Dynamic microscale flow patterning using electrical modulation of zeta potential

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Edited by David A. Weitz, Harvard University, Cambridge, MA, and approved March 7, 2019 (received for review January 2, 2019)

The ability to move fluids at the microscale is at the core of many scientific and technological advancements. Despite its importance, microscale flow control remains highly limited by the use of discrete channels and mechanical valves, and relies on fixed geometries. Here we present an alternative mechanism that leverages localized field-effect electroosmosis to create dynamic flow patterns, allowing fluid manipulation without the use of physical walls. We control a set of gate electrodes embedded in the floor of a fluidic chamber using an ac voltage in sync with an external electric field, creating nonuniform electroosmotic flow distributions. These give rise to a pressure field that drives the flow throughout the chamber. We demonstrate a range of unique flow patterns that can be achieved, including regions of recirculating flow surrounded by quiescent fluid and volumes of complete stagnation within a moving fluid. We also demonstrate the interaction of multiple gate electrodes with an externally generated flow field, allowing spatial modulation of streamlines in real time. Furthermore, we provide a characterization of the system in terms of time response and dielectric breakdown, as well as engineering guidelines for its robust design and operation. We believe that the ability to create tailored microscale flow using solid-state actuation will open the door to entirely new on-chip functionalities.

Manuscript EV2019-005696

Author contributions: F.P., V.B., G.V.K., and M.B. conceived the research; F.P. and V.B. performed the experiments; F.P. and V.B. analyzed data; F.P. and V.B. compared experimental data to the analytical model; F.P. fabricated the devices; F.P., V.B., G.V.K., and M.B. discussed the data; and F.P. and M.B. wrote the paper with input from all authors.

The authors declare no conflict of interest.

Significance

Traditional microfluidic devices make use of physical channels and mechanical actuators, in which geometries and functionalities are intimately related to one another, i.e., changing the flow field requires change at the mechanical level. In this work, we introduce a concept in which a microfluidic chamber with no preset structures or active mechanical components can be dynamically configured to produce desired flow fields.
range of zeta-potential values to be prescribed, thus enabling the flow field to be tuned in real time. We characterize the dielectric breakdown threshold for a range of dielectric materials and investigate the time response of our devices, providing engineering guidelines for the design of such systems. We then demonstrate the use of various electrode configurations for dynamically shaping microscale flows, creating dipoles, quadrupoles, and isolated flow regions, as well as the deformation of pressure-generated streamlines.

Concept of Flow Patterning Using ac-FEEO

As illustrated in Fig. 1, we use a Hele–Shaw chamber of thickness $h$ and length $L$ filled with an electrolyte in direct contact with a ground electrode and a driving electrode (Fig. 1A). The floor of the chamber contains an embedded electrode (gate electrode) with a characteristic dimension $r_0$, located at a distance $x_{el}$ from the ground electrode. The gate electrode is electrically insulated from the electrolyte by a dielectric layer of thickness $d$ and dielectric constant $\varepsilon_d$ (Fig. 1B). We actuate both the driving and the gate electrode with an ac potential at a frequency $\omega$ and amplitudes $\phi_i$, $V_i(t) = \phi_i(x_{el}) f(\omega)$, where the subscript $i$ indicates either the external ($ex$) or the gate electrode ($el$) potential. Assuming a thin electric double-layer regime (26, 27), and assuming that $1/\omega$ is longer than the charging time of the electric double layer, the EOF slip velocity can be described by the Helmholtz–Smoluchowski relation (28),

$$u_{\text{EOF}}(t) = -\frac{\varepsilon_l \zeta(t)}{\eta} E(t),$$

where $\varepsilon_l$ is the dielectric permittivity of the liquid, $\eta$ is its viscosity, $E(t) = V_{el}(t)/L$ is the electric field in the chamber, and $\zeta(t)$ is the zeta potential relative to the bulk. For $r_0 < L$, this bulk potential can be assumed to be uniform over the electrode and given by $V_{ch}(t) = (x_{el}/L)V_{el}(t)$. Regardless of the specific model used to describe the electric double layer [e.g., Guoy-Chapman, Stern, Bockris, etc. (ref. 28)], the zeta potential can be described by some function $G$ of the difference between the bulk and this will bulk potential, $\zeta(t) = G(V_{el}(t) - V_{ch}(t))$. Substituting the expression for $E(t)$ and $\zeta(t)$ into Eq. 1, the time-dependent EOF velocity can be expressed as

