PAEONIA INNOVATIONS

Novel Mid-Infrared Spectrometer for Reaction Monitoring

Introduction

Infrared (IR) spectrometers have become an essential tool in the field of chemical reaction monitoring. Their ability to provide real-time data on the concentration of reactants, intermediates, products and side products makes them invaluable for understanding the reaction mechanism, optimizing reaction conditions and ensuring product quality. This application note outlines the experimental setup and results obtained from using Paeonia Novel Mid-IR Spectrometer to monitor the aldol reaction of acetone in the presence of strongly basic ion exchange resin, providing insights into its effectiveness and reliability. Due to the sensor miniature size, it can be easily incorporated into the setup and only 3-5ml of sample is needed for the experiment.

The aldol reaction of acetone yield 4-hydroxy-4-methyl-2-pentanone (Diacetone alcohol; DAA) which can be catalyse by basic ion exchange resin[1]. It is an interesting experiment to demonstrate the application of Novel Mid-IR Spectrometer in reaction monitoring as the DAA can further react to form mesityl oxide and other side products. Through the result, we will be able to know when the maximum yield can be achieved and to stop the reaction before the desire product react further to form unwanted products.

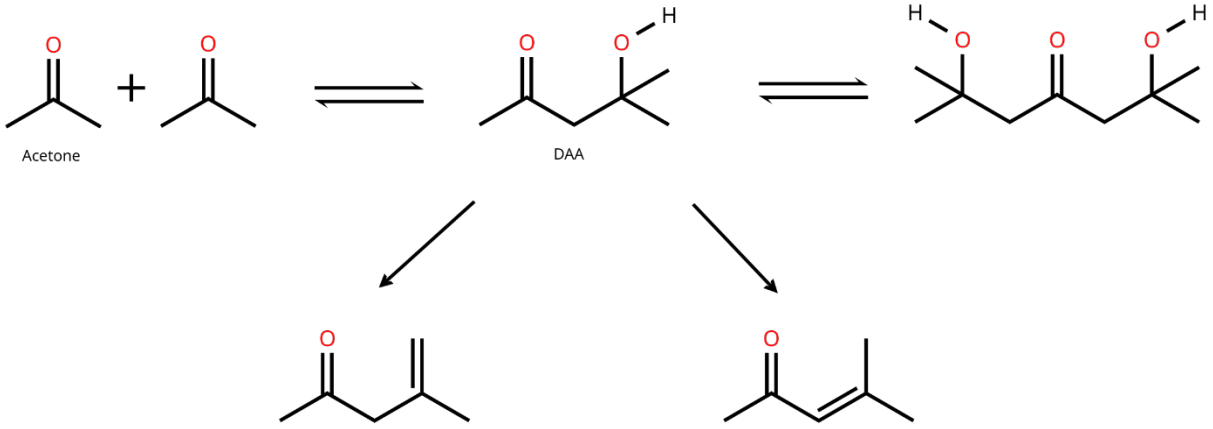

Figure 1: Reaction scheme of acetone to DAA

Experimental Setup

The experiment was conducted using a standard laboratory setup, which included the following components:

1. IR Sensor: Paeonia Novel Mid-IR Spectrometer capable of detecting wavenumber ranging from 900cm^{-1} to 1800cm^{-1}.

2. Calibration sample preparation: twelve DAA in acetone mixture calibration samples (approximately 0 – 25 mass % DAA in acetone) were gravimetrically

prepared in gas-tight crimp sealed glass vial and measured offline to construct the PLS regression model before the reaction setup (see Table 1 below).

3. Reaction setup: A 20ml glass vial equipped with a magnetic stirrer to ensure uniform mixing of reactants. No heating and cooling was needed and the reaction temperature was maintain at room temperature.

4. Data Acquisition System: A computer interface connected to the IR sensor for real-time data collection and analysis.

5. Reagents: Acetone and Amberlyst A 26 OH (basic ion exchange resin).

Index	Theoretical DAA weight %	Experimental weight %
S01	0.00	0.00
S02	0.25	0.26
S03	0.50	0.52
S04	1.00	1.03
S05	2.00	2.00
S06	3.00	3.00
S07	5.00	5.13
S08	7.50	7.54
S09	10.00	10.14
S10	15.00	15.05
S11	20.00	20.03
S12	25.00	25.39

Table 1: Calibration samples preparation

Procedure

To a 20ml crimp-seal vial equipped with magnetic stir bar, acetone (16.6ml) and Amberlyst A 26 OH (1g) were added. The vial was crimp-sealed and the reaction mixture was stirred at room temperature. The vial was connected to a 5.0µm nylon syringe filter, diaphragm pump and Novel Mid-IR Spectrometer through a 1/16'' OD tubing inserted through the cap to create a continuous flow to the spectrometer. Data was collected at regular intervals to monitor the concentration changes of the reactants and products throughout the reaction (2.5 hrs).

