Electroosmotic flow pumps with polymer frits

Shulin Zeng[a,*], Chuan-Hua Chen[a], Juan G. Santiago[a], Jing-Ran Chen[b],
Richard N. Zare[b], Jennifer A. Tripp[c], Frantisek Svec[c], Jean M.J. Fréchet[c]

[a]*Department of Mechanical Engineering, Stanford University, Stanford, CA 94305, USA*
[b]*Department of Chemistry, Stanford University, Stanford, CA 94305, USA*
[c]*Department of Chemistry, University of California at Berkeley, Berkeley, CA 94720, USA*

Abstract

Electroosmotic flow (EOF) pumps with flow rates on the order of 1 ml/min have been designed and fabricated. These pumps use EOF to propel liquid solutions in a compact system with no moving parts. The pumping chamber is filled with densely packed non-porous silica particles, and the particle bed is held in place using two polymeric porous frits. The pump frame is made of acrylic, the frits are made using UV photopolymerization within the custom-built acrylic frame, and the particles are packed using a novel side-bore packing technology. Platinum wire electrodes on both sides of the pump provide the electric field to drive the flow. Deionized water is used as a working fluid in order to minimize Joule heating and increase thermodynamic efficiency. The maximum flow rates and maximum pressures generated by the pumps are 0.8 ml/min and 2 atm, respectively, at 1.0 kV applied voltage. © 2002 Elsevier Science B.V. All rights reserved.

1. Introduction

Electroosmotic flow (EOF) micro- and meso-scale pumps with packed silica particles can generate pressures several orders of magnitude higher than microfabricated pumps [1–3]. EOF pumps based on capillary systems packed with silica particles are suitable for applications requiring pressure higher than 10 atm and flow rates as high as a microliter per minute [1]. However, many applications require flow rates on the order of several milliliters per minute. For example, the two-phase electrokinetic microchannel cooling system for VSLI chips developed by Jiang et al. [4] needs flow rates of 5 ml/min in order to remove heat power of 30 W/cm^2. Gan et al. [5] have demonstrated EOF pumps which consist of sintered glass bead structures and are capable of generating flow rates in the range of few milliliters per minute. However, because of a relatively high pore diameter of approximately 2–5 μm, this pump is limited to a pressure per voltage performance of approximately 0.003 atm/V. Other fields that may benefit from compact, high-flow rate EOF pumps include systems designed to cool solid-state laser devices and miniaturized systems for high-throughput high-pressure liquid chromatography.

A simple way to increase the flow rate of EOF pumps is to increase the cross sectional area and to reduce the length in the flow direction, while maintaining the small pore size required for high-pressure generation [1]. One of the challenges of fabricating packed-particle EOF pumps with large cross sectional area and short flow length includes making a reproducible, void-free porous frit (i.e. a filtering/sieving porous structure) that can withstand the large forces associated with high-pressure pumping and high-pressure packing. Another challenge is engineering of the fittings and packing method required to achieve a densely packed particle structure between the frits. Such frits need to be dielectric materials with pore sizes small enough to retain 1–3 μm silica particles, but not so small that they effectively block pressure-driven flow. Thermal sintering of silica beads is commonly used to make frits for capillary electrochromatography columns [6]. This method is simple and effective for columns with an inner diameter of 100 μm or less. Zeng et al. [1] developed sodium-silicate method to make frits for electrokinetic pumps of 500–700 μm inner diameter. However, the above two methods are not applicable when the diameter of the pump body is larger than 1 mm. Commercially-available PEEK [7] or glass frits [8] are not suitable because they do not have adequately uniform pore diameter distribution with an average size smaller than 3 μm (e.g. <0.5 μm standard deviation).

* Corresponding author. Tel.: +1-650-723-2171.
E-mail address: zengss@stanford.edu (S. Zeng).

Viklund et al. [9] have developed porous polymers with easily controlled pore sizes that are very promising for making large frits. For example, Chen et al. [10] have successfully used Viklund's recipes to fabricate large-bore polymer frits in fused silica capillaries for capillary electrochromatography columns. In the present work, we have adopted the polymers described by Viklund et al. [9], and Chen et al. [10] to make even larger-bore polymerized frits within our pump frames. The application of these frits makes the fabrication process of electroosmotic pumps versatile as these frits can be lithographically defined into a variety of channel structures including large (>1 cm diameter) pumping regions and etched or embossed microchannel structures [11]. The side-bore packing method we are describing here allows us to use 1–3 µm non-porous silica particles as a pumping structure. Non-porous silica particles are favorable because they are a robust and well-characterized material for EOF applications [2,6,10].

