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A compact, low-cost microliter-range liquid dispenser based on expandable microspheres

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Abstract

This work presents a new low-cost liquid dispenser for the dispensing of microliters to milliliter volumes. The dispensing mechanism is based on a thermal actuator where highly expandable microspheres expand into a liquid reservoir consequently displacing any stored liquid. All device components are made out of low-cost materials and the fabrication process has the potential for high volume batch manufacturing. The device utilizes the property of the expandable microspheres to form a heat insulating layer between the heat source and the delivered liquid. Moreover, it does not require any feed back or complicated flow metering. The device was successfully tested showing a mean dispensed volume of 101 μl with a standard deviation of 3.2% and with a maximum temperature of 59 °C in the liquid during actuation. It was shown that the dispenser is strong enough to deliver against counter pressures as high as 75 kPa. The device can also function as a low flow rate dispenser as demonstrated in a microfluidic dye laser application. The flow rate can be controlled between 1 $\mu\text{l h}^{-1}$ and 2400 $\mu\text{l h}^{-1}$ by adjusting the actuation power.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

With the advancement of microfluidics and the development towards analysis chips there is a growing need for on-chip actuation methods to propel liquid volumes. Specifically in applications where relatively large volumes (μl to ml) need to be propelled, classical approaches such as capillary driven flow or electro osmotic flow are less suited. Flow-through micropumps [1], on the other hand, may provide good accuracy and sufficiently large flow rates but are often too expensive to be integrated into disposable chips. Furthermore, in many disposable applications fluid pumping does not need to be of flow-through type; limited-use or one-shot propellants are in many cases a sufficient driver for fluids. The main incentive to use limited-use or one-shot propellants in integrated applications is to lower the cost of the microfluidic unit. In addition, some one-shot

propellants also feature characteristics such as very high pressure generation. Particularly, in applications where precise dosing is required, e.g. drug delivery, a desirable disposable pumping system may be identified from the following distinct functional requirements: low cost, precise dosing, low power consumption and robustness. A number of fundamentally different dosing solutions for limited-use or one-shot propellants which have the potential to meet these requirements have been proposed. To achieve precision in dispensed volume most systems use a geometrically defined sample reservoir which is released by activating a fluidic actuator. In many solutions liquid volume dispensing is based on phase-change phenomena. Such include chemical decomposition of AIBN (azobis-isobutyronitrile) caused by heating [2], and gas generation through electrolysis [3, 4]. Since a compressible gas is generated, these techniques are suitable for propelling liquid samples where no large counter

Table 1. Overview of actuation types for liquid propagation.

Actuator	Actuation	Physical principle	Actuator material state	Actuation energy required	Volume expansion	Temperature (°C)	Reversibility	Reference
Paraffin	Thermal	Phase-change	Solid	Moderate	40%	40–100	Yes	[5, 6]
Expancel	Thermal	Gas expansion	Microbeads	Large	>300%	70	No	This work
Hydrogel	Thermal	Chemical	Gel	Moderate	>100%	33 ^a	Yes	[12, 13]
AIBN	Thermal	Chemical	Powder	Moderate	100%	70	No	[2]
Air-burst	Thermal	Gas expansion	Gas	Moderate	100%	<40	No	[14]
Thermal expansion	Thermal	Thermo-pneumatic	Gas	Large	10–20%	40–100	Yes	[7–9]
Electrolysis	Electrical	Electrochemical	Liquid	Low	<10%	RT	Yes	[3, 4]

^a Temperature below this results in expansion.

pressure is encountered. In contrast to these, a very strong phase-change actuator for microfluidics is solid paraffin, which is melted, thereby causing an incompressible volume expansion [5, 6]. However, the volume expansion achieved by paraffin actuators is typically limited to a few tens of percent. From a fabrication point of view, pure thermopneumatic actuators [7–9] where a gas's thermal expansion is used might be an attractive alternative, but they are very limited in what pressure and thus the volume change that they can produce. Hydrogel actuators, on the other hand, show large expansions capabilities and can in many cases be used for actuating liquids. The actuation principles vary, e.g. through pH [10, 11], glucose [11] or thermally [12, 13] responsive hydrogels. Electrical actuation is preferred in order to control and easily program dispensing. Integration of heater structures is the simplest solution to achieve electrical control and thermally responsive hydrogels are therefore of most interest. However, thermally responsive hydrogels expand due to a decrease in temperature [12] and thus Peltier elements are needed for actuation. Another approach of integrating liquid propellants into a microsystem is to store energy in form pressurized gas. Hong *et al* presented on-chip air-bursting detonators which are activated by opening a thermoplastic membrane [14].