$$u_{\text{EOF}}(t) = -\frac{\varepsilon_l \zeta(t)}{\eta} E(t).$$

The time-averaged EOF velocity is obtaining by integrating Eq. 2 over one time period which, for the case where $f$ is a square-wave function (Fig. 1C), yields

$$u_{\text{EOF}}^\text{av} (\Delta \phi) = \frac{1}{2} \frac{\varepsilon_l}{\eta L} \phi_{el} |G(\Delta \phi) - G(-\Delta \phi)|,$$

where $\Delta \phi = \phi_{el} - (x_{el}/L)\phi_{el}$ is the difference between the gate potential and bulk potential amplitudes. Eq. 3 shows that $u_{\text{EOF}}^\text{av}$ is a symmetric function of $\Delta \phi$ regardless of the behavior of the function $G$. Fig. 1D shows an experimental measurement of the time-averaged EOF velocity as a function of the amplitude difference $\Delta \phi$, exhibiting the expected symmetry. We note that Eq. 3 is not valid outside the electrode region, where $\Delta \phi$ is not defined. In such regions the zeta potential can be assumed to be constant in time and related only to the native surface charge, and the time-averaged EOF therefore vanishes. In SI Appendix we provide a more generalized formulation of the system, accounting also for dc biases.
To achieve flow patterning using ac-FEEO, we use one or more spatially distributed gate electrodes. In the lubrication approximation \((L > r_0 \gg h)\), any discrete electrode (regardless of its specific shape) results in dipole-like circulation flow (SI Appendix, Fig. S6). Therefore, the choice of a disk-shaped electrode is a natural one as the depth-averaged velocity \(\vec{u}\) can be described by a simple analytical expression (10),

\[
\vec{u}(r, \theta, \Delta \phi) = \begin{cases} 
- \frac{e_1}{4\eta l} \phi_{\text{ext}} \frac{\zeta(\Delta \phi)}{2} \frac{(r_0)^2}{r} \left[ \cos \theta \theta + \sin \theta \theta \right] & r > r_0 \\
- \frac{e_1}{4\eta l} \phi_{\text{ext}} \frac{\zeta(\Delta \phi)}{2} \left[ \cos \theta - \sin \theta \theta \right] & r \leq r_0,
\end{cases}
\]

where \(r\) is the radial vector relative to the center of the disc and \(\theta\) is the angle between \(r\) and the electric field. In our experimental setup \(h = 15\mu m, r_0 \sim 100\mu m\), and \(L \sim 1\) cm, satisfying the lubrication conditions.

Fig. 1E and F shows the stream function (obtained from Eq. 4) for a negative and a positive \(\Delta \phi\), respectively, together with the experimentally measured vector flow field, for a 200-\(\mu m\) diameter disc-shaped gate electrode. This illustrates that the expected dipole flow can be obtained using the ac-FEEO mechanism and that the intensity and direction of the dipole can be indeed tuned by controlling \(\Delta \phi\). Movie S1 presents the visualization of this flow field during dynamic variation of \(\Delta \phi\).

System Design and Characterization

Central to the operation of the ac-FEEO is the ability to maintain capacitive charging over the gate electrode for a large number of charge and discharge cycles under high-driving electric fields (order of 100 V/cm). Clearly, a thick dielectric layer would be ideal to insulate the electrode against Faradaic currents, thus preventing bubble formation and pH changes resulting from electrolysis. However, the thickness of the dielectric layer should also be chosen to maximize the effect of the gate electrode on the induced zeta potential. A good approximation for the surface zeta potential as a function of \(\Delta \phi\) can be obtained from a capacitor model (12, 13, 15, 22, 28) accounting for the capacitance of the EDL \((C_{EDL} = \varepsilon_0 A / \lambda_{EDL})\) in series with the dielectric capacitance \((C_d = \varepsilon_d A / d)\),