This paper presents a brief report on our developments of high-flow rate EOF pumps using a novel in situ polymerized frit/packed-particle bed design. The fabrication process of the pumps and performance characteristics including the pressure and flow rates are described.

2. Fabrication

The components and fabricating procedures for the pump design are shown schematically in Fig. 1. The EOF pump consists of two polymer frits that sandwich a layer of densely packed silica particles as shown in Fig. 1(c). The frits act as filters that allow liquid to pass and retain silica particles of 1–3 µm diameter. The EOF pump is fabricated within an acrylic structure. We first fabricate the three pieces shown in Fig. 1(a) which are the two frit frames and the pump body. The two frit frames are screw-threaded in order to increase the joint/bond strength between the frits and the frame so that the frits can withstand a large thrust force associated with large pressure packing and pumping. The effective pumping cross section is about 1 cm in diameter. The three parts are bonded together as described below to form the pump.

The chemicals for making the polymer frits were purchased from Aldrich (Milwaukee) and used without any treatments. The monomers are trimethylolpropane trimethacrylate (TRIM) and 2,3-epoxypropyl methacrylate (glycidyl methacrylate, GMA). The porogens include toluene and 2,2,4-trimethylpentane (isooctane). The initiator is α-methoxy-α-phenylacetophenone (benzoin methyl ether). In general we followed published procedures for photopolymerization [9,10]. After the photopolymerization, soluble materials were removed from the frits by washing with ethanol or DI water using a high-pressure liquid chromatography pump. We found it very useful to reface (i.e. machine flat using a mill) the surface of the frits after polymerization. The typical thickness of frits is about 5 mm.

Fig. 2 shows a scanning electron microscope (SEM) image of a polymer frit. The photopolymer sample shown in Fig. 2 was polymerized in a 75 µm i.d. fused silica capillary for the purpose of this visualization using the same materials and procedures as used in the current work. These frits provide a strong porous structure to hold the silica particles in place in high-pressure and high-flow rate applications. After the
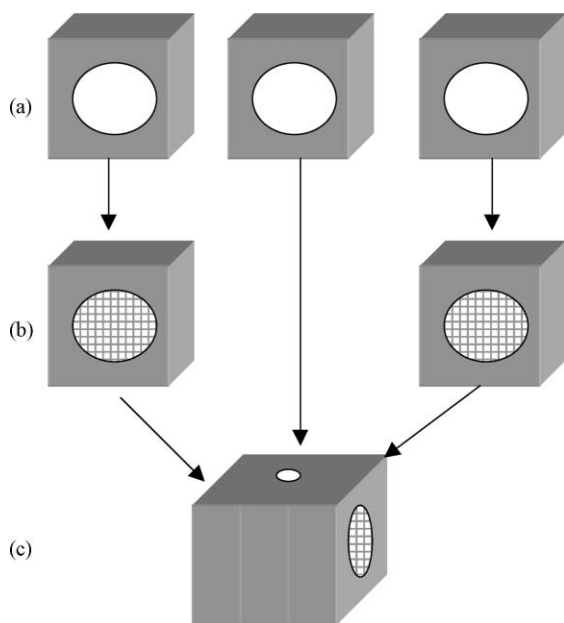

Fig. 1. Schematic illustration of parts and procedures for fabricating electroosmotic flow pumps. (a) three similar frames are fabricated in acrylic; (b) frits are polymerized into two of the frames; and (c) the three frames are bonded and particles are loaded through a side port on the middle frame.
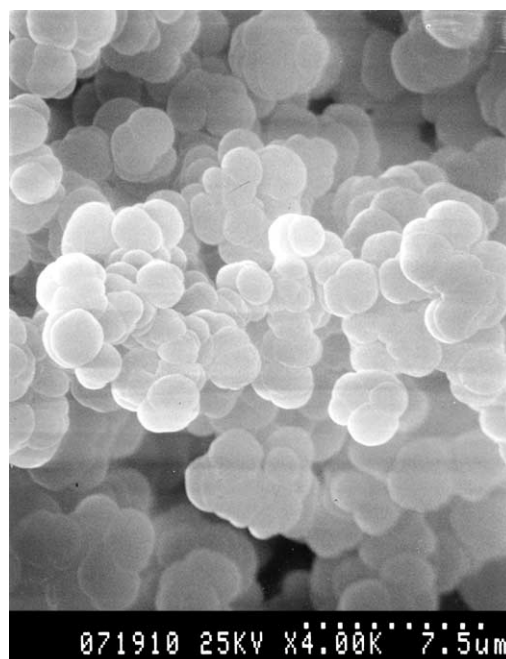

