Talanta 134 (2015) 443–447

Contents lists available at ScienceDirect

Talanta

journal homepage: www.elsevier.com/locate/talanta

Short communication

Rapid prototyping of pneumatically actuated hydrocarbon gel valves for centrifugal microfluidic devices

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ARTICLE INFO

Article history:
Received 3 September 2014
Received in revised form
13 November 2014
Accepted 14 November 2014
Available online 24 November 2014

Keywords:
Microfluidics
Centrifugal
Active valve
Flow control
Liquid sample processing
Hydrocarbon gel

ABSTRACT

A novel, easy to prototype hydrocarbon gel-based active valve was developed for use in centrifugal microfluidic devices. The valve has been demonstrated to restrict flow by an additional 1000 revolutions per minute (RPM) when compared to a passive capillary valve of the same size located at the same radius. Opening of the valve is accomplished in a contactless manner using a stream of focused compressed air. The ease of fabrication, low cost and small dimensions of the gel valve offer the potential for integration of multiple valves of this type into multi-process centrifugal microfluidic systems.

1. Introduction

Microfluidics allows the miniaturization of multiple analytical processes into a single micro total analysis system (μTAS). The small dimensions and potential portability of these systems render them attractive for in-situ chemical analysis and point of care diagnostics [1].

Centrifugal microfluidic (CM) devices incorporate the portability, efficiency and other benefits of traditional microfluidic systems with the consistent and tuneable flow control associated with centrifugal force [2–5]. In these devices, which often take the form of compact discs (CD), centrifugal force induced by rotation of the device initiates liquid flow, eliminating the need for external pumps and their connections. The radially uniform nature of centrifugal force allows simultaneous analysis on parallel units rendering CM conducive to high throughput analysis [2,4].

The creation of fully integrated systems incorporating multiple processes often requires precise flow control to ensure completion of each sequential unit operation. On CM devices, this requirement can be most often met by passive and active valves.

Passive valves, such as capillary, hydrophobic, Coriolis and siphon-based valves exercise flow control using processes correlated to the device's rotational frequency [2,3,5–8]. Capillary valves gate liquid flow by pinning the meniscus of the advancing liquid at the interface between the valve and a channel or chamber of much larger cross section until sufficient pressure is generated through centrifugal force for the liquid to flow (or “burst”). The wetting properties of the liquid, and the geometry of the channel, such as its cross section and head height, determine the frequency required to induce flow, known as the burst frequency [2,3]

The implementation of multiple sequential operations using capillary burst valves requires each valve to be more restrictive than the previous valve. Generally speaking, the smaller the valve, the higher the burst frequency. However, as the radial position (from the center) of a given size valve is increased, a lower burst frequency is observed as the centrifugal force increases with the radial position. The combination of these factors limits the number of sequential operations possible using passive capillary valves. Despite their prevalence, passive capillary valves can present irreproducible burst frequencies, and their integration into a rapid prototyping process is often time-consuming and labor-intensive [6]. Hydrophobic valves suffer from similar drawbacks as they rely on a related principle [2]. Coriolis valves provide an efficient method of flow switching; however they require high angular velocities to successfully switch the flow, limiting their compatibility with sequential operations. Siphon-based valves have been developed to control liquid flow through an integrated siphon. These valves often require surface treatment of the siphon channel, which complicates their fabrication and often limits their lifetime. In addition, the device must be stopped to prime the siphon, leading to the possibility of unintended liquid flow in other parts of the device due to capillary action in the absence of centrifugal force.

