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Inkjet print microchannels based on liquid template

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Cite this: DOI: 10.1039/xoxxoooooox

Received ooth January 2012,
Accepted ooth January 2012

DOI: 10.1039/xoxxoooooox

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A simple method to fabricate microchannels is demonstrated based on inkjet printing liquid template. The morphology of the liquid template can be well controlled by adopting ink with viscosity sensitive to temperature. The as-prepared Y-shape microchannel is used as a microfluidic reactor for acylation fluorigenic reaction in matrix of polydimethylsiloxane (PDMS). Arbitrary modification of the microchannels could be easily realized synchronously with the formation of the microchannels. By grafting polyethylene glycol (PEG) to the internal surface, an anti-biosorption microchannel is achieved. The facile method will be significant to fabricate microfluidic chip with functional modification.

Introduction

Microchannel system is an important tool to realize fluidics at micro level, which provides a powerful platform for the fields of biochemistry[1-4], nanoscale manipulation[5-8], digital fluidic devices[9] and bionics[10-12]. So far, the fabrication of microchannel system mainly relies on the photolithography-based techniques[13], such as imprint lithography[14-16], soft lithography[17] and electron beam lithography[18]. However, the photolithography-based technique highly depends on photo-mask and photosensitive materials, which limits the flexibility of fabrication[19]. As an efficient technique, printing technology has shown great prospects in pattern-based fabrication[20-22] and functional materials adopting[23], electronic device[24, 25], plate-making and microchannel[26, 27]. To fabricate microchannels by printing technique, a few strategies have been proposed. For example, do Lago and Wheeler et al. have developed an inkjet printing method to construct groove structure for microfluidic device[28-31]. However, post bonding or laminating step was needed to achieve enclosed microchannel. Furthermore, sacrifice template strategy was exploited by Lewis et al., in which enclosed mircrochannels were fabricated in one step[32]. Generally, polymer-based inks, such as wax[33], shellac[34] and sucrose[35], were used to prototyped solid template. However, polymer-based inks highly depend on the extrusion direct write printing due to their high viscosity[36], and it is complicated to remove the template. In contrast, liquid template could be easily achieved by inkjet printing and removed by evaporation. However, the controlling of Rayleigh instability[37] is a great challenge for building liquid template with controllable morphology. That is, under the effect of surface tension, the liquid pattern in plastic surrounding, such as air or liquid, tends to breakup and relaxes into discontinuous spherical droplets[38-40], which will break the integrity of template. Therefore, to obtain stable liquid template, the Rayleigh instability must be inhibited. In this paper, the Rayleigh instability is successfully inhibited by adopting the materials with viscosity sensitive to temperature. The liquid template with stable morphology is prototyped by inkjet printing and used to fabricate microchannel. A typical microfluidic reactor is demonstrated by acylation fluorigenic reaction in matrix of PDMS. Furthermore, arbitrary modification of the microchannels could be realized synchronously with the formation of microchannels through interfacial reaction. By grafting PEG to the internal surface, an anti-biosorption microchannel is obtained. The as-prepared microfluidic reactor and anti-biosorption microchannel will be of significance for the biochemical microfluidic device fabrication.

Fig. 1 is the scheme for the fabrication process of a typical microfluidic reactor by inkjet printing. The first step is pouring the PDMS prepolymer mixture into a container **(a)**. Then the Y-shape pattern is printed on the surface of the prepolymer mixture **(b)**. The Y-shape pattern is wrapped into prepolymer mixture spontaneously and acts as template **(c)**. Next, the prepolymer mixture thermally cures. Meanwhile, the liquid template evaporates, leaving the Y-shape microchannel in the PDMS matrix **(d)**.The matrix is peeled off from the container **(e)**, and a typical microfluidic reactor is obtained **(f)**.

