

## PRECISE FLOW-CONTROL USING PHOTO-ACTUATED HYDROGEL VALVES AND PID-CONTROLLED LED ACTUATION

*Colm Delaney*<sup>1</sup>, *Peter McCluskey*<sup>1</sup>, *Simon Coleman*<sup>1</sup>, *Jeff Whyte*<sup>2</sup>, *Nigel Kent*<sup>1,2</sup> and *Dermot Diamond*<sup>\*1</sup>

<sup>1</sup> Insight Centre for Data Analytics, National Centre for Sensor Research, Dublin City University, Dublin 9, Ireland

<sup>2</sup> School of Mechanical and Design Engineering, Dublin Institute of Technology, Bolton Street, Dublin, Ireland

### ABSTRACT

Herein we demonstrate remarkable control of flow within fluidic channels using photo-actuated hydrogel valves. By polymerizing the valves in situ it has been possible to create highly-reproducible valves. Through the use of an LED platform and a PID algorithm we have generated extremely accurate flow control and created prototype devices to document their potential application within the microfluidics field.

### KEYWORDS

**Stimuli-responsive, photo-actuators, hydrogel, valve, flow-control, PID**

Accurately controlling flow using actuators fully embedded within microfluidic devices has long been a hurdle within our research community. While inroads have been made, through the use of pneumatic valves based on soft-polymers[1], there still exists a paucity of low-cost and precise actuators. In recent years, several groups have turned to photo-responsive polymers as a means for fluid handling. Sumaru *et al.* first showed that hydrogels functionalized with a photoactive spiropyran molecule could be used to act as a photo-controlled valve within a microfluidic channel.[2] Conversion between the hydrophobic spiropyran and hydrophilic protonated-merocyanine forms causes the actuation of the polymer gel between swollen and contracted states.

Our group has made significant progress over recent years in the field of photo-actuated spiropyran materials,[3] firstly through the copolymerization of a proton source and more recently through the use of a photo-actuation platform. This achieved simple open/closed control of fluid flow within a microfluidic device which was subsequently improved by pulsing the light source.[4, 5] The work presented herein is a further advance on control of intermediate states

between fully open and closed valves. In particular, through the use of proportional integral derivative (PID) control of power to the illuminating LEDs, we have achieved unprecedented accuracy in controlling flow rate and avoided over/under-shoots upon actuation.[6]

### EXPERIMENTAL

The hydrogel valves were photo-polymerised in-situ from a monomeric cocktail which consisted of 200 mg NIPAAm, 8.35 mg MBIS (3 mol% relative to NIPAAm), 7.91 mg SPA-8 (1 mol% relative to NIPAAm), 7.42 mg PBPO (1 mol% relative to NIPAAm) and 6.05  $\mu$ L AA (5 mol% relative to NIPAAm) dissolved in 500  $\mu$ L of the polymerisation solvent (2:1 v/v, THF:DI water).

The microfluidic chips were designed using AutoCAD 2014 and were fabricated using clear 2 mm PMMA by rastering with a CO<sub>2</sub> laser ablation system (Epilog Zing Laser Series) at dimensions of 1 mm wide and 250  $\mu$ m deep. The valve chamber comprised a 1 mm diameter pillar centred within a circle of diameter 2.6 mm which extended from the wall of the channel, as seen in Figure 1. The channels were sealed with a 1 mm PMMA capping layer using previously described solvent bonding processes.[5] Polymerisation and actuation was achieved using blue surface-mounted LEDs (Kingbright HB Blue 450nm, 600 mW/4.5 lm/1.3 cd, 3.5V) at 450 nm wavelength. The valves were polymerised around cylindrical pillars that provided a physical anchor to prevent movement of the valve. The elliptical valve profile was adopted as more symmetrical circular/cylindrical profiles were found to expand into the channels and become lodged in position, permanently blocking the liquid flow. Once the unreacted monomer solution had been removed, the channels were filled with deionised water and left overnight to ensure full hydration of the hydrogel valves. As outlined previously,[5] a constant head of pressure was maintained throughout all the experiments using two

reservoirs of different volumes.