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http://dx.doi.org/10.1016/j.talanta.2014.11.029
0039-9140/

Active valves rely on a frequency independent process such as physically blocking a channel to restrict flow, allowing for more precise flow control and greater flexibility in design [7,8]. Currently, there are fewer and less diverse options for active valves when compared to their passive counterparts. Phase change-based valves represent the majority of presently available active valves. Active valves incorporating hydrogel, ice, wax and magnetic nanoparticle embedded wax have been demonstrated for use in microfluidic devices [9–11]. Amasia et al. [11] developed an ice plug for CM devices for use with PCR. However, it was necessary to stop the disc to open the valve, which is undesirable in many applications. Integration of multiple valves of this type requires a thermoelectric module for each valve position and the modules must be properly distanced to avoid the opening of neighboring valves, thereby limiting the number of valves that can be incorporated into a single device. In contrast, the paraffin wax-based method of Abi-Samra et al. [8] irradiates the entire disc and offers selective valve opening based on the difference in melting points of wax plugs with varying composition. However, this irradiation may be unsuitable for use with biological samples. In addition, the different wax compositions required for each valve complicate large-scale valve integration. The Laser Irradiated Ferrowax Microvalves (LIFM) developed by Park et al. [7] utilize iron-oxide nanoparticles incorporated in a ferrowax hydrocarbon mixture that is melted by a low intensity laser. This method allows sequential valve opening by focusing the laser beam on the radius pertaining to the wax plug to be opened. While LIFMs offer a fast and selective sequential valve solution, the required materials are expensive and need complex preparation and equipment, such as a moveable laser.

To facilitate the integration of multiple sequential operations on CM devices, an inexpensive, non-radiative, active valve with a simple fabrication process is desirable. We have previously developed a non-contact pneumatic pumping technique that can be utilized to enhance and expand the capabilities of CM devices [12,13]. In this paper, we extend the pneumatic method with a new active valve technique that uses an inexpensive, externally controlled hydrocarbon gel valve opened by a jet of compressed air.

2. Material and methods

2.1. Device design and fabrication

Fig. 1 presents the design of the CM device used to demonstrate the valve's effectiveness. The hydrocarbon gel utilized in the device is a commercially available petroleum jelly. The pneumatic micro-valve comprises a hydrocarbon gel plug (Fig. 1e and g) located inside an 800 μm deep channel (Fig. 1d) and positioned to block flow out of a 100 μm deep channel (Fig. 1c) connected to an 800 μm deep liquid reservoir (Fig. 1e). The plug is slightly thicker than the channel from which liquid flows while only partially covering the channel in which it resides. This allows easy displacement of the plug through air pressure applied at an inlet directly above the valve (Fig. 1d), forcing the plug to disperse in the radially outward direction. An air vent connected to the reservoir facilitates injection (Fig. 1a).

The device was constructed with the rapid prototyping method described by Kido et al. [14]. The five layers of the device (Fig. 2) were designed using the SolidWorks computer-aided-design software (SolidWorks Corp., Concord, MA, USA). Each disc layer (Fig. 2a, c and e) consisted of a 600 μm polycarbonate DVD machined using a four-axis CNC mill (MDX-40A, Roland Corp., Los Angeles, CA, USA). The disc layers were bonded together using 100 μm double-sided adhesive layers (Fig. 2b and d) (FLEXmount DFM-200-Clear V-95 150 poly V-95 400, FLEXCon, Spencer, MA, USA) with the corresponding channels and chambers cut by xurography using a cutting plotter (CE3000Mk2-60, Graphtec America Inc., Santa Ana, CA, USA).

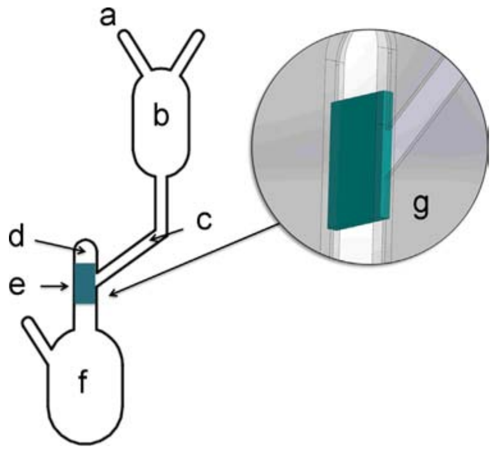

Fig. 1. Demonstration unit: Design of a single unit of the CM device. Each device contains six units with (a) injection and vent ports, (b) liquid reservoir, (c) 100 μm deep channel cut in the bottom adhesive layer, (d) 800 μm deep channel with air inlet at top, (e) petroleum jelly plug, (f) receiving chamber, and (g) expanded view of junction of plug highlighting the critical difference in channel depth and plug height.

