# DELAY VALVING IN CAPILLARY DRIVEN DEVICES BASED ON DISSOLVABLE THIN FILMS

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## ABSTRACT:

This work presents passive time delay valves for micro channels in two different working modes for microfluidic on chip timing. The delay valve designs are compatible with conventional lamination techniques for microfluidics and allow to pre-program advanced sequential operations independent from the geometries of the microfluidic system. The time delay of the dissolvable valves ranges from 1.2 s up to 36 s per valve resulting in a time range from 1.2 seconds up to 11 minutes for 19 serial valves.

## INTRODUCTION:

One of the challenges for self-contained point of care assays is the sequence (or chip timing) of different steps such as sample manipulation, incubation, reagent application and washing steps [1]. Delay valving to achieve on chip timing has been shown for paper microfluidics where sugar impregnated paper areas were used as delay channels to control fluid timing [2] or where dissolvable bridges created a timed flow shutoff [3,4]. Valving with dissolvable films has been demonstrated in centrifugal microfluidics, where the dissolving mechanism is triggered by the rotational frequency [5].

In this work we demonstrate passive dissolvable thin film valving in two different working modes (WM) which are compatible with conventional microfluidics. The valving principle includes a timing function where no external actuation is needed to open the valve and where the timing is programmed by the properties of the film (WM1) or the number of valves (WM2). Both working modes are used to demonstrate on-chip flow timing using the dissolving properties of water soluble Polyvinylalcohol (PVA) films.

## VALVE DESIGN:

**In WM1** the valve opening time is determined by the thickness of a single dissolvable film (see fig. 1). The dissolving of such a single film is a diffusion based process which makes it a nonlinear event. In **WM2** the delay is controlled by a serial multiple valve configuration using a single dissolvable film where each valve represents a discrete time delay according to the film properties. In WM2 the concentration of the dissolvable film material (PVA) in the liquid front increases with the number of valves that have been dissolved. This causes the dissolving time to gradually increase which, in the same way as for WM1, results in a nonlinear behavior. To prevent this effect, dead-end channels are added to the design. These dead-end channels will fill each time a valve is dissolved and remove the very front most liquid segment carrying the highest concentration of dissolved PVA. The dead-end channel design will make the timing behavior of the valves more linear compared to channels without the dead end, but to the expense of an increased dead volume of the system.

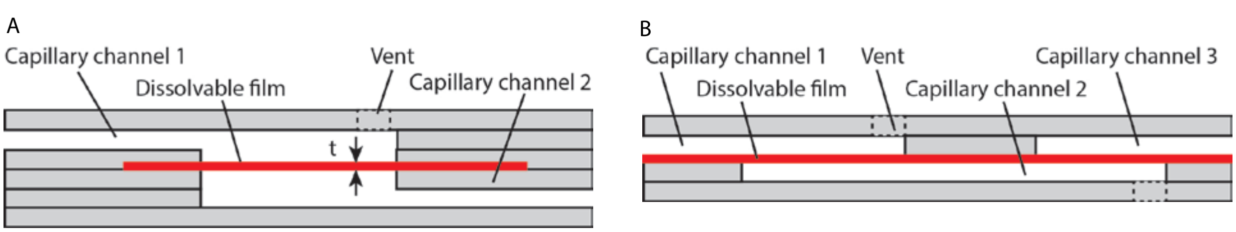


*Figure 1: A) Cross-section of a WM 1-valve; a single valve with defined thickness, t, delays the flow for a discrete time T. B) Cross-section of two WM 2-valves in a serial configuration. The valves delay the liquid flow for a discrete time $T_n$, each time the liquid passes the dissolvable film.*



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**EXPERIMENTAL:**

All devices are fabricated in a lamination based process using hydrophilic Xerox OH-films for channel covers and TESA spacer tape for capillary channel structures. The different layers are structured with a cutting plotter and then laminated together with a hot-role laminator. Dissolvable thin film valves are fabricated by spin coating water-based PVA solutions with different concentrations on silicon wafers. The films are then transferred to the microfluidic layers. For WM1 different dissolvable film thicknesses are applied to each valve manually. For WM2, a single layer with constant thickness is transferred from a wafer to the capillary spacer tape covering all valves at the same time.