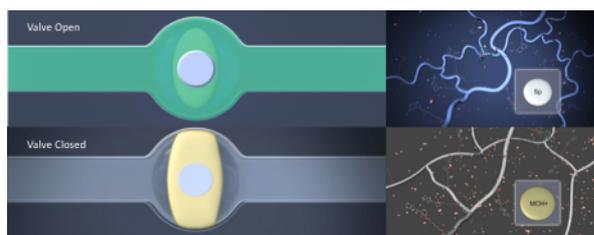


Figure 1: Hydrogel valves within microfluidic fluidic channels under light irradiation (open) and in the fully swollen (closed) states.

By introducing a monomeric cocktail mixture into a 200  $\mu\text{m}$  x 1 mm microfluidic channel, hydrogel valves were photo-polymerised in-situ using 450 nm LEDs from the actuation platform. Once any unreacted monomer was removed, the valves were left to fully hydrate overnight and swell to fill the width of the channel (Figure 1) thereby blocking liquid flow from the attached reservoir. This polymerization technique yielded valves with excellent reproducibility which were fully embedded within the microfluidic chip (Figure 2). The homogeneity of valve production is particularly evident in Figure 2 (b) which shows sequential actuation of four separate valves within a single microfluidic chip. Fast actuation times, with excellent stability (flow rate = 10  $\mu\text{L}/\text{min}$ ) and highly reproducible behaviour is evident for all four valves. This highlights the capacity of this methodology to incorporate valves within microfluidic manifolds in a controlled and reproducible manner. Even more fascinating, is the highly precise flow control that can be achieved using these photo-actuators.

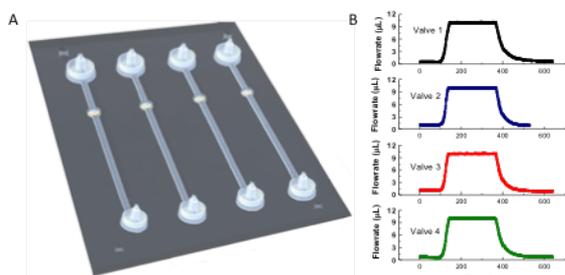


Figure 2: (A) Representation of photo-actuated valves within a microfluidic chip; (B) Actuation profiles (10  $\mu\text{L}/\text{min}$ ) for 4 valves on a single microfluidic chip.

## RESULTS AND DISCUSSION

Precise regulation of the relationship between flowrate and LED power has been achieved by using a flow meter to provide feedback to a PID controller. Through optimization of proportional ( $K_P$ ) and integral ( $K_I$ ) variables it has been possible to create a system which accurately detects disparity between the measured and set flowrates and which compensates by varying the light irradiation. An in-house developed user interface (Figure 3) enables the user to input control parameters, such as desired flowrate and maximum LED irradiation. The system then operates autonomously to deliver these flow profiles. An example of such a sequence can be seen in Figure 4. Over a period of 2 hours, the system actuates between three flowrates, with excellent accuracy. Moreover, the stability of the set flow rate over this extended period is particularly remarkable, as outlined in Table 1.

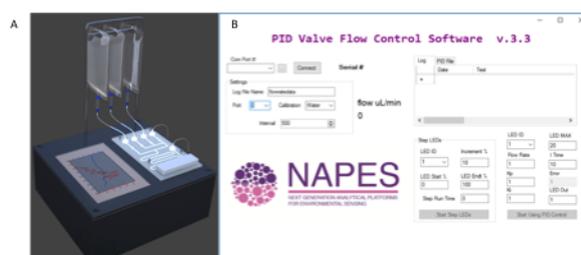


Figure 3. (A) Actuation platform with irradiation LEDs beneath the valve chambers in a microfluidic chip; (B) Graphic User Interface for valve-actuation platform.