\[
\zeta = \zeta_0 + \frac{C_d}{C_{EDL}} \Delta \phi = \zeta_0 + \frac{\lambda_{EDL}}{\varepsilon_0} \frac{\varepsilon_d}{d} \Delta \phi,
\]

where \(\zeta_0\) is the native zeta potential of the surface, and \(\lambda_{EDL}\) is the Debye length. Therefore, the most effective FEEO can be expected for a dielectric with the smallest possible thickness \(d\) and the largest possible permittivity \(\varepsilon_d\). The best dielectrics can be obtained in standard microfabrication processes that (29) exhibit dielectric breakdown values on the order of 1 V/nm. Given that the EOF driving voltages in our system are in the range of 100–400 V, a 500-nm layer of a high-quality dielectric is expected to withstand such potential differences.

Fig. 2 shows the measured dielectric breakdown field (breakdown voltage normalized by the dielectric thickness) for different dielectric coatings deposited with plasma-enhanced chemical vapor deposition (PECVD). See SI Appendix, Fig. S5 for details of the experimental setup and additional measurements for layer deposited with atomic layer deposition. The dielectric is inherently in an asymmetric configuration as it is in contact with a metal on one side and an electrolyte on the other. Because under an ac field the dielectric will be subjected to both positive and negative voltages, it is important to measure its breakdown for both cases. Pure SiN shows poor dielectric resistance, holding only up to ~0.1 V/nm.

\(\varepsilon_0\) performs significantly better for positive voltages, yet exhibits a clear asymmetry with a very low breakdown threshold for negative voltages (~0.1 V/nm). Doubling the thickness of this layer to 1 \(\mu m\) does not show an improvement. A two-layer composition of SiN on top of SiO\(_2\) provides a significant improvement in both breakdown voltage and symmetry; however, we note that flipping the order of the layers is not equivalent to a simple potential \((\Delta \phi > 0)\) but performing poorly for negative ones \((\Delta \phi < 0)\). Using a layer of SiO\(_2\) covered with a layer of SiN, boosts significantly the breakdown strength, up to ~1.3 V/nm for positive voltages; however, it shows poor repeatability (high error bars) and scarce symmetry. The best performance is obtained using SiON, withstanding up to ~1 V/nm for both positive and negative voltages; therefore we choose SiON as insulating dielectric layer. The error bars represent the 95% confidence interval of the mean (with at least 10 repetitions).

The principle of ac-FEEO relies on the ability to synchronize the net charge in the double layer with the phase of the driving electric field. The upper bound on the frequency of the driving electric field is therefore dictated by the EDL charging time, which is a function of both the charge relaxation time within the electrolyte and any resistor–capacitor timescales associated with the electronics. Higher frequencies lead to reduced EOF as shown by van der Wouden et al. (24). SI Appendix, Fig. S5 presents the response of our system to a sudden change of applied gate voltage for different gate-electrode dimensions (see experimental details in SI Appendix). The observed timescale for the electrodes used in this work \((200 \times 200 \mu m)\), defined as the time needed for the gate current to drop to 10% of its initial value, is ~5 ms. The maximum ac frequency that could be used while still benefiting from a fully charged EDL is ~200 Hz, and we use this value as a frequency upper bound when operating our system. We note that the response of the system to positive and negative potentials is highly symmetric and we attribute this to the properties of the SiON layer used. This is in contrast to the asymmetric behavior of the SiO\(_2\) layer reported by van der Wouden et al. (25).
Dynamic Flow Patterning

Flow-field patterns can be obtained by superposition of flows generated by a distributed set of gate electrodes. The ability to individually address each electrode and dynamically modify its associated zeta potential allows switching from one flow pattern to another in real time. Fig. 4 demonstrates this concept for a basic case of two 200-μm-diameter disc-shaped gate electrodes. At \( t_1 \) (Fig. 4D), the electrodes are assigned \( \Delta \phi \) values of +80 V (left electrode) and −80 V (right electrode), resulting in two dipoles with equal and opposite strengths, generating an EOF quadrupole. At \( t_2 \) (Fig. 4E), we change the \( \Delta \phi \) value on the left electrode to 0 V, thus effectively eliminating its influence. At \( t_3 \) (Fig. 4F), we match the \( \Delta \phi \) of the left electrode to the one on the right, resulting in a nested dipole configuration, consisting of recirculating flow around each electrode and a larger-scale recirculation between the two electrodes. The images presented here correspond to three time points of a continuous movie provided as Movie S2. These flow patterns can be well predicted by using Eq. 4 as shown in Fig. 4 A–C.