To test the WM1 chips, the channel vents are temporarily closed by laminating Parafilm® to the chip surface. This is done to allow pre-loading of the initial volumes and simultaneous activation of all channels by opening the vents at the same time. When the Parafilm is peeled-off, the vents are opened and capillary channel filling occurs immediately and die-colored liquid reach valves *V1-V4* simultaneously.

WM2 valves are tested by simply applying a sufficiently large droplet volume to the inlet area. Capillary channel filling occurs immediately and initiates the first of the serial valves ($V_1$). After the delay time $T_1$, the valve $V_1$ is opened and the channel can further fill and initiates the delay valve $V_2$. Similarly, the remaining valves are dissolved and opened sequentially.

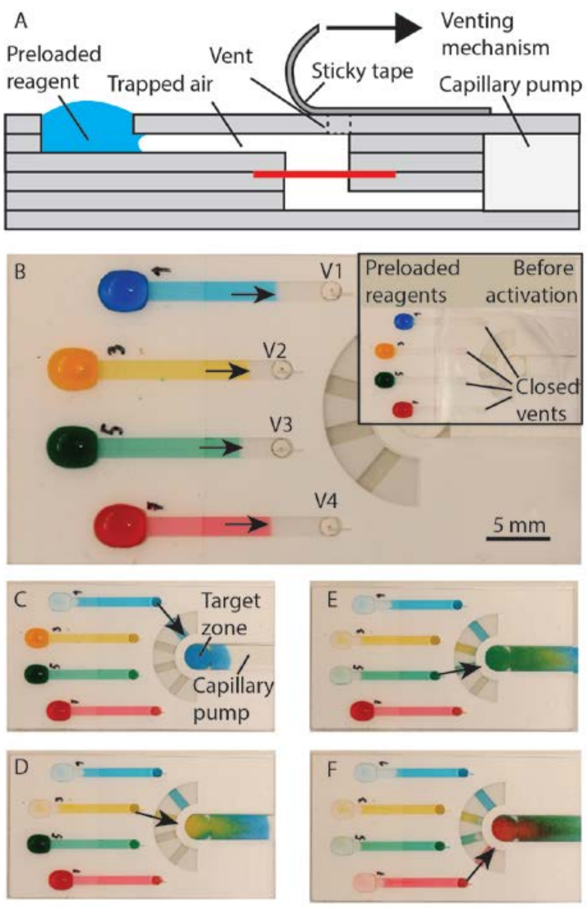


*Figure 2: A) WM1 valve cross-section showing the venting mechanism short before activation by peeling off the sticky Parafilm® B) Top view of the chip short after activation; simultaneous filling of channel 1-4. C) Release of V1 results in wetting of the target zone while V2-V4 are still closed. D)–F) subsequent release of the liquid according to the film thickness at V2-V4.*

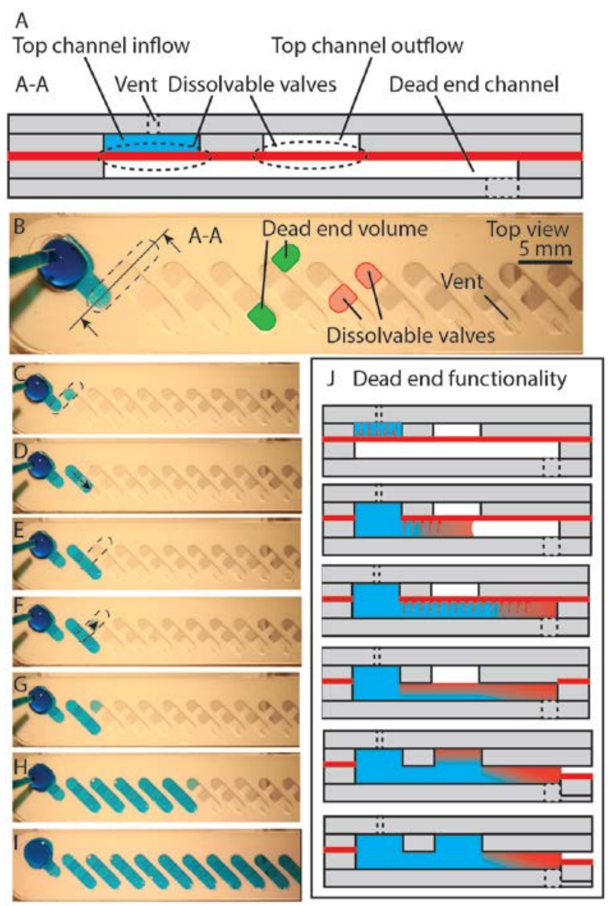


*Figure 3: A) Cross-section of two WM2 valves also showing the dead end channel. B)-I) Filling sequence of 19 valves. The channels in the lower level are not visible (dashed line). J) Scheme showing the functionality of the dead end channels. The red gradient demonstrates the increased concentration of dissolved PVA in the liquid.*

