respectively.

References

(1) Pinkerton, N., Schmidt, K., Xamplas, J (2016) "Estimation of Production Cost and Revenue" Retrieved from https://processdesign.mccormick.northwestern.edu/index.php/Estimation_of_production_cost_and_revenue.
(2) Towler G, Sinnott R. (2013) Chemical Engineering Design: Principles, Practice and Economics of Plant and Process Design. 2nd ed. Boston: Elsevier.

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