Fig. 2. SEM picture of polymer frits.

frits are fabricated, the three pump components are bonded together using Weld-on 3 (IPS Corporation, Gardena, CA) a solvent for acrylic. A small, side hole to the pumping chamber is drilled into the pump chamber (the frame in the middle). The bonded structure with a side hole is schematically shown in Fig. 1(c). This side hole is the packing channel for a slurry of non-porous silica particles (Micra Scientific Inc., Darien, IL). One end of a 5 cm long fused silica capillary (Polymicro Technologies Inc., Phoenex, AZ) is connected to the side hole using Loctite 352 UV curing epoxy (Loctite Corp., Rocky Hill, CT). The other end of the capillary is connected to a pressure-driven slurry packing system by plastic or stainless steel fittings (Upchurch Scientific, Oak Harbor, WA, and Alltech Associates Inc., Deerfield, IL), as previous described by Zeng et al. [1]. After the space between the two frits is packed with silica particles, the capillary is removed and the side hole is sealed using epoxy. The typical length of the packed-particle bed is about 2–4 mm.

3. Results and discussion

EOF pumps were characterized using setups and measurement procedures similar to those described by Zeng et al. [1]. Deionized water is used as working fluid. Flow rate is measured by weighing of the pumped fluid at the outlet versus time using an ACCULAB L-series weight scale (Newtown, PA). A 0–6 kV/5 mA Spellman power supply (Hauppauge, NY) is used to power the pump.

Buoyant forces move the electrolytic gas bubbles away from the electrodes and from the electrical current path of the system. We have designed the chambers of the electrodes in the pump (low and high-pressure chambers) so that the generated electrolytic gases escape from the device. As the pump operates and electrolytic reactions occur, the pH of solutions near the two electrodes change. Recirculation of the working fluid through the pump and through a closed-loop pumping system helps to homogenize the pH values and mitigates this effect. In general, pH variations can result in noticeable transients in flow rate and pressure [5].

Fig. 3 shows a plot of pump flow rate versus back pressure at 0.75, 1.0, and 1.25 kV applied voltages, respectively. The pump is 10 mm in diameter and about 15 mm in length, and is packed with 3.0 µm non-porous silica particles. The frits are about 5 mm thick. The working fluid is deionized water. The flow rates and the corresponding pressures obey linear relationships as previously predicted [1]. The extrapolated maximum flow rates of this pump would be 0.6, 0.8, and 1.2 ml/min at 0.75, 1.0, and 1.25 kV applied voltages, respectively. The extrapolated maximum pressures are about 1.4, 2.0, and 2.7 atm at the same voltages.

These EOF pumps generate much less pressure (2 atm at 1 kV) than the capillary EOF pumps (20 atm at 2 kV) [1]. This substantial decrease in pressure performance is probably due to the large flow resistance imposed by the (low
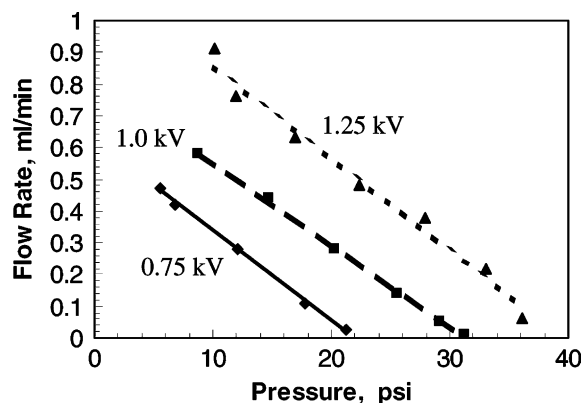

Fig. 3. Characterization of electroosmotic flow pumps at different applied voltages.