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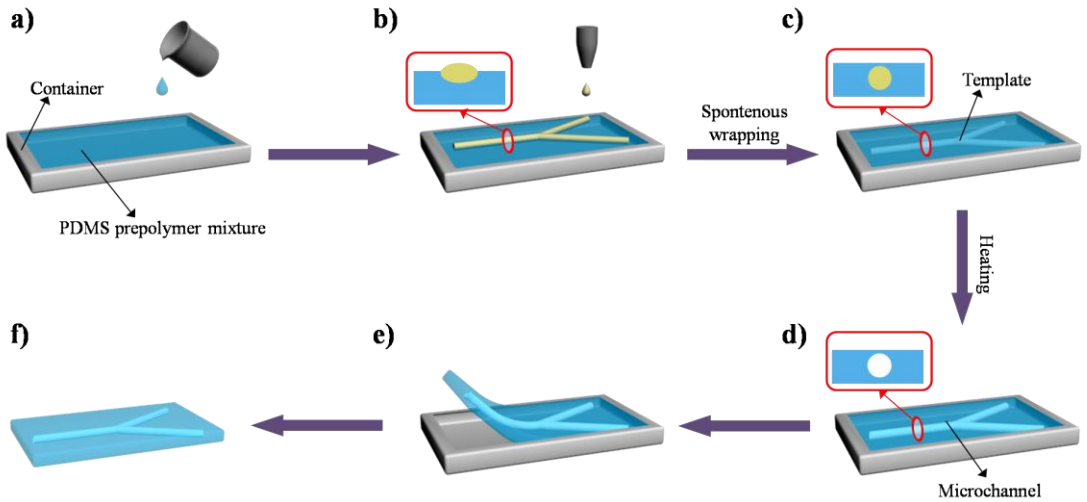

Fig. 1 Scheme for the fabrication process of a typical microfluidic reactor by inkjet printing: **a)** Pouring PDMS prepolymer mixture into the container; **b)** Printing a Y-shape pattern on the surface of prepolymer mixture. Inset: Relative position sketch of pattern and prepolymer mixture surface; **c)** Standing for a few seconds to allow the pattern to be wrapped spontaneously. Inset: Relative position sketch of pattern and prepolymer mixture surface; **d)** After heating the prepolymer mixture with liquid template, the prepolymer mixture thermally cures and the liquid template evaporates, leaving the microchannel in PDMS matrix. Inset: Section sketch of microchannel in matrix; **e)** Peeling off the fabricated PDMS matrix with microchannel; **f)** The typical microfluidic reactor in PDMS matrix.

Experimental

Prepolymer mixture for printing substrate

Prepolymer mixture for printing substrate was prepared by mixing PDMS prepolymer and crosslinker (Slygard 184, Dow Corning) at the mass ratio of 10:1 and deformed by centrifuging at 200 rpm (ZONKIA HC-3018). Then the mixture was poured into a container with a depth of about 1 mm, and kept it a period of time to flow level.

Ink

The ink was prepared by mixing undecanol (Alfa Aesar Inc., Chinese) and ethyl lactate (Alfa Aesar Inc., Chinese) at the mass ratio of 3:1 (see **ESI† S1**). For functional modification of the internal surface of microchannel, vinyl-terminated Poly (ethylene glycol) methacrylate (OEGMA) was added into the ink at 5 wt %.

Wetting behaviour of the ink on the surface of prepolymer mixture

PDMS prepolymer mixture was poured into a rectangular glass tank and a droplet of the ink (about 3μL) is injected onto the surface of the prepolymer mixture. An OCA20 machine (dataphysics, Germany) was used to observe the wetting behaviour of the droplet on the surface of PDMS prepolymer mixture aside.

Printing

The prepared ink was injected into a cartridge and printed by a Dimatix Materials Printer (FUJIFILMDMP-2800 series, Japan) with a 10 pL drop orifice (DMC-11610). The glass container with prepolymer mixture was placed on the platform of the printer. The temperature of the glass container was controlled by water bath. A Y-shape pattern was inkjet printed on the surface of prepolymer mixture at a drop space of 5 μm. The morphology stability of the pattern was observed by a build-in camera of the printer. After printing, the PDMS prepolymer mixture was heated at 90℃ for 3h in oven.

Wetting and anti-biosorption properties of microchannels

To examine the wetting property, about 1 μL deionized water was injected into the microchannels by a syringe. An optical microscope with CCD camera was used to observe the meniscus morphologies of water in the microchannels and evaluate the contact angle of water on the internal surface of the microchannel.

FITC-labelled BSA protein (10 mg/ml, Bellancom Chemistry) was dissolved in borate buffer solution (pH=7.4) at the volume ratio of 1:10. To examine the anti-biosorption, the prepared FITC-labelled BSA protein solution was injected into microchannels and then incubated in the dark at 37℃ for 1 h, followed with rinsing with borate buffer solution. Fluorescence images were recorded on an Olympus fluorescence microscope (BX51, Olympus Co., Ltd) (excitation, 495 nm; emission, 525 nm). The average fluorescence intensity is directly proportional to the amount of adsorbed proteins.