A particularly interesting configuration consists of two concentric electrodes, i.e., a disc-shaped electrode (inner) of radius \( R_{in} \) surrounded by an annulus-shaped one (outer) having an outer radius \( R_{out} \). For such a case, theory predicts that setting \( \Delta \phi_{in} = (|R_{out}/R_{in}|)^{1/2} \Delta \phi_{wall} \) would result in an internal recirculation while maintaining zero velocity outside the outer electrode (Fig. 5B). Fig. 5A demonstrates the implementation of this configuration, showing that such a bounded flow field is indeed feasible. Some “leakage” of the flow field is however observed in the experiments; this is due to the imperfection of the annulus shape that contains a slit serving as a path for the electrical connection to the inner disc. Furthermore, the existence of the electrical lines themselves adds an additional perturbation to the flow. These perturbations also exist in Fig. 4 but they are less visible there because the velocity field magnitude is substantial compared with such perturbations. Setting \( \Delta \phi_{in} = 0 \) V (Fig. 5 C and D) switches the flow field to a unique configuration in which a pressure jump at the outer edges of the annulus is compensated by an opposite pressure jump at the inner edges, leaving the inner region free of both slip velocity and pressure gradients. As a result, in the inner region the velocity field is uniformly zero, thus creating a finite stagnation volume within the flow.

Dynamic Streamlines Shaping

A set of gate electrodes can also serve as an effective way of shaping existing flow fields created by, for example, pressure-driven flow or dc EOF. Fig. 6 shows the effect of a 2 x 4 array of disc-shaped electrodes on uniform flow generated by a pressure gradient. At \( t_1 \), we activate the electrode array, assigning two values of \( \Delta \phi \), a positive and a negative one, in a checkerboard

A key advantage of using gate electrodes for controlling flows is the ability to switch from one flow pattern to another. The timescale associated with such switching is limited not only by the EDL charging process but also by the viscous response, \( \tau_v = h^2/\nu \), where \( \nu \) is the kinematic viscosity of the liquid. For a 15-μm-high chamber and an aqueous solution, as used in our experiments, \( \tau_v \sim 0.2 \) ms, significantly shorter than the electric response, thus not limiting the switching time. However, for \( h \sim 100 \mu m \), \( \tau_v \) is on the same order of magnitude of the EDL charging and could dominate the dynamic response of the system.

To completely characterize our system, Fig. 3 presents the measured time-averaged EO wall mobility (\( \mu_{EO,av} = \mu_{EO,E} \)) as a function of the applied amplitude difference \( \Delta \phi \). While, as expected, higher EO wall mobility is obtained using a low-pH buffer (10 mM acetic acid and 1 mM NaOH, pH 3.8), the use of physiological pH buffer (10 times diluted PBS) also provides significant EO mobility, indicating the potential use of ac-FEEO flow patterning for biochemical applications. Both curves show a linear dependence within this range of applied potentials, consistent with Eq. 5.

The analytical predictions and experimental visualization of flow streamlines generated by two 200-μm-diameter disc-shaped gate electrodes for different \( \Delta \phi \) combinations. (A and D) At \( t_1 \), we set the electrodes to opposite \( \Delta \phi \) values, generating opposing dipoles and resulting in a quadruple flow field. (B and E) At \( t_2 \), we switch the \( \Delta \phi \) of the left electrode to zero, effectively eliminating its effect on the flow. (C and F) At \( t_3 \), we match the \( \Delta \phi \) of both electrodes, resulting in a flow configuration having two regions of local recirculation nested within a larger recirculating flow. The driving amplitude is \( \phi_{wall} = 200 \) V, and because the electrodes are located at the center of the chamber \( \phi_{wall} \sim 100 \) V. A time-lapse movie showing the flow fields and the transition between them is provided in Movie S2.
4 array of gate electrodes. We establish a uniform flow in the and of the disc electrode to zero, and demonstrate the $\phi$ combina-
300 V in the electrode region. A time-lapse movie showing the flow
$\Delta t$ o a ll www.pnas.org/cgi/doi/10.1073/pnas.1821269116
2 − $|C|$ values, which were then used in
40 and Paratore et al. $\phi_0$; the streamline enters the central outlet undisturbed. At
$\mu_1$ Analytical predictions and experimental visualization of flow-field shaping using a 2
160 chamber by pressure gradient and at $t_1$, we set the electrode potentials to a checkerboard pattern with values of
$\phi_{\text{ex}} = 300$ V, and approximately $\phi_{\text{ex}} = 160$ V in the electrode region. A time-lapse movie showing the flow
fields and the transition between them is provided in Movie S4.