In this paper we use highly expandable Expancel[®] microspheres as a driver for liquids. The microspheres contain pressurized gas and thus the principle of stored pressurized gas is similar to the work of Hong *et al*. However, since the gas is stored inside microbeads and in a condensed state, it makes it easier for process integration and allows much larger volume expansion. An overview of some of the most important properties of the above-mentioned actuation principles are presented in table 1. Previously our group has shown the use of expandable microspheres in microfabrication, where we showed bead expansion in microchannels [15] and as a novel expandable composite by integrating expandable microspheres into a PDMS matrix [16]. In this paper, we describe work where expandable microspheres are mixed with glycerine to allow for volume expansions several times the original volume. This mixture is used to realize a compact, low-cost liquid dispenser with high precision suitable for disposable drug delivery applications.

2. Design

The liquid dispenser design presented in this paper is based on a thermal actuator consisting of Expancel[®] expandable

Figure 1. Working principle of Expancel[®] microspheres: (a) analogy with a spring system where potential energy is released due to weakening of the thermoplastic shell; (b) single beads expand up to 60 times their original volume V_0 when heated.

microspheres. These microspheres consist of a copolymer shell encapsulating a partly condensed, i.e. highly pressurized, hydrocarbon (isobutane). When the spheres are heated the shell softens and the volume increases due to the high pressure inside the sphere.

Thus, the microspheres comprise pneumatic energy that is stored in the sphere and the actuation energy required is solitary determined by the energy needed to soften the thermoplastic shell (figure 1). The expansion of the spheres is irreversible; when the heating stops the thermoplastic shell cools down and its shape remains. The size of the unexpanded microspheres is about 10 μm in diameter and a single sphere can expand up to 60 times the original volume. Figure 2 shows SEM pictures of the expandable microspheres in both the unexpanded and expanded states.

By utilizing the relatively large expanding property of the microsphere we have designed a liquid dispenser where the microspheres replace a stored liquid portion. Figure 3 shows the principle of operation. A thin layer of expanding microspheres is heated from one side. As the spheres reach the transition temperature of 70 °C they expand into a liquid reservoir consequently ejecting the stored liquid through an outlet hole. The expansion is irreversible; hence a valve structure to prevent back flow is not needed. However, to avoid contaminating the delivery liquid, a membrane is needed to separate the liquid from the microspheres. Since the expandable microspheres consist to a large extent of gas they are also an excellent heat insulator. During actuation, i.e. while the spheres expand, the heat conduction through the expanding layer decreases considerably. This important property prevents the delivered liquid from extensive heating,

Figure 2. SEM pictures of Expancel[®] microspheres before (a) and after (b) expansion. Note that the magnification is the same in both pictures.

Figure 3. Principle of operation. As the expanding microspheres reach the transition temperature of 70 °C they expand into the liquid reservoir consequently ejecting the stored liquid through the outlet hole.

which is a necessary requirement when dispensing potentially temperature sensitive liquids such as drugs.

Macroscopically the microspheres appear as powder and incorporation into a matrix material eases microfabrication. In this design we have chosen a liquid carrier to achieve a microsphere paste that has a large expansion capability and, in an expanded state, conforms well to the geometry of the liquid reservoir. The good fill factor of the paste ensures a well-defined and accurate dispensed volume. We have chosen glycerin as the microsphere carrier liquid since the thermal properties of glycerin promote the distribution of the heat to the spheres and it also has a lower heat capacity than, for example, water. In addition, the low vapor pressure of glycerine also prevents evaporation of the microsphere mixture.

The primary target applications for the presented dispenser are disposable drug delivery units and μl -volume dispensing for lab-on-chip purposes. In particular, we see the proposed device as a suitable dispenser unit for controlled drug delivery with microneedles [17]. Therefore, the device is entirely based on low-cost materials and processes.