**RESULTS AND DISCUSSION:**

WM1: When activating four pre-loaded channels (fig. 2) by simultaneous opening of the channel vents (fig. 2A), capillary channel filling occurs (fig. 2B) and the liquid front reaches valve V1-V4 at the same time. V1-V4 consist of 1, 3, 5 and 7 layers of a 3.75 µm thick PVA film which results in dissolving times of 2, 7, 64 and 125 seconds, respectively, showing that the dissolving time of the valves increases non-linearly with the film thickness. However, a sequential reagent release could be achieved (fig. 2C-F). The release times for the valves range from approximately 1 s to 125 s and are plotted in figure 4A.

WM2: In the serial configuration (fig. 3) one channel wall always consists of PVA which causes PVA saturation at the liquid front and increased valve dissolving times. To solve this problem, the liquid front is exchanged at each valve, by adding dead-end channels to the design (fig. 2A, J). Although dissolving times are not increasing linearly with film thickness (fig. 4A), the serial arrangement (WM2) allows timing delays that increase approximately linearly with the number of passed valves (fig. 4B). This is also evident when comparing WM1-V4 with a total thickness of 26.3 µm to the equivalent thickness of four 7.2 µm-valves (corresponding to 28.8 µm) in WM2. The dissolving delay of 125 seconds for the WM1-valve shows a much longer dissolving delay compared to the 19.2 seconds for four consecutive WM2-valves. A total delay time control range from 0.6 seconds (1 valve, 2.7 µm PVA film thickness) up to 11 minutes (19 valves, 23.5 µm PVA film thickness) was demonstrated (fig 3B).

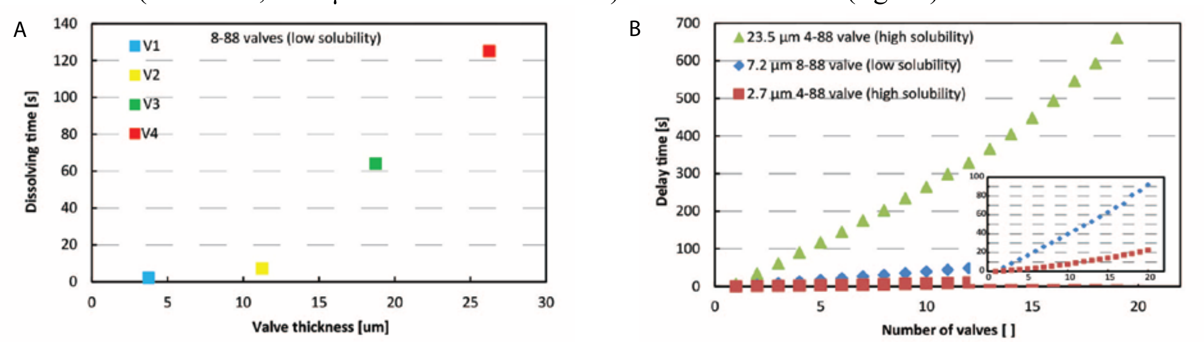


*Figure 4: Measurement results extracted from video recordings. The dissolvable films consist of Mowiol® 8-88($M_W$ ~67,000) or Mowiol® 4-88 ($M_W$ ~31,000) with different thickness. A) Four WM1-valves with different thickness and the corresponding dissolving times. B) 19 serial WM2-valves fabricated with three different film thicknesses covering a range of 0.6 seconds up to 11 minutes of total time delay.*

**CONCLUSION:**

This work presents passive delay valves for on chip timing of capillary driven microfluidics. The delay valves can be used in two different working modes depending on the requirements to the system. Both working modes can be used for timed reagent release in more advanced lateral flow assays such as ELISA and allow to pre-program the timing or the sequence of different operations such as reagent application, incubation, and subsequent washing steps. A total delay time control range from 0.6 seconds (1 valve, 2.7 µm) up to 11 minutes (19 serial valves, 23.5 µm) was demonstrated.

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