Application Note

Hydrosilylation Reaction Profiling

Track Reaction Progression and Kinetics

Based on work presented by **Dow Toray Co., LTD**

Real-time Raman measurement of reactants, products and by-products

Applications of silicone stem from its diverse properties, and altering these properties through well controlled chemistry enables creation of fit-for-purpose products. In this application, a silicone compound was synthesized by reacting a siloxane and an olefin (figure 1). The researchers wished to understand the kinetics of this hydrosilylation and determine if in-situ Raman spectroscopy could provide the quantitative conversion rate. In-situ analysis mitigates the need for off-line measurement with its inherent difficulties including irreproducible sampling, sample preparation, and analysis time delays.

To track this reaction, a ReactRaman system equipped with fiber optic probe monitored the Si-H bond of the hydride functionalized siloxane, the C=C bond of the olefin reagent, as well as bonds associated with the final product. Calibration plots (figure 2) were developed in order to track the reaction % conversion rate.

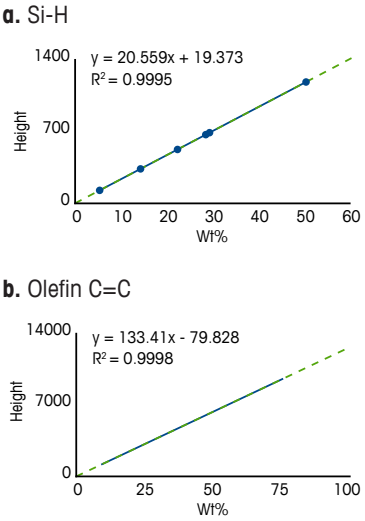

Figure 2. Calibration plots relating weight% with peak height for Si-H and C=C bonds in starting materials.

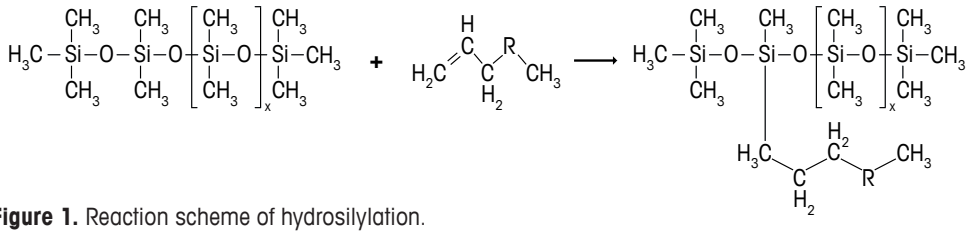

Figure 1. Reaction scheme of hydrosilylation.

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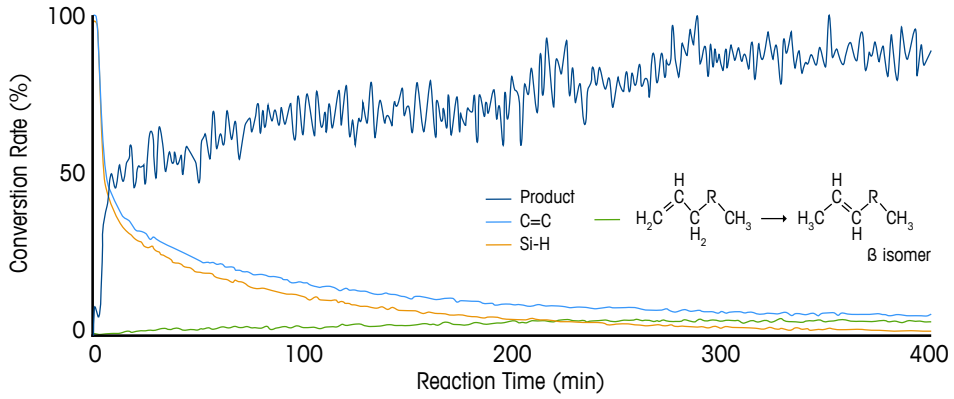


Figure 3. Quantitative tracking of the hydrosilylation of a hydride functionalized siloxane. As the reaction proceeds, individual trends show disappearance of reactants, formation of product and presence of beta-olefin by-product.

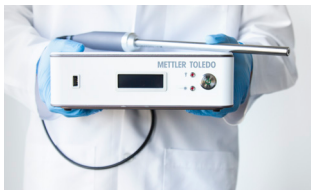
Results

By collecting Raman spectra every 30 seconds (figure 3), profiles for the starting materials and product were obtained that show the presence of two kinetic regimes. Initially, the reaction was very fast with 50 % conversion occurring in the first 30 minutes. After this period, the reaction required nearly 6.5 hr to reach completion. Additionally, Raman measurements detected and tracked an olefin beta isomer by-product.

Conclusions

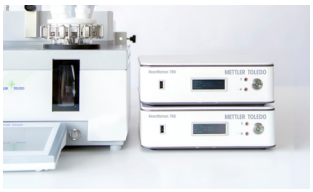
In this hydrosilylation, in-situ ReactRaman effectively tracked % conversion, confirming that the reaction proceeded correctly. Initially, rapid conversion was observed, followed by a period of slow conversion to completion. In contrast to offline analysis, where key data points in a rapid kinetic regime may be missed, real-time analysis provides actionable data and the acquisition of a more complete kinetic profile.

ReactRaman™



Small Footprint, Big Performance
Combine best-in-class performance with a flexible and portable design; delivering outstanding results wherever it's needed.

One Click Analytics™
iC Raman software combines a peak picking algorithm with functional group intelligence to drastically reduce analysis time.



An Integrated Approach
iC software integrates analytics with process conditions to provide comprehensive process understanding and control.

Application of ReactRaman include:

- Reaction Kinetics
- Flow Chemistry
- Crystallization
- Polymorphism
- Polymer Synthesis
- Hydrogenation
- Bioprocess Monitoring
- Biocatalysis

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