3. Fabrication

Figure 4 shows a schematic view of a demonstrator device that was fabricated and evaluated. The structure consists of a cone shaped 100 μl liquid container made of acetal plastic (POM, Polyoxymethylene). The liquid container is separated from the actuation material by a 200 μm thin elastic vinyl membrane. This membrane is clamped into the liquid container by a 500 μm high ring also made of POM, thereby

Figure 4. Schematic view of the prototype device. The ring with the expandable paste together with the membrane is clamped into the liquid container, thereby stretching the membrane to become flat.

stretching the elastic membrane. A mixture of expandable microspheres (Expancel[®] 820DU) and glycerine is screen printed into this ring. The size of the ring is set to hold a actuator material volume of 35 μl . A standard FR-4 printed circuit board (PCB) is used to produce the necessary heat for actuating the device. A PCB with a photolithographically defined copper heater is attached on top of the ring and is therefore in immediate contact with the expandable mixture. The PCB is fixated to the container by glue. The device was tested with DI-water as the dispensed medium and devices were filled by submerging into DI water and applying vacuum.

The expandable material was prepared by mixing Expancel[®] 820DU microspheres (Akzo Nobel Surface Chemistry AB, Sundsvall, Sweden) with glycerine at a concentration of 0.7 g ml^{-1} . This mixing rate results in a rather viscous paste which is suitable for screen printing and still wet enough for good heat conductivity.

In a separate study made on microspheres mixed into DI water, it was found that different ways of pretreatment of the mixture has a significant influence on the expansion level. Figure 5 shows the expansion of 15 μl mixture samples (0.2 g ml^{-1} DI water) which were treated differently prior to expansion. In these tests the sample was placed in a brass holder which was uniformly heated to 70 °C and the volume expansion was then measured.

Figure 5. Different pretreatments show a significant variation in the level of expansion for expandable microspheres mixed in DI water. Pretreatment with ultrasound showed a considerable expansion increase.

It was found that mixtures which were outgassed for 10 min and exposed to ultrasound (Branson[®] 220) for 15 min, expand approximately 60% more than untreated mixtures. Outgassing of the mixture removes trapped air bubbles which most likely promote expansion slightly. However, the beneficial effect of ultrasound is not clearly understood. A possible explanation is that the effect is a result of the weakening of the spheres' thermoplastic shell due to minor heating created by the ultrasound.

Based on the results of the above-mentioned investigation, the same pretreatment with outgassing and ultrasound was applied to the glycerine/microspheres paste used in the dispenser.

4. Experimental details

The fabricated dispenser structure was tested with respect to precision in delivered liquid and the ability to dispense against counter pressure. The maximum temperature of the delivery liquid during actuation was also measured.

The dispensed volume was measured by connecting an assembled device to a glass capillary using interconnecting silicone tubing. The system was primed with liquid and the propelling liquid meniscus in the glass capillary was used as measure for the dispensed volume. The location of the meniscus during actuation was continuously recorded by a digital camera which resulted in a read-off error equivalent to approximately $0.39 \mu\text{l}$. Figure 6 shows a schematic drawing of the measurement setup together with a photograph of the dispenser.

The same experimental setup was used to test liquid delivery against different counter pressures. Air pressure up to 75 kPa was applied to the open end of the glass capillary and the movement of the liquid meniscus was observed. The applied counter pressure was measured by a pressure transducer (Motorola MPX 100DP). The actuation power was the same for all the tested counter pressures but the actuation time, which was stopped when steady-state was reached, was different.

Figure 6. Schematic drawing of the measurement setup for measuring delivery precision and work against counter pressure.

The temperature of the liquid in the reservoir was measured by placing a small NTC-thermistor (Mitsubishi TN10) inside the liquid container. The thermistor was placed in the middle and in close contact with the membrane where the highest temperature occurs. Two effects were considered during this test: the maximum temperature in the liquid during actuation and the ability of the material to actively insulate from heat. Therefore the temperature was continuously recorded throughout the actuation using a computer running LabView[™].

5. Results and discussion

The device was successfully tested with DI water as the delivery liquid and with a dispensed volume of $101 \mu\text{l}$. Figure 8 shows typical dispensing curves.

