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Pressure-driven spontaneous ion concentration polarization using an ion-selective membrane

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ABSTRACT

In this study, the spontaneous ion concentration polarization phenomenon induced by pressure via a cation-selective membrane was theoretically and experimentally investigated. Unlike conventional electrokinetic ion concentration polarization, which uses electric current as a driving flux of cations through the membrane, advection caused by pressure is used as a transmembrane driving flux of cations to spontaneously and stably form an ion depletion zone in the present ion concentration polarization technique. The ion depletion zone produced in a simple experimental setup was used to filter electrolyte and preconcentrate ions and microparticles. Different from the general assumption of the negligible thickness of the electric double layer in microchannels, the low concentration in the ion depletion zone considerably increased the length of the electric double layer. This enhanced the formation of the ion depletion zone. The present results can improve the understanding on ion transport in the ion concentration polarization system and can be utilized to develop a portable water desalination device for rural/remote areas and for preconcentrating biomolecules.

Introduction

Recent advances in fabrication technologies have rendered micro-/nanofluidic devices reliable and suitable for microfluidic bioreactors on a single substrate, called lab-on-a-chip [1]. On-chip processing is cost-effective and offers high throughput and small sample volume requirement. Thus, it is suitable for diagnostic, biomedical, and pharmaceutical applications [2]. Owing to these advantages, many nanostructures and nanofluidic systems have been developed [3]. In particular, nanoscale fluid dynamics has led to the discoveries of new fundamentals. One of the new fundamentals is the overlapping of an electric double layer (EDL) in nanostructures. The overlapping of EDL allows the permselective transport of ions [4].

Under DC bias, permselective ion transportation through a cation-selective membrane from the anodic side to the cathodic side creates a strongly polarized concentration called ion concentration polarization (ICP). In this situation, the concentrations of ions decrease on the anodic side of the membrane (called ion depletion zone (IDZ)) and increase on the cathodic side (called ion enrichment zone). Many studies have been conducted not only on the complex nonequilibrium electrokinetic phenomena, such as deionization shock wave [5–8], over-limiting current [9,10], and vortex generation [11], but also on engineering applications, such as seawater desalination [12–14], preconcentration of samples [15,16], energy harvesting [17], and ionic transistors [18]. However, the over-limiting current and ununiform vortex generation induce instability, which limits practical engineering applications [11,19,20]. From an economic point of view, ICP is an energy-intensive process because of its continuous use of a high power source [21]. In addition, electrolysis-based generation of air bubbles and pH change of the solution [22] adversely affect the preconcentration of biological samples. Thus, a detailed understanding on the different approaches for generating ICP is highly needed.

Non-electriokinetic ICP was introduced, which IDZ was spontaneously formed by the capillary force of a hydrogel, a driving force for transmembrane ion transportation. In capillary-based ICP, the formation of the IDZ by capillary force took several hours and the applied flowrate was under ~0.15 nl/min [23,24]. In this study, the basic physics of ICP spontaneously formed within a few seconds under ~0.3 μl/min induced by the advection caused by pressure difference, the driving force for transmembrane ion transportation, was examined. This phenomenon is called spontaneous ICP (s-ICP). Fig. 1a illustrates the concept of s-ICP in a single microchannel. A hydrated Nafion composed of 4 nm spherical clusters connected by short channels of 1 nm diameter in theoretical model allowed the selective transport of cations and water molecules by advective flux [25,26]. This biased ionic transportation creates the IDZ on the flow-incoming side of the

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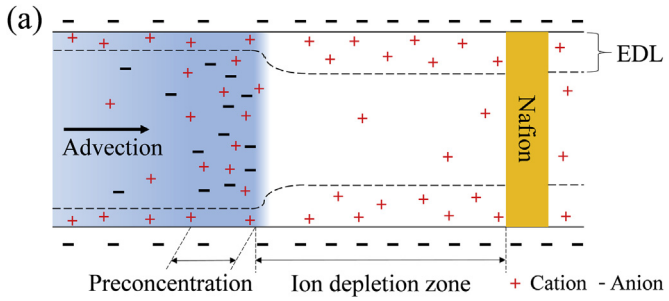

Fig. 1. (a) Schematic of the s-ICP formed in the microchannel.

Nafion to satisfy electro-neutrality, resulting in the induction of the s-ICP phenomenon. Thus, the IDZ can be generated more energy-efficient than the electrokinetic ICP. The power consumption between pressure-driven s-ICP and electrokinetic ICP is compared in Note S1. When the IDZ is formed near the Nafion, ions have difficult passing through the IDZ. Thus, they are preconcentrated, and seawater is desalinated simultaneously. As ions are filtered by the IDZ, their concentration in the IDZ region is extremely lower than that of the bulk solution. As a result, the magnitude of surface zeta-potential increases, and the EDL is elongated. Accordingly, different from the conventional assumption of the negligible length of EDL on the generation of the IDZ in microfluidics devices, the elongated length of EDL inside the IDZ should be considered in the design of microscale devices.

