Atlas Syringe Pump XL

Batch chemistry application note – pH control of an un-buffered solution using the Atlas Syringe Pump XL

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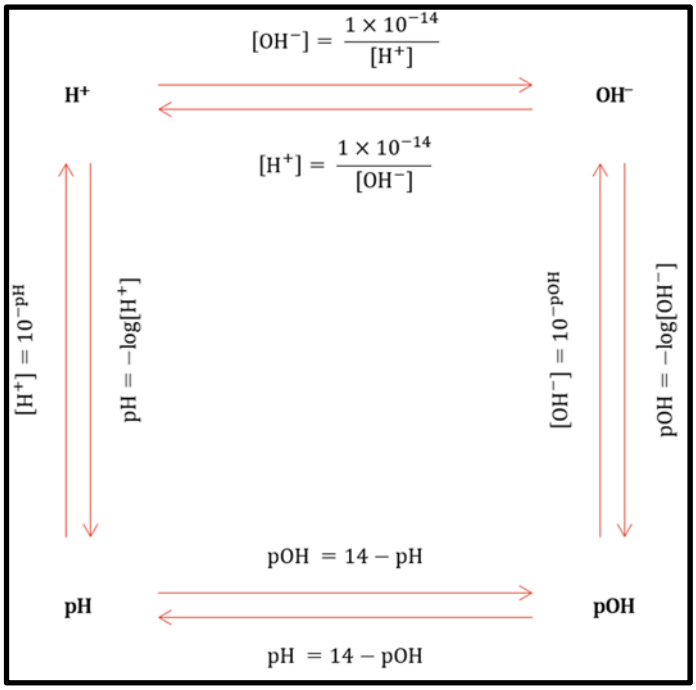

1 Summary

This application note demonstrates how to perform pH control of a batch reactor using the Atlas Syringe Pump. The example described in this application note uses the Atlas Syringe Pump to automatically dose solutions of sodium hydroxide and sulfuric acid to control the pH of 300 mL unbuffered water to pH 7.

2 Introduction

A wealth of chemical and biological transformations exist that show strong correlation between pH and rate of reaction[1]. Many starting materials, reactive intermediate and products are also sensitive to acids/bases, so accurate pH control is essential for reactions to progress. Automated pH control can save chemists valuable time, by removing the need to manually add small amount of acid/base over the course of a reaction.

2.1 Equipment

The experiment uses the Atlas Syringe Pump XL (equipped with 25 mL syringes) with pH node and probe connected, 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 XL (2200376)
- pH and Temperature Node (2200071)
- Node Extension (2101023)
- pH probe – 400mm x 12mm with Swivel Adaptor (2200029)

All equipment was setup as shown below:

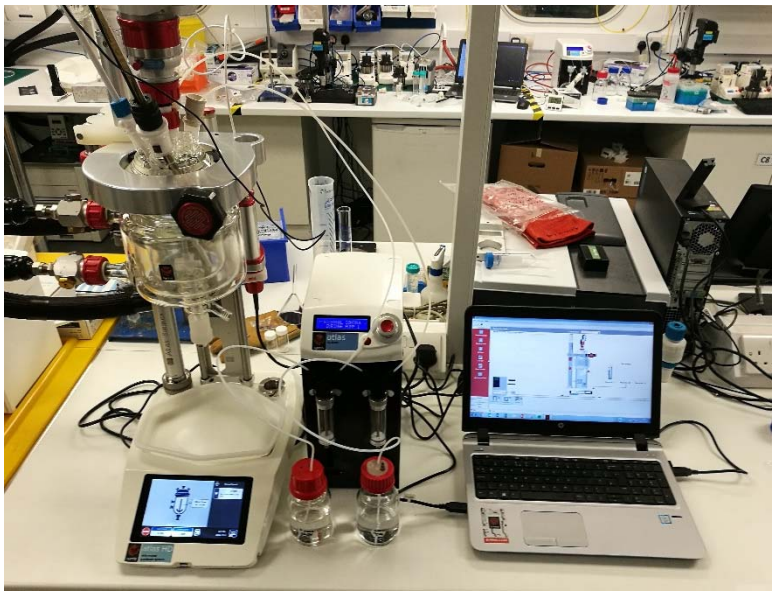

Figure 1 - Experimental setup

[1] Johnson L.L., Pavlovsky, A.R., Johnson, A.R., Janowicz, J.A., Man, C-F.M., Ortwine, D.F., Purchase, C.F., White, A.D. and Hupe, D.J., (2006) A rationalization of the Acidic pH Dependence for Stromelysin-1 (Matrix Metalloproteinase-3) Catalysis and Inhibition, J. Biol. Chem., 275 (15), 11026-11033

2.2 Experimental setup

Atlas Syringe Pump XL setup:

Pump fitted with 25 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 50 mL/min, the maximum pump rate was set to 4 mL/min.

Pump channel A – 0.001 M NaOH(aq)

Pump channel B – 0.001 M H_2SO_4(aq)

The pump is connected to PC via USB to LEMO cable. A node extension, pH node, and pH probe were attached to the rear of the syringe pump.

Prior to experimentation, each pump channel was primed and the pH probe was calibrated using buffer solutions of pH 4.05 and pH 7.00.

PC setup:

In the Atlas PC software, a recipe was designed to control the pH of 300 mL water to pH 7, with a dead-zone of 0.5 (pH will be controlled outside of the range pH 6.75-7.25), with a maximum combined dosed volume of acid/base of 200 mL. The stirrer was set to run at 300 rpm.

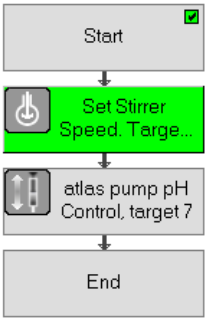

Figure 2 - Atlas 1 Recipe

Atlas HD Setup:

An Atlas HD System was setup with a 500 mL torispherical jacketed vessel and propeller stirrer, and filled with 300 mL water. A pH probe and temperature probe were placed into the water. Each dosing line from the Atlas Syringe Pump XL was fed through the vessel lid.

2.3 Method

With the equipment setup as described above, pH fluctuation (such as those seen during many chemical transformation) were simulated by the addition of 1 mL 0.01M H_2SO_4(aq), as a single portion, to the vessel.

Following the addition of the acid, the Atlas Syringe Pump XL automatically controlled the pH back to 7, where the system was allowed to stabilize.

Following stabilization 3 mL 0.01M NaOH was added, as a single portion, and again the system was allowed to control the pH back to 7 and stabilize.

3 Results

The pH, temperature, and cumulative dosed volumes for the overall pH control experiment are shown in the below graph:

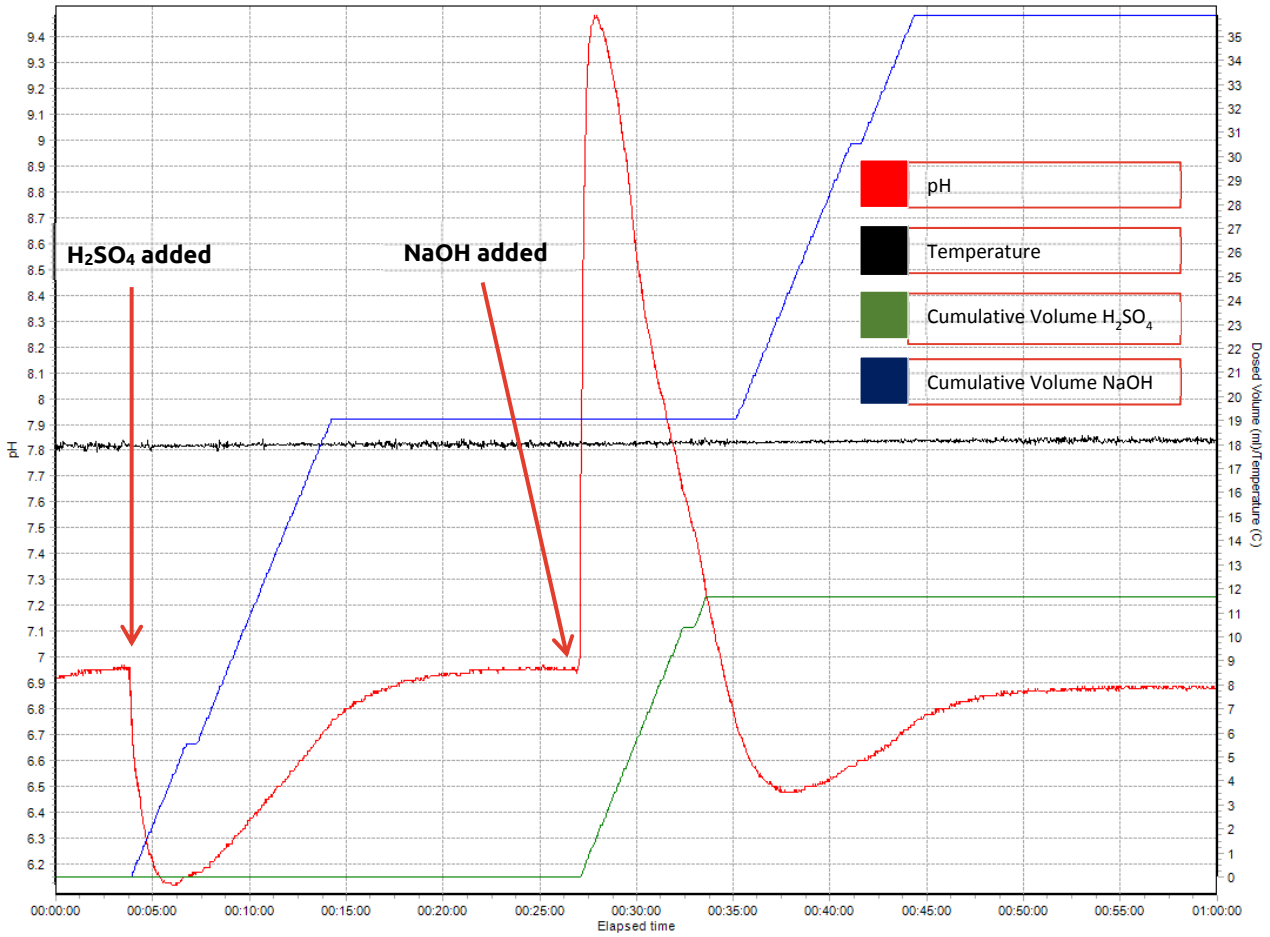

Figure 3 - pH control graph

After the initial addition of NaOH to the vessel, acid is automatically dosed into the reactor from the Atlas Syringe Pump XL. The pump stops dosing acid when it reaches the “dead zone” specified in the recipe (6.75 to 7.25 pH) and, following stabilization, the pH stabilizes within the desired range after just 10 minutes.

Upon addition of H_2SO_4 to the vessel, base is automatically dosed into the reactor from the Atlas Syringe Pump XL. As before, the pump stops dosing base when it reaches the “dead zone”, however due to the molarity of the dosed acid, a small overshoot occurs. The Atlas Syringe Pump XL then doses base to compensate for the overshoot, and after stabilization, the pH returns to the desired range within 20 minutes.

Expanded graphs of each pH control action are shown below:

pH control following NaOH addition:

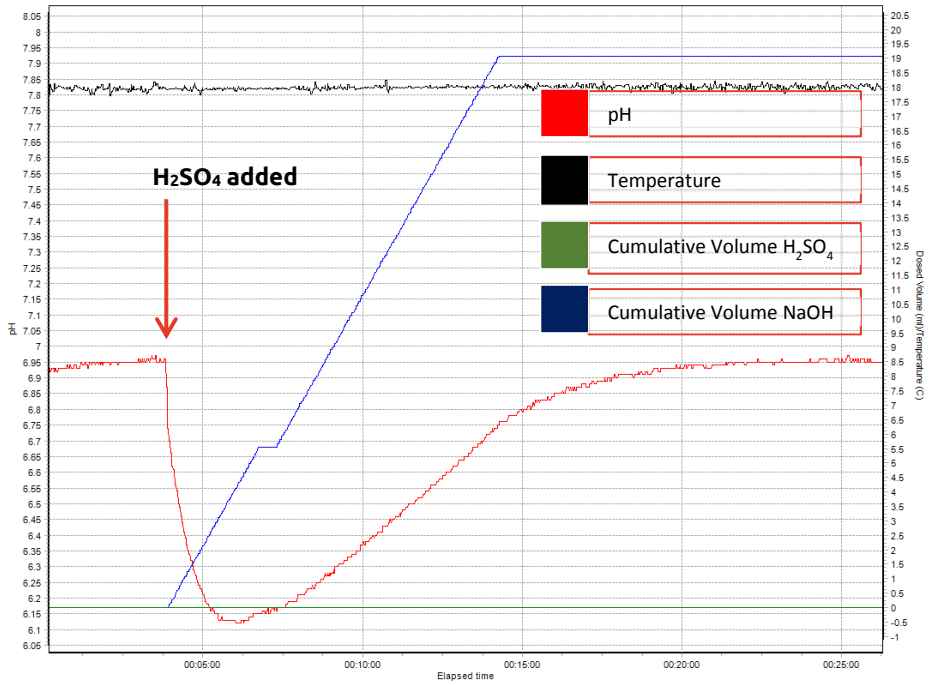

Figure 4 - pH control following base addition

pH control following H_2SO_4 addition:

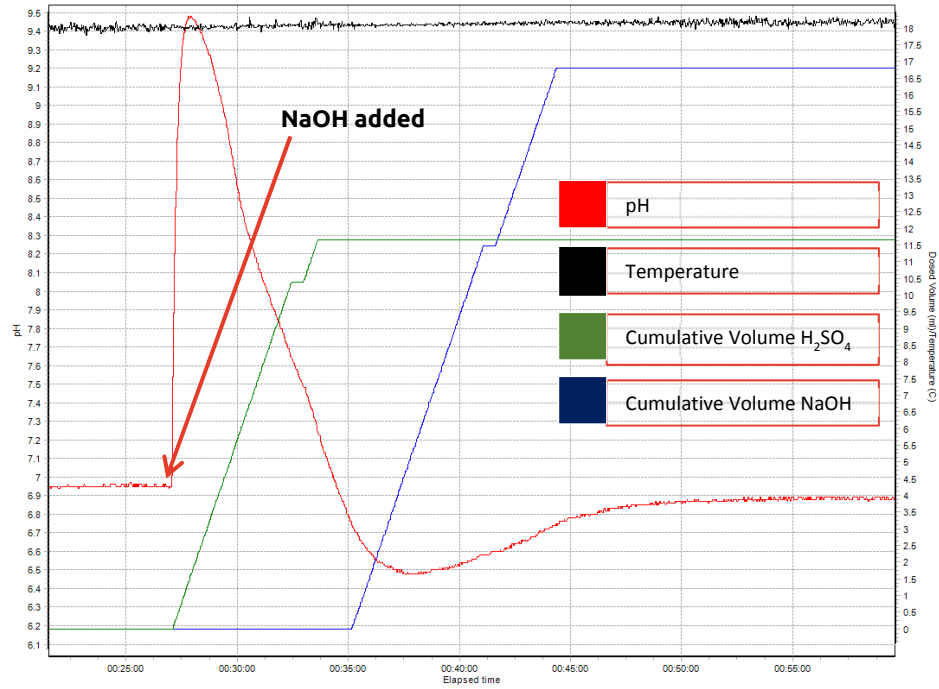

Figure 5 - pH control following acid addition

4 Conclusion

Accurate and rapid pH control has been demonstrated using the Atlas Syringe Pump XL under challenging, unbuffered conditions. Acid and/or base are dosed into the reaction automatically, without the need for user intervention.

The pump can also be used in “standalone” mode, without the use of PC software, allowing pH control with any of your existing reactor systems. Contact Syrris to discuss your chemistry today.