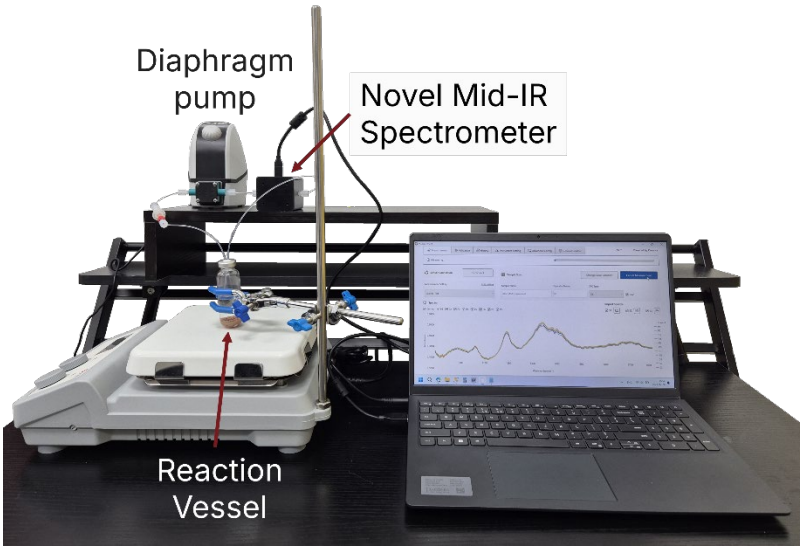

Figure 2: Experiment setup

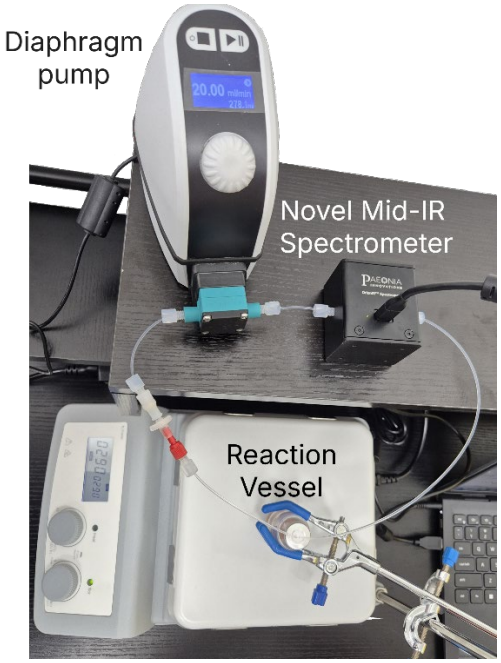

Figure 3: Top view of experiment setup

Results

For the DAA calibration, a total of 12 samples were analyzed, with 48 spectra collected for each sample. Each spectrum represented the average of 16 scans. The calibration focused on the spectral range of 1080 to 1378 cm^{-1}, as this range contains the key features of DAA and acetone. A 5-component PLS calibration model was applied to the dataset, yielding excellent test metrics with an R^2 of 0.999 and an RMSE of 0.20. The parity plot for the PLS model is shown in Figure 4 below.

The reaction data were processed within the same spectral range, and the concentration of DAA was predicted using the PLS model.

The concentration data obtained from the IR sensor during the reaction indicated the following:

- **Reaction Progress**: As the reaction progressed, a decrease in the absorbance of reactants was observed, while the absorbance of the product increased, confirming the conversion of reactants to products.
- **Quantitative Analysis**: The data allowed for the calculation of reaction kinetics, with the rate constant determined from the absorbance changes over time. The reaction reached equilibrium after 60 minutes, as indicated by the stabilization of the absorbance readings, suggesting that the conversion of reactants had stabilized.