pattern. As expected from our theoretical predictions, the velocity
components perpendicular to the electric fields bend the incoming
flow, resulting in a sinusoidal streamline. At $t_2$, we set $\Delta \phi = 0$ to all
electrodes and the flow field relaxes back to its original state. At $t_3$, we invert the checkerboard pattern, resulting again in a sinusoidal
shape but with a phase shifted by 180°. In the simulation, the in-
coming velocity field was set to 40 $\mu$ m/s to match the experiments,
and the slip velocities of the electrodes were tuned to obtain the
desired flow pattern. Through the use of the independent mea-
surements of the EO wall mobility reported in Fig. 3, these slip
velocities were translated to $\Delta \phi$ values, which were then used in
the experiments with no additional fitting parameters. As shown
in Fig. 6, the agreement between theory and measurements is not
only qualitative but also quantitative, showing the streamlines being
deflected to the same extent.

Such flow shaping can also be integrated as part of more
elaborated devices. As an example, Fig. 7 shows the use of two
electrodes to deflect a central inlet streamline into one of the
three possible outlets. At $t_1$, the electrodes are set to create a
counter-clockwise flow, thus deflecting the incoming streamline
toward the lower outlet. At $t_2$, both the electrodes are set to have
a $\Delta \phi = 0$; the streamline enters the central outlet undisturbed. At $t_3$, we set the electrodes to create a clockwise velocity pattern,
thus routing the streamline to the upper outlet.

Conclusion and Outlook
We presented the use of ac-FEEO as a mechanism to create
dynamic flow patterns in a Hele–Shaw configuration. We showed
that the basic flow pattern of an EOF dipole predicted by the
theory can be experimentally reproduced by this mechanism,
with dipole strength set by the voltage amplitude difference be-
tween the electrode and the bulk. In contrast to zeta-potential
modification using chemical patterning (11, 30), electric control
allows setting the values of EOF within a continuous range of
positive and negative values, and to rapidly switch between them.
The timescale for such switching depends on the EDL charging
time and the viscous time, which in our system is on the order of
5 ms. We investigated several electrode configurations and
demonstrated a variety of flow patterns that can be realized. To
the best of our knowledge, some of these patterns, such as the
localized recirculation and the stagnation volume, have not been
demonstrated by other means. Furthermore, we showed that ac-
FEEO can interact with existing flow fields and dynamically tune
their streamlines.

Precise microscale flow control may be useful in several appli-
cations. For example, whereas large particles can be manipulated
in microsystems by other mechanisms such as dielectrophoresis or
optical tweezers, controlled transport of small molecules remains
challenging. The technique presented here could be particularly
useful to bridge this gap, and allow control of the mass transport
of small chemicals and biomolecules such as proteins, DNA,
peptides, etc. Because this technique drives the fluid itself and not
the single particles, it may be useful for heat-transfer management
in microdevices, and the pressure field formed may be leveraged

Fig. 5. Analytical predictions and experimental visualization of flow streamlines generated by a 200-$\mu$m-diameter disc-shaped electrode sur-
rounded by a 400-$\mu$m-outer-diameter annulus, for different $\Delta \phi$ combina-
tions. (A and C) At $t_1$, the potential amplitude ratio of the two gate
electrodes is set such that outside the annulus the two EOF dipoles a-
cel cancel each other (inner $\Delta \phi = -120$ V and outer $\Delta \phi = 40$ V), resulting in an
isolated region of recirculating flow surrounded by a quiescent liquid. (B and
D) At $t_2$, we set the $\Delta \phi$ of the disc electrode to zero, and demonstrate the
opposite case of a finite stagnation volume surrounded by flow. The am-
pitude in the channel for both case is $\phi_{\text{ex}} = 300$ V, and approximately
$\phi_{\text{ex}} = 160$ V in the electrode region. A time-lapse movie showing the flow
fields and the transition between them is provided in Movie S4.