Characterizations

Rheology property of the ink at different temperature was characterized by an AR2000EX Rheometer (TA instrument) with a parallel flat plates (D=40mm). A gap of 1000 μm was used, and the temperature swept from 40 ℃ to 0 ℃ with a cooling rate of 2 ℃/min. All measurements were conducted at a constant shear rate of 10 rad • S^{-1}. The densities of ink and prepolymer mixture were determined by the weight-volume method. SEM images were obtained using a field-emission scanning electron microscope(S-7500, Japan Hitachi). X-ray photoelectron spectroscopy (XPS) was performed to analysis the content of elements on the Thermo Scientific ESCA Lab 250Xi using 200 W monochromated Al Kα radiation. The 500 μm X-ray spot was used for XPS analysis. The full image of the microchannel reactor was captured by a digital camera 60D (Canon Co., Ltd).

Results and Discussion

When the ink droplets are printed on the surface of prepolymer mixture, it is needed that the prepolymer mixture to wrap the ink droplets to form liquid template. In the case of an ink droplet wetting on the surface of the liquid substrate, four possible equilibrium regimes have been proposed[41]: (1) total wetting where the ink droplet spreads until it reaches an equilibrium thickness on the order of molecular size; (2) partial wetting where the ink droplet forms a lens on the surface of liquid substrate; (3) pseudopartial wetting where the ink droplet forms a lens with a thin film covering the liquid substrate surface; (4) pseudototal wetting where the ink droplet forms a lens covered by a thin film of the liquid substrate.

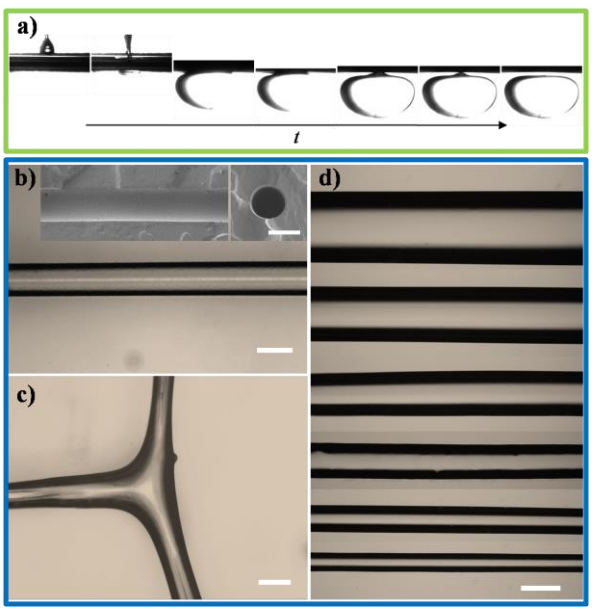

Fig. 2 a) Wetting behaviour of an ink droplet on the surface of PDMS prepolymer mixture; **b)** Optical image of a microchannel with diameter of about 100 μm (scale bar=100μm). Inset: SEM images of cross section (right) and longitudinal section (left) of a 100μmmicrochannel (scale bar=100μm); **c)** Optical image of a branch-shape microchannel (scale bar=50μm); **d)** Optical images of microchannels with different diameters (200 μm-900 μm) (scale bar=500μm).

To obtain embedded template, pseudototal wetting regime is needed. **Fig. 2 (a)** presents the wetting behaviour of an ink droplet on the surface of PDMS prepolymer mixture, in which the serial patterns indicate the evolution process of wetting with time. As viewed from aside, the ink droplet forms a lens after being injected on the surface of PDMS prepolymer mixture. The lens is slowly wrapped by the prepolymer mixture in the following few seconds, which indicates the pseudototal wetting regime of the ink droplet on the surface of PDMS prepolymer mixture. Finally, the ink droplet is totally wrapped into the PDMS prepolymer mixture. The densities of the ink and the PDMS prepolymer mixture are measured to be 0.86 g/ml and 1.04 g/ml respectively (**Table S1**). Due to the lower relative density, the ink droplet will not sink down in the prepolymer mixture continually, which will guarantee the stable position of template in the prepolymer mixture (see **Fig. S2**).