charge density) polymer frits. Although these frits allow for the fabrication of a large-bore pumping structure, they have negligible EOF, and the small pores add high fluidic resistance to each side of the pump. It is difficult to accurately calculate the flow resistance of the frits due to lack of pore structure parameters. As a rough estimation, the pressure drop across the two frits (10 mm of porous material in total) would be about 3 atm at a flow rate of about 1 ml/min if it is assumed that the frits have the porosity, tortuosity and pore size values of 40%, 1.6, and 1.0 µm, respectively; parameters similar to those for the packed-bed capillary pumps described by Zeng et al [1]. The pressure drop caused by the polymer frits was independently measured using a HPLC pump to flow water through the frits at 1 ml/min. The measured pressure drop is approximately 3 atm. The frits, therefore, have a fluidic structure similar to that for packed silica-particle beds. Reducing the thickness of polymer frits after the pumps are fabricated should be able to boost the pump efficiency greatly. However, decreases in frit thickness greatly reduce the mechanical strength of the frits.

Although the flow rate of the pumps described here is a factor of three smaller than that of the 3.5 cm diameter pump structures described by Gan et al. [5] there is an increase in the flow rate per unit voltage and unit area performance of the pump. The current pumps generate 0.001 (ml/min)/V/cm^2 which can be compared to the value of 0.0006 (ml/min)/V/cm^2 reported by Gan et al. [5]. There are several ways to potentially increase the flow rate of our device. First is reduction of the thickness of frits to minimize their flow and electrical current resistances. A second is a simple increase of the cross-sectional area of the pumps. Third is an increase of the pump pressure and flow rate performance by optimizing the buffer concentrations, including pH to achieve higher zeta potentials.

Fig. 4 shows total electric current through the pump versus applied electric voltages. In one of the experiments shown in the figure, the outlet was blocked so that there was zero net flow through the pump, which corresponds to the maximum pressure generation mode of operation. In a second experiment, there is zero net back pressure across the
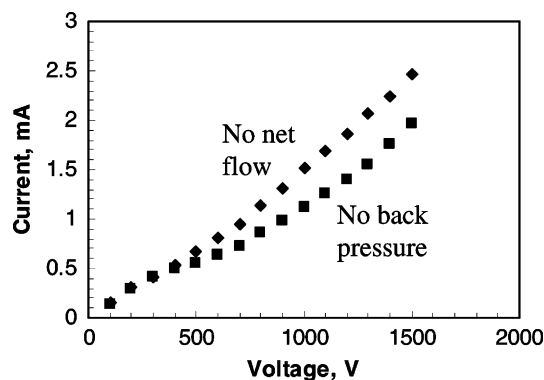
Fig. 4. Electric current vs. applied voltage at zero back pressure (squares) and at zero flow rate (diamonds).

pump, corresponding to the maximum flow rate mode of operation. When the applied voltage is <400 V the measured current in these two modes are nearly identical. At higher applied fields, approximately 30% higher currents are measured for the no flow mode than for the no back pressure mode. This observation may be explained by a speculation that fluids in the zero net flow mode have higher conductivity (liquids at higher temperatures have lower viscosity and therefore higher conductivity). In the zero net flow mode, the generated Joule heat is removed only by conduction through the pump. Whereas, the flow of working fluid through the pump helps remove the generated heat via forced convection in the no pressure mode. Finally, in both modes of operation the voltage–current curves have gradually increasing slopes as the applied voltage goes higher than about 1000 V. This observation is consistent with a temperature increase due to Joule heating and indicates that the convective cooling of the pumped flow stream is not sufficient to maintain the pump at constant temperature.

4. Conclusions

Key issues in the fabrication of a large EOF pump include fabrication of robust frits from a dielectric material and dense packing of small particles with high surface charge densities in the pumping chamber. EOF pumps with large flow rates have been fabricated by employing a novel photopolymerization method to make frits and by applying a side-bore packing method to produce a bed of 1–3 μm particles. The photopolymer described here offers significant design flexibility as it can be photopolymerized in a variety of structures including millimeter-scale flow passages and 10 μm and larger scale microchannels. The maximum flow rates and maximum pressures generated by the pumps are 0.8 ml/min and 2 atm, respectively, at 1.0 kV applied voltage. Flow rate capacity in the milliliter-per-minute range will significantly broaden the application spectrum of electroosmotic flow pumps.

Acknowledgements

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