A partially assembled disc was constructed from the base disc and bottom adhesive layer (Fig. 2a and b). Keeping the protective peel on the adhesive, a dot of commercial petroleum jelly was placed in the 800 μm vertical channel blocking the 100 μm diagonal channel (Fig. 1c). A thin piece of flexible plastic was used to level the surface of the plug with the top protective layer on the adhesive. After peeling off the protective layer, the ~4 mm long plug projected slightly above the plane of the 100 μm channel. The remaining layers were then assembled with each layer being sealed to the previous with a hand roller. The final device was not cold-laminated to avoid the irreproducible displacement of the gel plug due to the added pressure.

Although the disc is designed with multiple air inlets on the same radius for rapid and consecutive opening of parallel valves, all inlets except the one being tested were covered using tape to allow the testing of one valve at a time without compromising the integrity of the surrounding valves.

2.2. Experimental setup

A centrifugal experimental setup previously described by Duford et al. [15] was utilized to evaluate the pneumatic valve device. This setup was augmented with a color digital camera (GRAS-14S5C-C, Point Grey, Richmond, BC, Canada) positioned at 90° to the disc surface to allow acquisition of in-motion images. The pneumatic system utilized for valve actuation was derived from the setup described by Kong and Salin [16] with modification to the air outlet. A microcapillary pipette tip (200 μL, Denville Canada, Toronto, ON, Canada) was attached to the tubing to focus the air stream on the disc. The nozzle was positioned 5 mm above the disc to maintain a non-contact configuration. The radial position of the air nozzle was controlled via a linear actuator and set to 39 mm from the disc center so that the nozzle aligned with the air inlet (Fig. 1d). The components of the experimental setup and pneumatic system were synchronized via software (LabVIEW, 8.6, National Instruments, Vaudreuil Dorion, QC, Canada). Experimental settings such as the radial position of the air nozzle, air triggering, rotational frequency and data acquisition parameters were pre-set by the user into the LabView program.

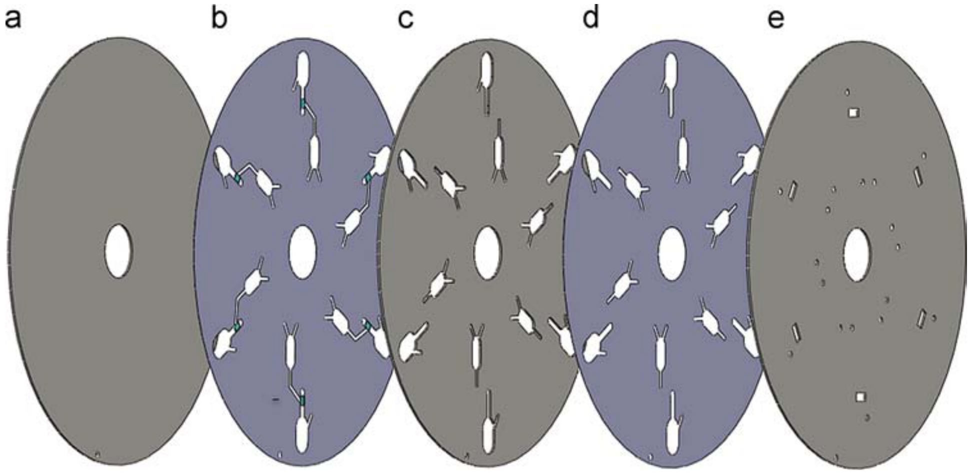

Fig. 2. Exploded view of CM device layers: (a) 600 μm DVD base layer, (b) 100 μm adhesive layer, (c) 600 μm DVD middle layer containing reservoirs and the main channel into which the hydrocarbon gel plug is incorporated, (d) 100 μm adhesive layer containing channel leading from reservoir to plug, and (e) 600 μm DVD top layer containing air inlets and injection holes.