Fig. 6. Analytical predictions and experimental visualization of flow-field shaping using a 2 × 4 array of gate electrodes. We establish a uniform flow in the
chamber by pressure gradient and at $t_1$, we set the electrode potentials to a checkerboard pattern with values of $\Delta \phi = 150$ V and $\Delta \phi = -150$ V and an external
amplitude to $\phi_{\text{ex}} = 300$ V. The multiple dipoles superpose with the uniform flow, resulting in sinusoidal shaping of the central streamline. When the gate
potentials are set to zero ($t_2$) the flow relaxes to its original shape. At $t_3$, we flip the original checkerboard pattern, obtaining a sinusoidal shape with a shifted
phase. The gate potential values used in the experiments are derived from theoretical predictions together with the calibration curve of Fig. 3, with no
additional fitting parameters. A time-lapse movie showing the evolution of the streamline formation is provided in Movie S5.
to actuate deformable surfaces, such as free surfaces or elastic actuators. In this work, we used few individual electrodes providing access to only a limited set of flow patterns. An ideal flow control system would allow any desired flow pattern on a large scale. One could envision such a system constructed from a large number of individually addressed electrodes, likely in array format.

Materials and Methods

We characterized the EOF velocity as a function of the applied amplitude difference \( \Delta \phi \). We used a 100-µm-wide, 15-µm-high, and 1-cm-long straight channel containing an array of gate electrodes distributed over the entire length of the channel and composed of 100 × 100-µm² units, spaced 10 µm edge to edge. These gate electrodes are set to give an equal \( \Delta \phi \) along the channel to ensure a homogeneous EOF slip velocity throughout the channel. We measured the depth-averaged velocity by particle image velocimetry (PIV) analysis, using 0.8-µm carboxyl fluorescent particles (Spherotech Inc.) as flow tracer and PIVlab for the image analysis. Assuming a pure Couette-type flow, we estimate the slip velocity to be twice the measured depth-averaged velocity, and use this to calculate the velocity (shown in Fig. 1D) and the EO wall mobility (shown in Fig. 3) via the Helmholtz–Smoluchowski relation.

Additional information on visualization conditions, image analysis, and device fabrication is provided in SI Appendix.

ACKNOWLEDGMENTS. We thank S. Dehe for introducing us to the work of van der Wouden et al. (24, 25) on ac-based actuation; S. Dehe and B. Rofman for further useful discussions on this topic; E. Boyko, D. Taylor, X. van Kooten, and Y. Temiz for useful discussions; D. Dávila Pineda and U. Drechsler for continuous help and support on the microfabrication; and S. Rubin and K. Gommed for collaboration on early attempts using dc actuation. F.P., V.B., and G.V.K. acknowledge P. Renaud, E. Delamarre, and W. Reiss for their continuous support. F.P. was supported by the Initial Training Network, Virtual Vials, funded by the FP7 Marie Curie Actions of the European Commission (FP7:PEOPLE-2013-ITN-607322). This project has received funding from the European Research Council under the European Union’s Horizon 2020 Research and Innovation Programme, Grant agreement 678734 (MetamorphChip).

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Fig. 7. Demonstration of flow-field shaping integrated in a microfluidic device. The gate electrodes can be used to shape an existing flow field and direct an incoming streamline into one of the three outlets. At \( t_1 \), we impose \( \Delta \phi = -310 \text{V} \) and \( \Delta \phi = 350 \text{V} \) to the top and bottom electrode, respectively, to induce a counterclockwise velocity field, thus pushing the streamline to the bottom outlet. At \( t_2 \), we set \( \Delta \phi = 0 \) to both electrodes and the flow returns to its native state, with the central streamline continuing to the central outlet undisturbed. At \( t_3 \), we flip the initial \( \Delta \phi \) assignment to the electrodes directing the streamline to the upper outlet. A time-lapse movie showing this switching process is provided in Movie S6.