Similar to the behaviour of an ink droplet discussed above, the pattern printed on the surface of PDMS prepolymer mixture could also be wrapped into the prepolymer mixture. During the thermal curing of PDMS prepolymer mixture at 90 ℃, the pattern wrapped in the substrate play the role of template. With the holding time increasing, liquid template evaporates, leaving the microchannel in PDMS matrix. **Fig. 2 (b)** shows the microchannel with a diameter of about 100 μm. The insets present the cross section morphology, showing the smooth and cylindrical profile of the microchannel. The diameter of microchannels could be easily controlled by designing the scale of template printed. As shown in **Fig. 2 (d)**, the microchannels with the diameters of about 200 μm, 300 μm, 400 μm, 500 μm, 600 μm, 700 μm and 900 μm are successfully fabricated.

In addition to the linear microchannels, branch-shape microchannels are also achieved. Due to the effect of curvature difference, the sharp angle between adjacent branches is unstable. The mass transfer driven by the curvature difference[42] in the template would turn the unstable sharp angle into the stable fillet angle (details in **ESI† S2**), which sequentially leads to the formation of the microchannels with fillet angle, as shown in **Fig. 2 (c)**.

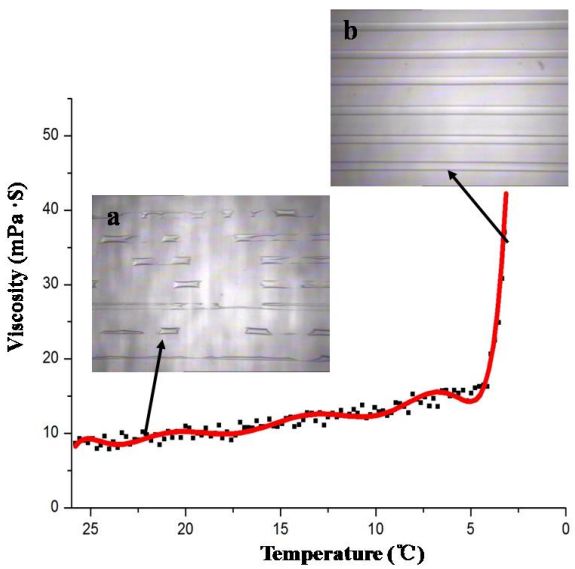

Fig. 3 Rheology property of the ink at different temperature. Inset**:** The digital photography of the pattern printed on the surface of PDMS prepolymer mixture at about 22 ℃ **(a)** and at about 3~4 ℃ **(b)**.

For the formation of microchannels, the morphology of the liquid template should be well controlled. Generally, a liquid

thread in another liquid tends to breakup and relaxes into uncontinuous spherical droplets under the effect of surface tension[43], which will break the continuous morphology of liquid template. Rayleigh et al has revealed that higher viscosity could contribute to reduce the breakup of liquid thread, which can be represented by the relaxation time τ, which is equal to $\eta^3/(\rho\gamma^2)$[37,44], where η is the viscosity of the liquid thread, γ is the interfacial tension and ρ is the density difference between two phases. According to the above discussion, manipulating the viscosity of the liquid template (η) could be used to control the Rayleigh instability (τ). That is, Rayleigh instability would be inhibited by simply increasing the viscosity of the liquid template. To achieve this, the ink with viscosity sensitive to temperature is adopted to prototype the liquid template, while PDMS prepolymer mixture with high viscosity is adopted as the liquid substrate. **Fig. 3** presents the relationship of the rheology property of the ink with temperature in the range of about 25 ℃ to 3℃. At 22℃, the viscosity of ink is about 9.8 mPa·S. While at around 4℃, the viscosity increases to above 40 mPa • S rapidly.

The insets in **Fig. 3** show the continuity of pattern at different temperature. When the temperature of PDMS prepolymer mixture is controlled at 22 ℃, the pattern lines printed on the surface of prepolymer mixture are unstable and break as shown in **Fig. 3 inset (a)**. Due to the high viscosity of substrate, the individual parts relax into spherical droplets slowly compared with the normal liquid thread in air[45]. When the temperature of PDMS prepolymer mixture is decreased to about 3~4 ℃, the viscosity of ink increases to above 40 mPa·S at the moment of contacting the cold prepolymer mixture surface. It is shown that the pattern lines printed on the surface of prepolymer mixture are quite stable and have no sign of any break in **Fig. 3 inset (b)**.