2.3. Experimental procedure

To determine the frequency at which the naturally restricting 100 μm channel allows flow, herein referred to as the 'unsealed burst frequency', a hydrocarbon gel plug was displaced (opened) on a demonstration unit using compressed air. A 30 μL aliquot of distilled deionized water (DDW) containing commercial red food coloring was injected into the liquid reservoir (Fig. 1b). The device was then spun at a series of increasing rotational frequencies from 200 RPM to 1500 RPM in increments of 100 RPM every 10 s to determine the frequency at which liquid started flowing through the 100 μm channel to the receiving chamber. The same procedure was applied for a demonstration unit with an intact valve to determine the maximum frequency up to which the plug restricts flow (working range).

To test the opening of the valve, a 30 μL aliquot of colored DDW was injected into the liquid reservoir of a demonstration unit sealed with a petroleum jelly valve (Fig. 3). The disc was initially spun for 10 s at 900 RPM, a frequency significantly higher than the unsealed burst frequency, to verify flow restriction. The duration and flow rate of the pulse of air were established experimentally. The images collected while in motion were used to confirm opening of the valve and the subsequent transfer of the liquid into the receiving chamber.

3. Results and discussion

The burst frequency of the valve with and without the plug was first established. Without the plug in place, liquid was found to flow through the open 100 μm channel at a rotational frequency of 300 RPM ($n=3$). With the plug in place, the maximum leak-free rotational frequency achieved was 1300 RPM ($n=3$) for 10 s of rotation. Above this frequency, the liquid travels around the junction between the 100 μm and the 800 μm channel (Fig. 4). This is most likely due to a failure in the adhesive layer at the angular junction. The failure may be due to the adhesive layer not being completely sealed since the final device was not cold-laminated to avoid the dispersion of the plug. Furthermore, imperfect alignment of the adhesive layer during the fabrication process can create 'microchannels' around the milled feature, which could lead to leaks around the gel plug if the microchannel extends beyond the length of the gel plug. Images obtained during the leaking process show no signs of liquid flowing through the plug. Instead, it appears that the liquid flowed around the valve through the adhesive layer by either of the two mechanisms suggested previously, demonstrating that the reliability of the valve operation depends strongly on the reliability of the construction method utilized. A more robust bonding method or gel injection post disc fabrication via the air inlet (Fig. 1d) would avoid this issue and allow for the full realization of the valve's blocking potential. It is important to note that the observed maximum achievable rotational frequency (1300 RPM) is dependent on the radial position of the valve, with valves located closer to the disc center being more likely to achieve even higher blocking frequencies without leaking.

The active valve opening sequence demonstrated the practical potential of the seal. The theoretical sequence for this is shown in Fig. 3a–d. At 900 RPM, the plug completely restricts liquid flow (Fig. 3e). Upon activating the air stream, the seal is quickly broken during the 1-s pulse of air (Fig. 3f). The optimal flow rate was determined to be 0.25 standard cubic feet per minute (SCFM), as this blast of air did not force the remnants of the plug down into the receiving chamber. The duration of the pulse of air was set to the shortest time interval allowable in the software, 1 s. The plug is dispersed and remnants are deposited in the channel leading to the receiving chamber. Liquid immediately starts to flow through the 100 μm channel around the plug remnants towards the receiving chamber and the entirety of the liquid is transferred within 10 s (Fig. 3g and h). The liquid is only in contact briefly with the plug material as it is forced radially outwards.

In order for valve opening to occur, the applied pressure of the air stream must overcome the adhesion forces between the gel and the device. This minimum pressure is dependent on the applied flow rate and can easily be determined on a prototype using a simple flow meter. The impact of the device rotational frequency on valve opening is difficult to determine without high speed imaging of the plug displacement to verify whether the gel was displaced during the first exposure to the air stream or through any of the consecutive pulses (15 pulses over a 1-s burst of air at 900 RPM). Imaging the valve with a high speed camera could assist in investigating the displacement mechanism and the effect of rotational frequency.

The valve geometry was chosen to be rectangular to simplify the opening process and minimize the dispersion of the gel after opening. As this configuration presented the desired results, alternative geometries were not studied.