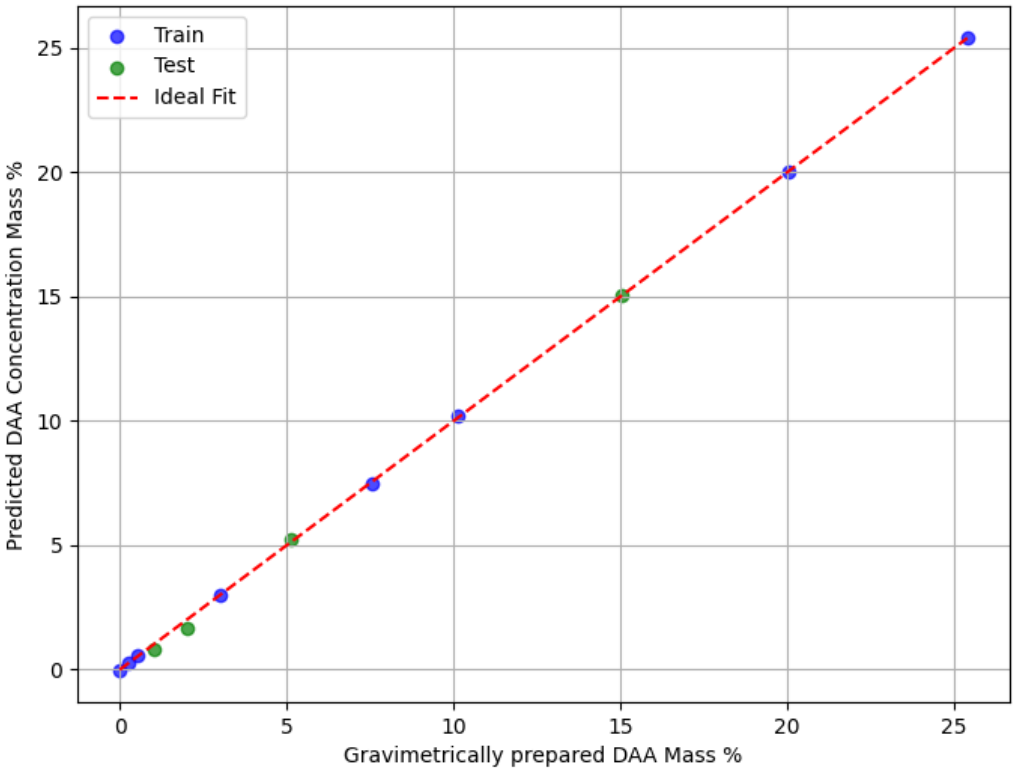

Figure 4: Parity plot of PLS model.

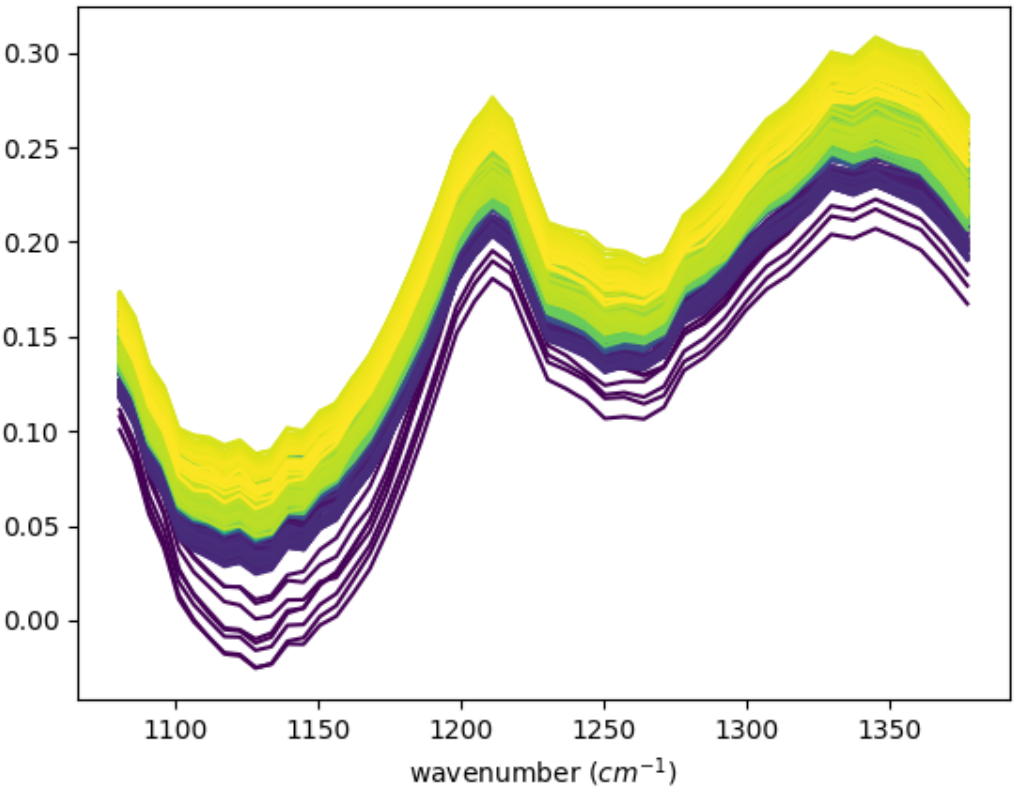

Figure 5: Absorbance spectra of reaction over time (dark purple-start of reaction; yellow-end of reaction)