The following subsections report results for the three main investigated properties: precision in delivery, ability to work against counter pressures and temperature.

5.1. Delivery precision

Precision measurements were made by testing the device 20 times by successive re-filling of expandable paste and dispenser liquid (DI water). The PCB-heater was heated with a power of 1 W during a 150 s actuation. The mean dispensed volume after 150 s was measured as $101 \mu\text{l}$ with a relative standard deviation of 3.2%. No back-flow was observed after the actuation was ended.

The precision of this device is high considering that the device was manually assembled. Deviations from the nominal value are likely caused by the level of flatness of the separating vinyl membrane and the filling procedure. Figure 7 shows photographs of the expandable material in different states. Figure 7(a) shows the unexpanded state and figure 7(b) shows the expanded state corresponding to an expansion of approximately 200%. Note the conformity of the expanded actuator shape with the cone shaped structure of the reservoir. The third picture (figure 7(c)) shows the result of an unconstrained expansion of the material, i.e. when no liquid container was used. This expansion corresponds to an expansion of 600%.

5.2. Counter pressure

Figure 8 shows a graph of the measured dispensed volume against different counter pressures in the range of 0 kPa to

Figure 7. Photographs of the expandable paste on the PCB-heater. The initial volume (a) is 35 μl .

Figure 8. Dispensed volume as a function of time for different counter pressures.

75 kPa. The actuation power in these tests was 1 W and from the graph it is evident that the actuator is strong enough to deliver against these pressures. However, as can be seen in the graph, when delivering against counter pressures the actuator does not reach up to the nominal value of 100 μl . The reason for this is that the expandable microspheres are compressible with the fully expanded spheres being the most compressible ones [18]. Partly expanded microspheres, however, can withstand pressures up to 500 kPa without being compressed considerably. In the current design though, the expansion level over the expandable paste is not constant as a result of the non-homogeneous heating. The microspheres closest to the heater will be more expanded than the ones far from the heater. As a consequence the expansion potential of the spheres is not used optimally. Therefore a larger actuation power or a more homogeneous heating would not only lead to a more rapid expansion but also to more evenly distributed expansion levels of the spheres, a lower compressibility and thus improved delivery against counter pressure.

5.3. Temperature

The temperature of the delivery liquid is important to control to avoid damage on possible temperature sensitive liquids such as drugs. Figure 9 shows the measured temperature of the liquid during 150 s of actuation with 1 W. The measurement shows that during a short period the temperature peaks at 59 $^{\circ}\text{C}$. It can also be seen that after approximately 70 s the temperature rise decreases. As can be seen from figure 8, this corresponds to the time when the expandable material starts

Figure 9. Temperature in the dispensed liquid during actuation. The temperature is measured at the membrane interface in the center of the device.

to expand (i.e. the time at which the material has reached the transition temperature of 70 $^{\circ}\text{C}$). As the expansion continues the material turns into an efficient heat insulator and the temperature decreases, despite the device still being heated.

For drug delivery applications and especially protein-based drugs, the temperature should not be higher than 40 $^{\circ}\text{C}$; therefore a peak temperature of 59 $^{\circ}\text{C}$ is too high. For such applications a thermal buffer (e.g. an intermediate material) or a better thermal insulator than the thin flexible membrane would be needed between the expandable paste and the delivery liquid.

Another optimization point concerns the heat insulating effect. Since the expandable paste starts to insulate as soon as the transition temperature is reached, it indicates that a non-constant heating scheme might be advantageous. For example, a short pulse with higher heating power in the beginning of the actuation cycle would give a large expansion but also insulate against further heating. If such an optimized procedure is fast enough it would suppress heating of the delivery liquid.

6. Possible applications

As discussed earlier, the primary target application is disposable drug delivery and especially a unit which can be used in conjunction with microneedles to precisely deliver physiologically relevant liquid volumes. The relatively uncomplicated design is realized using only low-cost materials and processes and is hence a good candidate for disposables. The investigated prototype dispenses 100 μl . Since the volume is determined by the size of the reservoir, other dispensing volumes can be achieved by simply replacing the reservoir. The ultimate limit for the smallest possible dispensing volume is essentially the size of the microspheres.