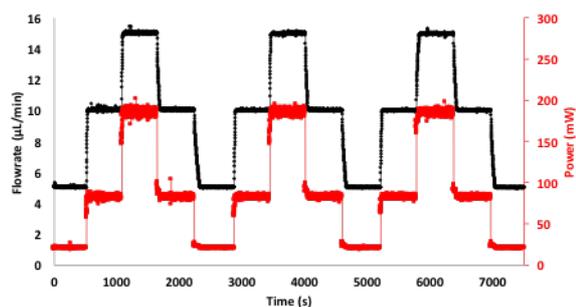


Figure 4. Cycles of (5.0, 10.0, 15.0, 10.0, 5.0  $\mu\text{L}/\text{min}$ ) shown in black ( $K_P = 10.0$ ;  $K_I = 0.1$ ), overlaid with power supplied to the LED to achieve actuation in red.

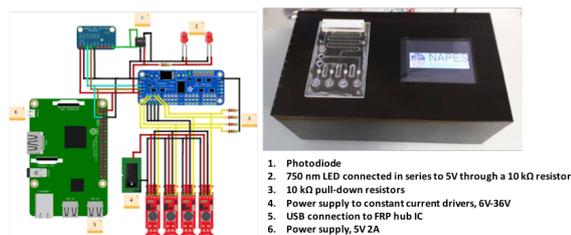
Our results also suggest there is a high correlation between flow rate and LED power. These values, shown in red in Figure 4, point to the possibility of predicting the flowrate of such a characterized system through the LED power, without the need for an external flow-sensor.

Additionally, simultaneous tracking of flowrate and LED power can serve as a diagnostic of valve performance, thereby opening the possibility for developing complex fluidics incorporating multiple valves.

*Table 1. Summary of statistics for data presented in Figure 4. Flow rate units are  $\mu\text{L}/\text{min}$ ; Power units are  $\text{mW}$ . Averages and standard deviations are for  $n=100$  consecutive points selected randomly from each steady-state flow-rate region.*

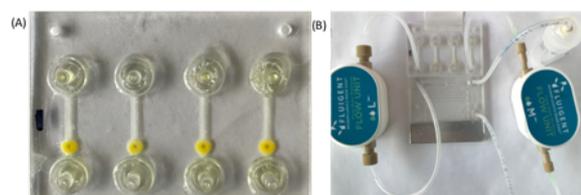
Set Flow Rate	Average Flow Rate	SD	%RSD	%RE	MOD error	Power
5.0	5.039	0.018	0.351	0.772	0.039	20.598
10.0	10.050	0.048	0.476	0.502	0.050	82.280
15.0	15.048	0.047	0.315	0.317	0.048	185.260
10.0	10.064	0.039	0.391	0.638	0.064	82.197
5.0	5.032	0.018	0.366	0.636	0.032	20.650
10.0	10.045	0.029	0.290	0.449	0.045	82.316
15.0	15.030	0.037	0.246	0.200	0.030	185.506
10.0	10.043	0.034	0.343	0.428	0.043	82.474
5.0	5.021	0.020	0.390	0.425	0.021	20.598
10.0	10.023	0.029	0.287	0.226	0.023	82.602
15.0	14.996	0.034	0.226	-0.027	0.004	184.974
10.0	10.033	0.026	0.259	0.327	0.033	82.226
5.0	5.019	0.016	0.321	0.388	0.019	20.573
	Average =	0.0292	0.3068		0.0316	

To develop on these findings and demonstrate the applicability of our system, a detection platform, based on a Paired Emitter-Detector Diode (PEDD) system was fabricated with a photodiode and two 750 nm LEDs placed at  $90^\circ$ . The LEDs and photodiode were incorporated into a 3D printed holder which was specifically designed to minimise stray light and ensure the reproducibility of measurements. As before, the LED control platform operated via a 12-bit 16 channel PWM controller (PCA9685) connected to an Arduino Uno microcontroller using the i2c protocol. An output value of 4096, equivalent to an output current of 150 mA, set the LED at 100% intensity. Figure 5 shows a schematic of the four constant current drivers which controlled the LEDs used to actuate four polymer actuators, where each driver was connected to a different output of the PWM controller, thereby allowing individual control of each photo-actuated valve. A UART (serial) connection between the Arduino and the laptop enabled the control software to send the PWM value used to maintain or vary the flow rate.