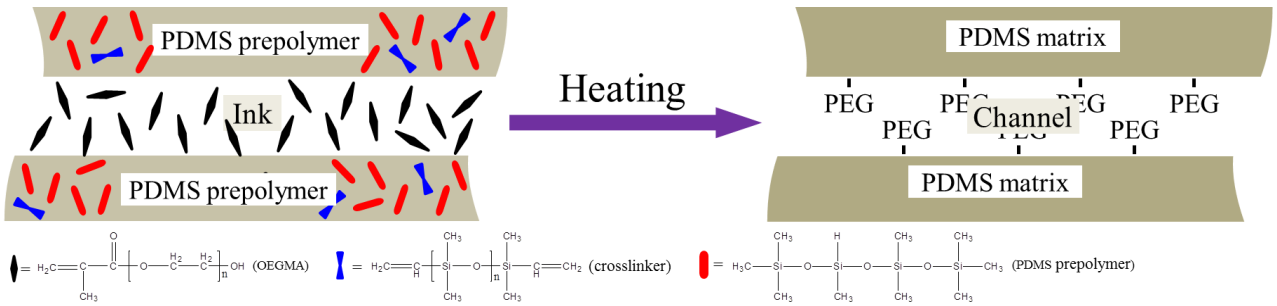

Fig. 4 Scheme of synchronous modification based on hydrosilylation reaction. The vinyl-terminated OEGMA in ink takes part in the hydrosilylation reaction at the interface of ink and prepolymer mixture.

Functional modification has been an important research topic in the application of microfluidic device[46], in which post-treatment is usually needed to graft functional groups on the inner surface of the preformed microchannel[47]. Here, the unique formation mechanism of microchannels using liquid template implied that we might simply the two-step procedure to one step via interfacial reaction during the formation of the microchannels. According to **Eq. S1 (1)**, the hydrosilylation is the basic reaction of PDMS curing, and vinyl is the key reactive chemical group that react with silane[48]. Adding vinyl-terminated functional molecules to the template is a convenient way to chemically modify the PDMS[49]. **Fig. 4** shows the scheme of synchronous modification of PEG based on hydrosilylation reaction. The vinyl-terminated poly(ethylene glycol)methacrylate (OEGMA) molecules are added into the ink. After being printed, the ink droplets are wrapped into the PDMS prepolymer mixture subsequently. During the thermal curing of PDMS prepolymer mixture, the vinly-terminated OEGMA molecules participated in the hydrosilylation reaction at the interface of the ink and PDMS prepolymer mixture (see **Eq. S1 (2)**). Following with the formation of the microchannels, the PEG groups are grafted onto the internal surface of PDMS microchannel ultimately. The existence of PEG groups on the internal surface of micorchannels has been confirmed by the high resolution X-ray photoelectron spectra (XPS) of C 1s peaks, as shown in **ESI† S5**. Based on this strategy, arbitrary modification could be realized by adding vinyl-terminated functional molecular into the ink.

To visualize the wetting effect of modifying PEG group to PDMS microchannel, the meniscus morphologies of water in PEG modified microchannel and unmodified microchannel are shown in **Fig.5 (a)**. In unmodified microchannel, the meniscus morphology shows the contact angle of about 90°, which reflects the hydrophobic property of PDMS. While in PEG modified microchannel, the contact angle of water is only about 60°. When one end of microchannel was immersed into water, a capillary rise was observed in the PEG modified microchannel but no rise in the unmodified microchannel (see **ESI† S6 and Fig. S10**). The favourable wettability of PEG modified microchannel makes it promising in anti-biosorption application[50].

The anti-biosorption property of PDMS grafted with PEG groups plays a crucial role in the application of microfluidic chip for biochemistry[49]. In this work, FITC-labelled BSA protein was used to test the anti-biosorption property of microchannels. **Fig.5 (b)** shows the fluorescence intensities of internal surfaces for the unmodified and PEG modified microchannel after FITC-labelled BSA protein incubation for 1h at 37 ℃. The nonspecific adsorption of BSA protein on the internal surface of PEG modified microchannels is significantly decreased compared with the unmodified PDMS microchannels. Therefore, the anti-biosorption property of PDMS microchannels is improved greatly after being modified with PEG groups.