For dispensing larger volumes, the dispenser in general requires more expandable paste and thus more energy for heating. However, as illustrated in figure 7(c), the 35 μl of paste used in the demonstrator is capable of filling out a volume of approximately 250 μl . This means that larger volumes can be dispensed even without increasing the actuation energy or the amount of microspheres.

Another issue concerning drug delivery is the time needed to dispense. From figure 8 it can be seen that the time needed to start dispensing is about 50 s and thus this is the time it takes to heat the microspheres to the transition temperature. The demonstrator device is not optimized in terms of heat transfer or power consumption. Therefore, several measures can be made to achieve faster dispensing. For example, increasing the thermal conductivity of the paste by modifying the microsphere matrix or improving the heat transfer by a suitable heater design would decrease dispensing time. This is also the case if the dispenser is made smaller.

For drug delivery applications though, the most critical issue concerns the heating of the delivery liquid. Also concerning this issue, a number of additional actions can be made. For instance, the wall material of the liquid reservoir can be changed to suppress heating, the cone-shaped cavity of the liquid reservoir can be replaced with a more efficient semi-spherical shape (cf figure 7(b)–(c)). The vinyl membrane can be replaced with a better heat insulator, e.g. a sheet of a highly elastic polymer incorporating expanded microspheres would probably be a good candidate since the gas-filled spheres insulate well.

Another possible application for the device is pure dispensing with very low flow rate. The flow rate can be varied by adjusting the applied power to the heater, i.e. less actuation power leads to slower expansion which in turn results in a lower dispensing rate. This property was utilized and experimentally verified in a special application where a low flow rate was needed. Using the same dispenser configuration as described in section 3, the device was used in a microfluidic dye laser system to create a regenerating flow of Rhodamine dye [19]. This application requires a low flow rate rather than delivery of a precise dose. Therefore the device was evaluated for variable flow rates by decreasing the supplied actuation power. Flow rates were measured using the measurement setup depicted in figure 6 except that a finer capillary was used to reveal small changes. The lowest measured flow rate was 1 $\mu\text{l h}^{-1}$. This low flow rate can be compared with the nominal flow rate of the standard dispensing mode of approximately 2400 $\mu\text{l h}^{-1}$ (100 μl during 150 s). However, the current design is not intended for constant flow rates and the expansion, especially at low dispensing rates, is not completely linear.

The microfluidic dye laser system required a flow rate between 1–10 $\mu\text{l h}^{-1}$. To be within this range the dispenser was set to deliver 2 $\mu\text{l h}^{-1}$ corresponding to a supplied power of 301 mW. The device was successfully tested, operating for 11 h. With the low flow rate the device can potentially operate up to 50 h which is more than sufficient for the application. The maximum temperature in the delivery liquid was measured to 39.5 °C and did not influence the function of the dye laser.

Other important application related issues concerns shelf-life and contamination risks between paste and delivery liquid (e.g. membrane permeability). These and other long-term

effects were, however, not explicitly studied in this work and thus needs to be further investigated.

7. Conclusions

We have introduced a new low-cost dispenser device suitable for liquid delivery in the microliter to milliliter range. The dispenser uses a compact actuator based on expandable microspheres in which the active material expands approximately 200%, displacing a stored liquid volume.

The device was successfully tested with a mean dispensed volume of 101 μl and a standard deviation of 3.2% without indications of any back-flow. It was shown that the device can deliver against counter pressures as high as 75 kPa. However, delivering against counter pressures limits the dispensed volume. The maximum temperature in the dispensed liquid peaks at 59 °C during a short period but decreases because the expanding material turns into an efficient heat insulator. The device also functioned as a low flow rate dispenser and can be tuned to deliver flow rates between 1 $\mu\text{l h}^{-1}$ and 2400 $\mu\text{l h}^{-1}$ by adjusting the actuation power.

The present demonstrator device leaves enough room for improvements to increase precision, reduce required actuation energy and to adapt the device to thermal requirements of the delivery liquid. The device forms a potential candidate for low-cost active liquid delivery of relatively large volumes for use in microfluidic dispensing or drug delivery applications.

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