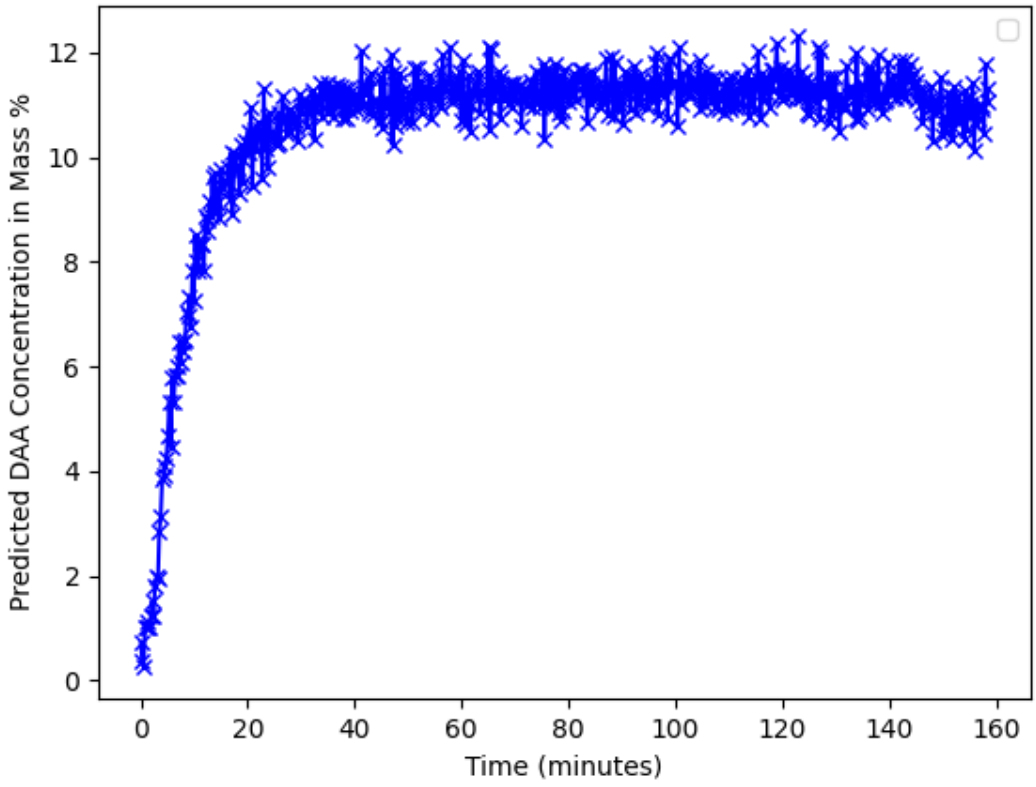

Figure 6: Predicted DAA concentration over time

Key Features

1. Real-Time Monitoring: IR sensors provide continuous data, allowing for immediate adjustments to reaction conditions.

2. Non-Destructive Analysis: The non-invasive nature of IR spectroscopy means that samples remain intact for further analysis.

3. Wide Range of Applications: Suitable for both liquid and gas phase reactions, making them versatile for various chemical processes.

4. High Sensitivity: Capable of detecting low concentrations of reactants and products, ensuring accurate monitoring.

Applications in Reaction Monitoring

1. Kinetics Studies: IR sensors can be used to monitor the progress of reactions over time, providing insights into reaction rates and mechanisms.

2. Quality Control: In industrial settings, IR sensors can ensure that the concentrations of reactants and products remain within specified limits, thus maintaining product quality.

3. Process Optimization: By analyzing real-time data, chemists can optimize reaction conditions such as temperature, pressure, and concentration to enhance yield and efficiency. It can also monitor the reaction endpoint.

4. Safety Monitoring: In reactions that produce hazardous byproducts, IR sensors can detect these compounds early, allowing for timely intervention.

Conclusion

The integration of Novel Mid-IR Spectrometer into reaction monitoring systems offers significant advantages in terms of efficiency, accuracy, and safety. The use of Novel Mid-IR Spectrometer for monitoring the reaction provided valuable real-time data, enabling precise tracking of reactant consumption and product formation. Moreover, the compact size of the Novel Mid-IR Spectrometer enables real-time, on-line analysis of small-scale reactions, thanks to its portability and flexibility. This is especially crucial for experiments that involve valuable or costly reagents, where only small reaction volumes can be used. The ease of integration with other setup components also makes the sensor a convenient analysis tool for experimental setups. For researchers and industry professionals alike, adopting IR sensor technology is a step towards more effective and reliable reaction monitoring.

References

(1) Eisenacher, M., Venschott, M., Dylong, D. *et al.* Upgrading bio-based acetone to diacetone alcohol by aldol reaction using Amberlyst A26-OH as catalyst. *Reac. Kinet. Mech. Cat*. **2022,** *135*, 971–986. https://doi.org/10.1007/s11144-022-02168-z

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