The utilization of active gel valves allows a substantial enhancement when compared to the use of standard passive capillary valves, due to the frequency independent nature of the flow

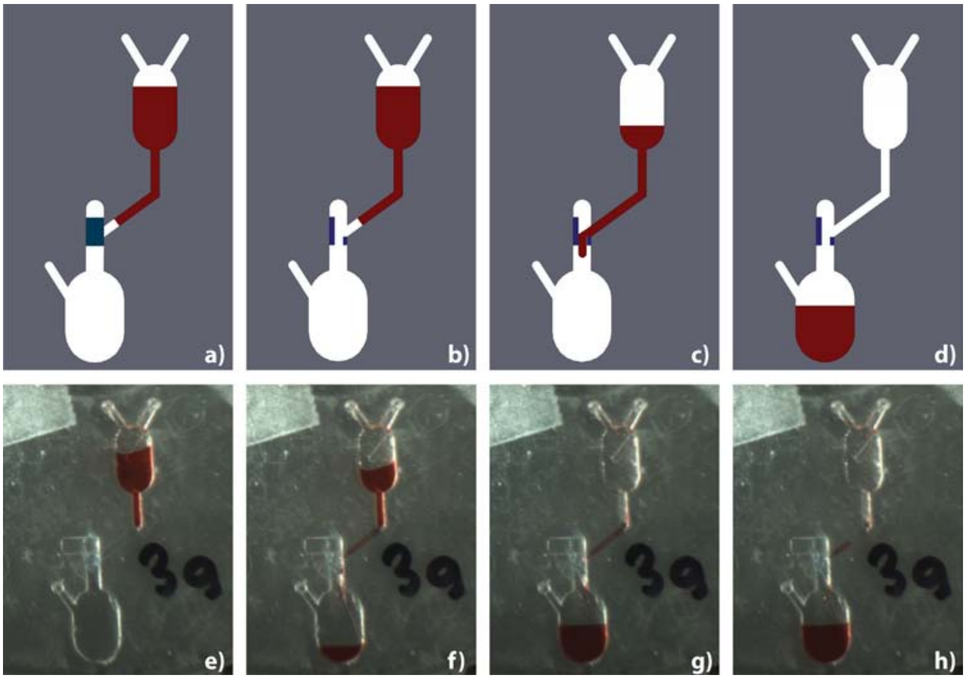

Fig. 3. (a–d) Operational principles of the pneumatically actuated hydrocarbon gel valve. (a) Intact plug restricting flow while spinning, (b) plug dispersed by air stream, (c) liquid flows from reservoir through the channels to the receiving chamber, and (d) reservoir emptied into receiving chamber. (e–h) Experimental Images: (e) $t=14$ s spinning at 900 RPM with an intact plug restricting flow, (f) $t=16$ s seal opened by air stream while spinning at 900 RPM, liquid immediately starts to flow, (g) $t=18$ s continuing spinning at 900 RPM, liquid flowing out of reservoir to receiving chamber, and (h) $t=20$ s reservoir emptied.

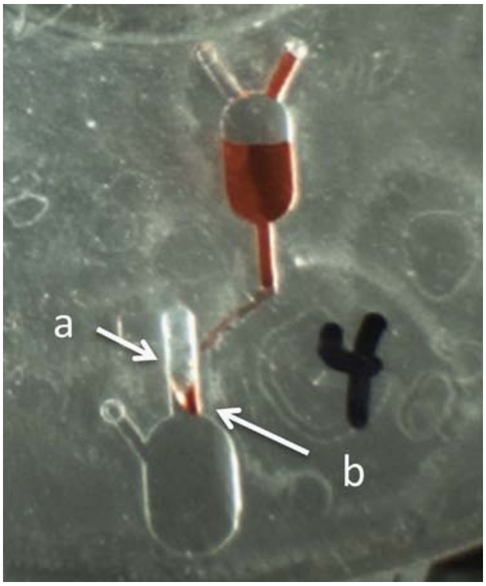

Fig. 4. Failure in adhesive layer showing: (a) an intact plug blocking 100 μm channel, and (b) liquid from 100 μm channel which has bypassed the plug.

control. This type of flow control enables the design of more complex devices without the constraints of traditional valves. Furthermore, it facilitates the incorporation of many rotational-frequency dependent unit operations such as shake-mode mixing or sedimentation without initiating flow.