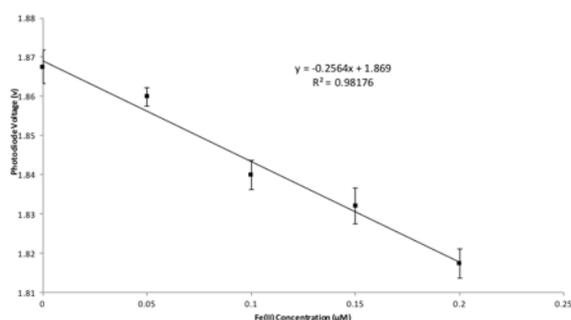


*Figure 5: Detection and actuation platform showing the wiring diagram used and the final design including a touchscreen interface.*

Full integration of both valve and detection modules then allowed for 1) control of flowrate of reagent, 2) mixing of reagent and sample and 3) detection of absorbance at 750 nm. The final prototype, shown in Figure 6 shows the modular nature of the design which allows for off-chip flowrate determination and complete mixing of reagent and sample. Four photo-actuated valves, shown in yellow, allow for selection between calibration samples of high, low and medium concentrations. The sample continues through the flow-cell where it is then analysed in real time using the PEDD system. These values are then relayed, through the Arduino microprocessor, to a computer where they are saved and processed, as seen in Figure 7.



*Figure 6: Modular nature of the analytical-detector chip; (A) photo-actuated microvalves polymerized in situ; (B) combined device, showing off-chip flow-rate measurements for both sample and reagent flows.*



*Figure 7: Calibration curve showing on-chip determination of Fe(II) concentration.*

## CONCLUSION

We have demonstrated flow control through the incorporation of stimuli-responsive materials within microfluidic channels. We subsequently elaborated on this discovery to yield highly-precise flow control through the use of a PID controller. Using an adapted platform, it has also been possible to document potential application of this technology. These results raise new challenges for researchers interested in fabricating and characterising complex microfluidic systems with multiple integrated valves. It is possible that a sub-set of flow sensors could be used to characterise a larger population of valves by switching individual or groups of valves and observing the impact on the output flow-patterns obtained from the valves, and applying machine learning approaches to identify the optimum channel/valve/flow sensor arrangement.

## REFERENCES

- [1] I.E. Araci, S.R. Quake, Microfluidic very large scale integration (mVLSI) with integrated micromechanical valves, *Lab on a Chip*, 12(2012) 2803-6.
- [2] S. Shinji, S. Kimio, O. Katsuhide, H. Kazuaki, T. Toshiyuki, K. Toshiyuki, Photoresponsive polymer gel microvalves controlled by local light irradiation, *Sensors and Actuators A: Physical*, 140(2007) 176-84.
- [3] W. Francis, A. Dunne, C. Delaney, L. Florea, D. Diamond, Spiropyran based hydrogels actuators — Walking in the light, *Sensors and Actuators B: Chemical*, 250(2017) 608-16.
- [4] J. ter Schiphorst, S. Coleman, J.E. Stumpel, A. Ben Azouz, D. Diamond, A.P.H.J. Schenning, Molecular Design of Light-Responsive Hydrogels, For in Situ Generation of Fast and Reversible Valves for Microfluidic Applications, *Chemistry of Materials*, 27(2015) 5925-31.
- [5] S. Coleman, J. ter Schiphorst, A. Ben Azouz, S. Bakker, A.P.H.J. Schenning, D. Diamond, Tuning microfluidic flow by pulsed light oscillating spiropyran-based polymer hydrogel valves, *Sensors and Actuators B: Chemical*, 245(2017) 81-6.
- [6] C. Delaney, P. McCluskey, S. Coleman, J. Whyte, N. Kent, D. Diamond, Precision control of flow rate in microfluidic channels using photoresponsive soft polymer actuators, *Lab on a Chip*, 17(2017) 2013-21.

## CONTACT

Colm Delaney, colm.delaney@dcu.ie