SYRRIS

Atlas Syringe Pump

Batch chemistry application note – Temperature-dependent dosing with Atlas Syringe Pump

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1 Summary

This application note demonstrates how to perform temperature-dependent dosing into a batch reactor using the Atlas Syringe Pump. The example described in this application note uses the Atlas Syringe Pump to automatically dose a sodium hydroxide solution into a batch reactor containing sulfuric acid, pausing when the temperature is outside of a given temperature window.

2 Introduction

Many chemical transformations release or absorb energy, and chemists need to be aware of these factors when planning reactions to avoid thermal run-away or unwanted side reactions.

Temperature-dependent dosing can significantly reduce the risks posed by endothermic and exothermic reactions, pausing reagent addition when the temperature is outside of the optimum reaction conditions, thus avoiding the laborious process of manual reagent addition.

2.1 Equipment

The experiment uses the Atlas Syringe Pump (equipped with 2.5 mL syringes), controlled by the Atlas PC software, dosing into an Atlas HD reactor system. The part numbers for the equipment used are shown below:

- Atlas Syringe Pump (2200072)
- Atlas 1 Software (2300102)
- Atlas HD DN80 Kit (UK/EU) (2201405)

All equipment was set up as shown below:

Figure 1 - Experimental Setup

2.2 Experimental Set Up

Atlas Syringe Pump set up:

Pump fitted with 2.5 mL syringes, with each 4-way valve using port A for aspiration and port B for dispensing, ports C & D were blocked with blanking plugs. Each pump has a maximum fill rate of 10 mL/min, the maximum pump rate was set to 10 mL/min.

Pump channel A – 10 M NaOH(aq)

Pump channel B – Not used

The pump is connected to PC via USB to LEMO cable.

Prior to experimentation, pump channel A was primed to ensure all tubing is full.

PC set up:

In the Atlas PC software, a recipe was designed to control the temperature of the reactor to 20 °C, and once stable, dose NaOH (aq) into the reactor. Dosing was set up to stop once the reactor temperature exceeds 23 °C, and resume dosing when the temperature falls below 20.1 °C. The stirrer was set to run at 300 rpm throughout the experiment.

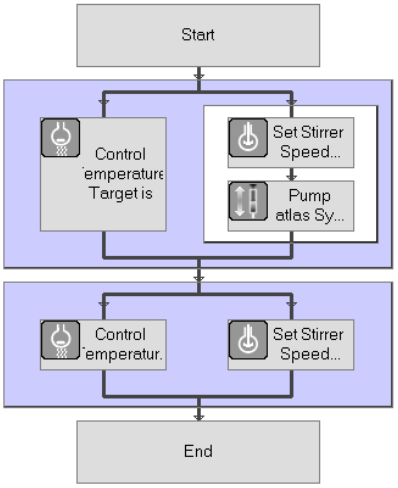

Figure 2 – Temperature-dependent dosing recipe

Atlas HD set up:

An Atlas HD System was set up with a 500 mL torispherical jacketed vessel and propeller stirrer, and filled with 300 mL 5M $H_2SO_{4\ (aq)}$. A temperature probe was placed into the vessel. The dosing line from the Atlas Syringe Pump was fed through the vessel lid.

Circulator set up

A Huber Unistat 405 circulator was connected to the PC via Atlas Port (2101020).

2.3 Method

With the equipment set up as described above, the experiment was started.

Following the addition of NaOH, reactor temperature will begin to rise and once the temperature exceeds 23 °C the dosing will pause. The circulator will continue to control the reactor temperature, and dosing will resume when the temperature falls below 20.1 °C.

3 Results

The reactor temperature, circulator temperature and cumulative dosed volume of NaOH (aq) for the temperature-dependent dosing experiment are shown in the graph below:

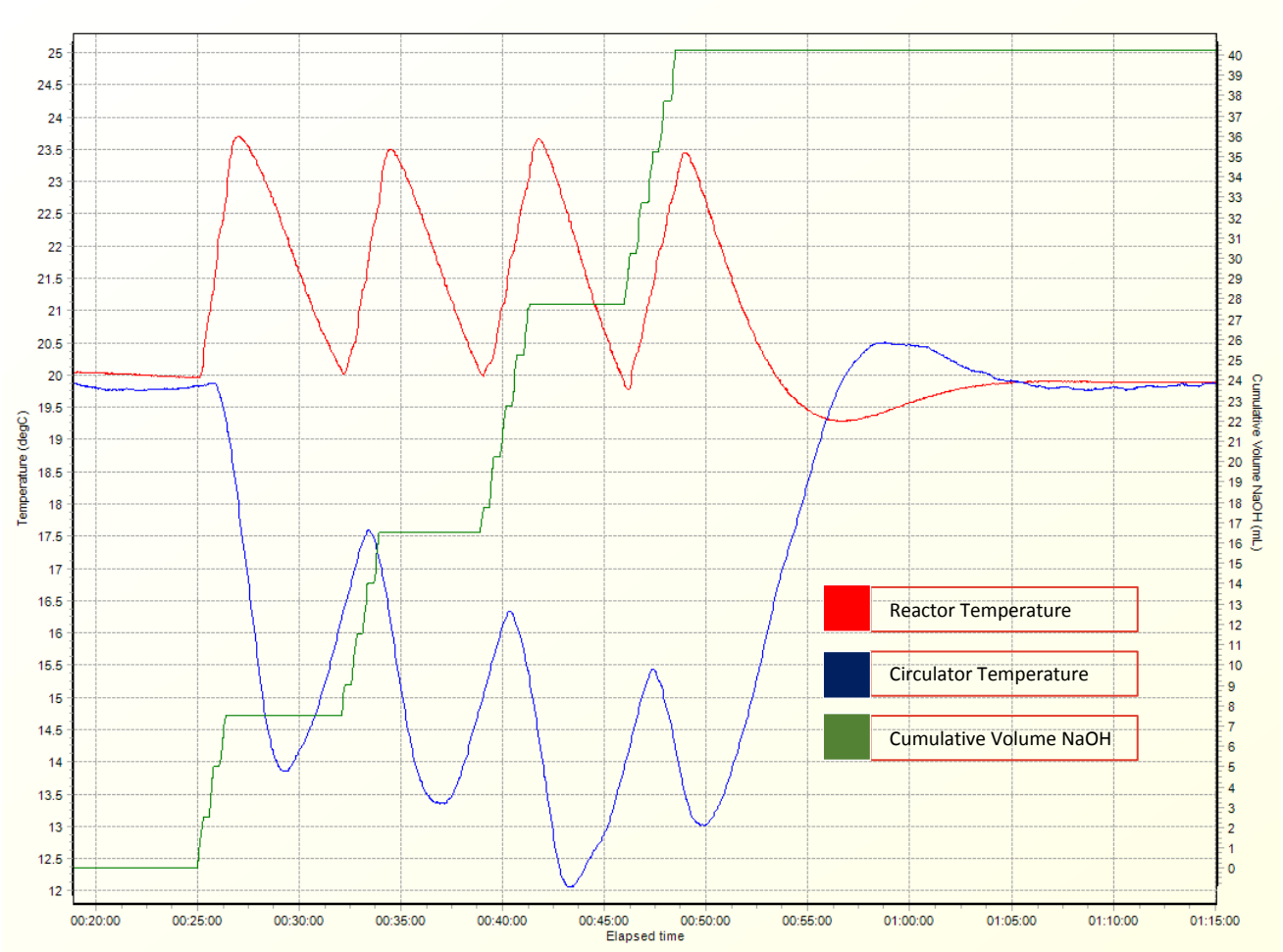

Figure 3 – Temperature-dependent dosing graph

After starting the experiment, NaOH is dosed into the vessel containing H_2SO_4, the exotherm created raised the temperature of the reactor. When the reactor temperature exceeded 23 °C, dosing ceased and the system was actively cooled back to 20 °C by the circulator. Once the temperature fell under 20.1 °C, dosing recommenced, and this process was repeated another 3 times. At the end of the experiment the temperature was allowed to settle back at 20 °C.

4 Conclusion

As demonstrated, the Atlas Syringe Pump can be used to automatically mitigate the effect of exotherms created when dosing, by dosing only within a defined temperature window. Whilst only an exothermic process has been shown in this application note, the same technique can be applied to endothermic reactions, saving valuable time and effort over manual reagent addition.