Additionally, the inclusion of gel valves between passive capillary valves negates the requirement for successively higher burst frequencies. This greatly simplifies production as the sequential valves no longer need to be highly restrictive, allowing the utilization of simpler passive valves such as those cut out of the pressure sensitive adhesive. These adhesive valves typically require much less than 1000 RPM to burst, ensuring that there is no accidental triggering of the interspersed gel valves.

The simplicity of the valve's opening mechanism grants this type of valve a significant advantage for the implementation of multiple sequential valves on a single device. The compressed air nozzle is positioned by a linear actuator, allowing for precise control of the radial position of the air stream. The application of pneumatic pumping at different radial positions using a linear actuator was previously demonstrated by Kong et al. [16]. The actuator's rapid response and movement enable the displacement of the air stream during platform rotation to as many radial positions (valves) as required by the user. Combined with the precise alignment (~1 mm) between air nozzle and air inlet necessary for valve opening, this feature should enable the integration of a large number of sequential valves on a single disc as valves can be placed radially close together without the risk of breaching a neighboring valve. The sequential activation of a large number of valves can be easily programmed, as it only requires knowledge of the radial position of each valve.

The experiments performed in this work involved a single valve opening operation as the inlets surrounding the valve of interest were covered during testing. However, the disc design is compatible with consecutive rapid valve opening, as valves for each unit on equivalent radii can be opened during a 1 s application of air. While only six units were included in the demonstration device in order to obtain clear pictures of the valve opening process, many more can be fitted on a single device as the only required component is a channel fitted with an air inlet, a feature that is often already present on CM devices and that occupies little device real estate. As such, the presented valve technique should not only enable the sequential actuation of several valves on a single unit but also the parallel actuation of several units at the same time, taking advantage of the exceptional potential for parallelism of CM.

Furthermore, a significant advantage to the physical seal of the gel valve is the potential for liquid storage in the device. Traditionally, liquid samples cannot be stored in devices due to the evaporation that takes place through vents, siphons and passive valves that are not closed systems.

The effect of the plug's brief contact with the liquid was not tested in this work. However, as aqueous samples were used and the plug was made of a hydrophobic hydrocarbon material it can be presumed that the interaction between the two components is minimal. In addition, the sample/plug contact time is minimal during liquid transfer, reducing the risk of contamination. Therefore, the usage of inexpensive and widely accessible petroleum jelly follows the same premise as other hydrocarbon-based active valves. Platforms including reagents that may be sensitive to the hydrocarbon material can be designed with a longer valve-containing channel to ensure that none of the valve material reaches the receiving chamber. Given the small dispersal pattern observed during experiments, such a safeguard should be easy to implement.

The main focus of this gel valve technique is to provide a low-cost alternative to more expensive and complicated active valves. While the presented valves are of single-use, their low cost and ease of implementation render them congruent with the desired design paradigm of inexpensive, disposable analytical devices made for a single analysis.

4. Conclusions

We have successfully demonstrated an inexpensive, easy to fabricate, active hydrocarbon gel-based valve on a CM device. The plug is made from widely available materials and does not involve complex preparation or time-consuming installation methods. The flexible nature of the valve mechanism could allow it to be implemented in manufactured end products. The focused stream of compressed air used to open the valve and the small dimensions of the plug render this method conducive to serial valve opening dependent on radial position of the air inlets. The valve successfully restricts liquid flow for an additional 1000 RPM in comparison to an unsealed unit of the same size and location, permitting the inclusion of multiple frequency dependant unit operations. Additionally, the valve can be used in conjunction with passive capillary valves to eliminate the need for consecutively smaller valves and higher burst frequencies. The physical seal created by the gel valve can also be exploited to allow storage of liquids in the device.

Acknowledgments

The authors gratefully acknowledge support from the Discovery Grant program (Grant no. RGPIN 1126) of the Natural Sciences and Engineering Research Council of Canada.

Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.talanta.2014.11.029.

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