An organic phase chemical reaction was performed to demonstrate the application of the as-prepared microfluidic reactor. A typical Y-shape microfluidic reactor was fabricated and the acylation fluorigenic reaction was carried out. None-fluorescent dansyl chloride and tetraethylenepentamine solution in ethanol were injected into the two branches of the Y-shape microreactor respectively (**Fig. 5 (c)**). When the two reagents met at the intersection, fluorescent dansyl-amide was produced and gave a clear fluorescence emission under 365 nm ultraviolet light exposures. The organic phase reaction attempts reveal the wide adoptability of fabricating microchannels by inkjet printing liquid template.

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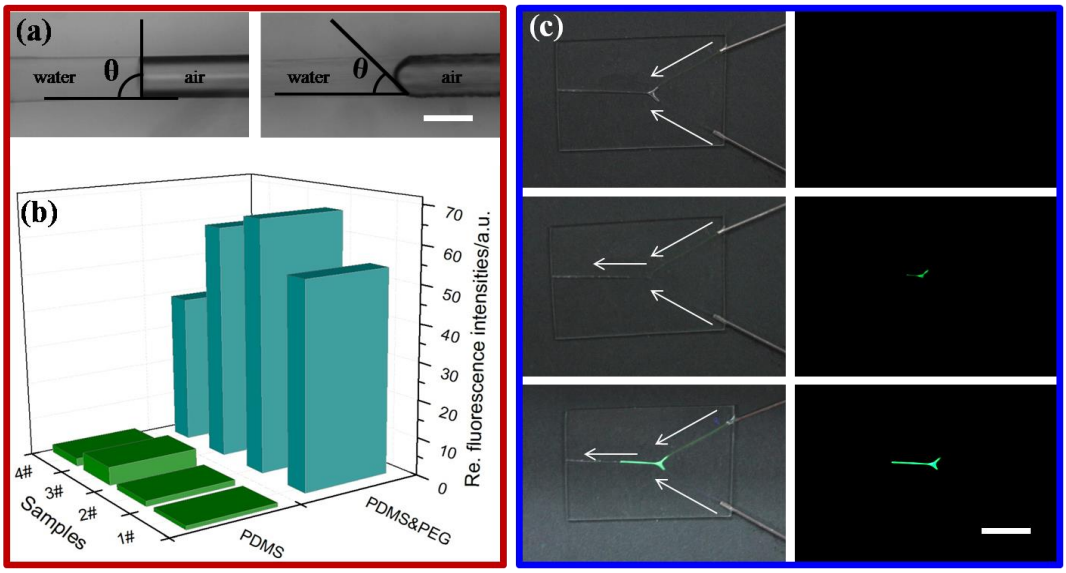

Fig. 5 a) The images of meniscus morphology of water in PDMS microchannel (left) and PEG modified microchannel (right) (scale bar=1000 μm); **b)** Fluorescence intensity of surface of unmodified PDMS and PEG modified microchannels after FITC-labelled protein incubation followed by borate buffer rinsing; **c)** Digital photographs of bioluminescent reaction in a typical microfluidic reactor (scale bar=1 cm).

Conclusion

In summary, we have demonstrated the fabrication of microchannels based on liquid template by inkjet printing. The morphology of the liquid template was well controlled by adopting ink with viscosity sensitive to temperature. It is explored how to inhibit the Rayleigh instability and achieved a liquid template with stable morphology in PDMS prepolymer mixture. Through interfacial reaction, PEG is grafted on the inner surface of PDMS microchannel. This approach could perform the arbitrary modification on the internal surface of the PDMS microchannels synchronously with the formation of microchannels by adding vinyl-terminated functional molecular into the template. The strategy for fabrication and synchronous modification microchannels has vast prospect on complex vascular network with biological function, such as self-healing and bio-absorption.

Acknowledgements

F. Y. Li and Y. L. Song thank the financial support of 973 Program (Nos. 2013CB933004), the National Nature Science Foundation (Grant Nos. 51473173, 51203166, 51473172, 21301180 and 21303218), and the "Strategic Priority Research Program" of the Chinese Academy of Sciences (Grant No. XDA09020000).

Notes and references

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†Electronic Supplementary Information (ESI) available: S1. Preparation of printable ink; S2. Curvature analysis of the branched microchannel; S3. Curvature analysis of the junction between two channels with different diameters; S4. Fabrication resolution of microchannels; S5. Different in capillary rise between unmodified microchannel and PEG modified microchannel. See DOI: 10.1039/b000000x/

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