Material and methods

Design and fabrication of microfluidic device

A single microfluidic microchannel (70 μm high, 1000 μm wide) was designed. It was fabricated using the standard soft-lithography technology. Briefly, a SU-8 master (Microchem, MA, USA) was fabricated using standard photolithography processes. The surface was silanized using trichloro (3,3,3-trifluoropropyl)silane (Sigma-Aldrich) in a vacuum jar for 1 h. Polydimethysiloxane (PDMS, Sylgard 184; Dow Chemical, MI, USA) was then cast, cured at 95 °C, and then peeled off. After punching the PDMS, the PDMS device was prepared. After fabricating the Nafion membrane using a sheath method or surface patterned method [27], the microfluidic device was bonded to a glass substrate by oxygen plasma (Femto Science, Korea) treatment (Fig. 2 d).

For the wall effect experiment, we designed a single microchannel (100 μm high, 1500 μm wide) with/without an inclined additional wall (300 μm wide, 3 mm long, 5° angle of inclination). It was fabricated by using a milling process on acryl plate. The next fabrication procedures of the PDMS and microfluidic device were the same as above.

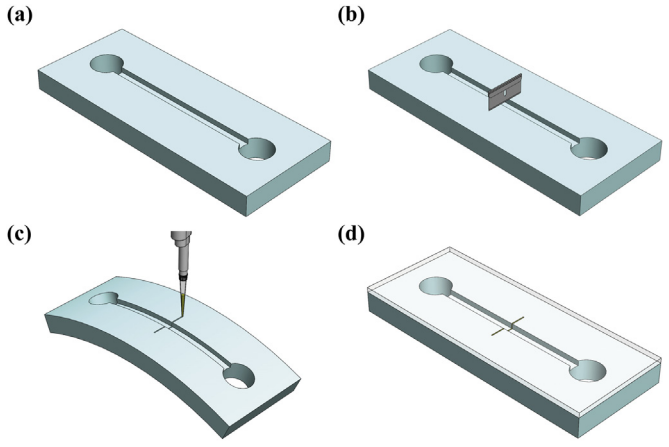

Fig. 2. Fabrication processes of the Nafion-inserted chip. (a) PDMS microchannel. (b) Cutting of the microchannel with a blazer. (c) Insertion of a Nafion solution into the sheath of the bending microchannel. (d) After evaoporating the solution at 80 °C in the oven, the PDMS microchannel and glass substrate were bonded by using oxygen plasma treatment.

Fabrication of Nafion membrane

To fabricate a Nafion membrane, Nafion resin solution (Sigma-Aldrich, USA) of 20 wt% concentration was used. Two types of Nafion membranes were fabricated in consideration of each experiment. The first is a channel-inserted Nafion fabricated by sheath method. After preparing a PDMS device (Fig. 2a), the microchannel was cut with a blazer as shown in Fig. 2b. Then, the Nafion solution was inserted into the sheath by pipetting (Fig. 2c). The solution was evaporated at 80 °C in the oven. The second one is a surface-patterned Nafion fabricated with the flow-based surface-patterning method [27].

Experimental setup

Fig. 3a shows a schematic of the experimental setup, in which two reservoirs were connected by a microchannel (80 μm high, 1000 μm wide). The cured Nafion did not cover the whole cross section of the microchannel in consideration of the smooth electrolyte flow (Fig. 3b). The Nafion solution was poured into the sheathed point of the microchannel and cured as a cation-selective nanoporous membrane (details of the fabrication of the device are given in Fig. 2). The electrolyte solution in the channel was driven by a syringe pump or hydrostatic pressure difference between the two reservoirs.

Visualization of formation of the IDZ

The dynamic movements of fluorescent particles and dye were observed with a fluorescent microscope (Zeiss Axiovert 200, Zeiss, Germany) attached with 2.5 × objective lens (numerical aperture (NA) of 0.075) and 20 × objective lens (NA = 0.4). A digital CCD camera (RETIGA-4000R, QImaging, Surrey, BC, Canada) was used to acquire images of fluorescent particles and dye solution at 33 fps (frame per second) and 1 fps, respectively.

Zeta-potential measurement using the Helmholtz-Smoluchowski equation and a micro-PIV (particle imaging velocimetry) technique

In the image tracing method, fluorescent particles are seeded in an electrolyte solution to quantitatively visualize the 2D velocity field of the electroosmotic flows [28]. The average electroosmotic flow velocity was measured to determine the zeta-potential of the microchannel under DC electric field. Since the height of the channel is much larger than the electric double layer thickness, the measured average electroosmotic flow velocity, $v_{average}$, is related to the zeta-potential of the microchannel by the Helmholtz-Smoluchowski equation [29,30]:

$$v_{average} = \frac{\varepsilon_r \varepsilon_0 \zeta}{\mu} E$$

where ε_r is the relative dielectric constant, ε_0 is the electrical permittivity of vacuum, ζ is the zeta-potential of the microchannel, μ is the solution viscosity, E is the applied electric field strength.

A KCl solution (0.01 mM, 0.1 mM, 1 mM) seeded with fluorescent particles of 1 μm in diameter filled the channel (1000 μm wide, 50 μm high, 2 cm long). Since the width of the PDMS microchannel is much larger than its height, the edge effect is minimized and the velocity profile can be assumed as uniform along the width direction. An electric field (15.2 V/cm) was applied through the channel with a power supply to generate electro-osmotic flow.

The velocity of the fluid under the electric field was measured using a micro-PIV (particle imaging velocimetry) technique. Unlike

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