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DEVELOPMENT OF GATE-CONTROLLED DC ELECTROKINETIC MICROPUMPS

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Prachya Mruetusatorn
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ABSTRACT

Lab-on-chip (LOC) devices have received considerable attention in research and development for automated, high-throughput biological and chemical analysis. While much progress has been accomplished; however, fluid flow control still needs improvement and reminds one of the significant challenges for the future practical LOC devices. This thesis explores the application of electroosmosis (EO) technique and field effect flow control (FEFC) technology for micropumps, an important microfluidic component of LOC systems.

In this work, electroosmosis method was employed to electro-kinetically move the working fluid under a longitudinal electric field, and the FEFC technique was also utilized to manipulate the Electroosmotic Flow (EOF) through applying a normal electric field to influence the surface charge at the fluid-microchannel wall interface for an independent control over the EOF. Major accomplishments in this thesis are, study on channel geometry effect with no gate control component, and a single microchannel with gate control component.

A number of micropumps with different channel geometries were fabricated using soft lithography technique. PDMS prepolymer served as a top wall and both side walls of the microchannel, with a glass slide as the bottom (in the case of gate control, Indium Tin Oxide glass slides were used). On the gate control region, through adjusting the secondary electric field over the gate, FEFC can locally manipulate EOF. It helps produce a range of flow rates, enhance flow rates, and control flow direction. Moreover, micropumps were interfaced with another microchannel section for sample delivery.
To improve the microfluidic device, electro-fluid flow models were developed to describe and predict electric field distribution, velocity field distribution, flow direction, and FEFC phenomena using Finite Element Analysis tool (FEMLAB). The simulation results agreed well with experimental results.
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# Abbreviation and Acronyms

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<tr>
<td>AC</td>
<td>Alternating Current</td>
</tr>
<tr>
<td>ACEO</td>
<td>AC Electro-Osmosis</td>
</tr>
<tr>
<td>AP-ACEO</td>
<td>Asymmetric Polarization-ACEO</td>
</tr>
<tr>
<td>DC</td>
<td>Direct Current</td>
</tr>
<tr>
<td>DI</td>
<td>Deionized</td>
</tr>
<tr>
<td>EK</td>
<td>Electro-Kinetic</td>
</tr>
<tr>
<td>EOF</td>
<td>Electro-Osmotic Flow</td>
</tr>
<tr>
<td>FEA</td>
<td>Finite Element Analysis</td>
</tr>
<tr>
<td>FEFC</td>
<td>Field Effect Flow Control</td>
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<tr>
<td>FIA</td>
<td>Flow Injection Analysis</td>
</tr>
<tr>
<td>ITO</td>
<td>Indium Tin Oxide</td>
</tr>
<tr>
<td>LOC</td>
<td>Lab-On-a-Chip</td>
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<tr>
<td>MEMS</td>
<td>Micro-Electro-Mechanical-Systems</td>
</tr>
<tr>
<td>PDMS</td>
<td>Poly-Di-Methyl-Siloxane</td>
</tr>
<tr>
<td>PMMA</td>
<td>Poly-Methyl-Meth-Acrylate</td>
</tr>
<tr>
<td>μLC</td>
<td>Micro-Liquid-Chromatography</td>
</tr>
<tr>
<td>μTAS</td>
<td>Micro-Total-Analysis-Systems</td>
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1. INTRODUCTION

1.1 Introduction

Lab-on-Chip (LOC) devices miniaturize laboratory functions onto a single chip of typically millimeters to a few square centimeters in size, and by doing so, achieve speed, efficiency, reduced sample consumption, and multiplexing detection for the miniaturized total analysis systems [1]. Length scaling using scaling laws plays an important role to shorten fluid transport times and lower sample diffusion in the range of micro/nano meters [2].

Liquid chromatography, for instance, is a method to separate a liquid mobile phase mixture through a column containing a stationary phase. The separation time of the mixture is determined by the extent of retention of each solution in the mobile phase with stationary phase [3]. The speed of the system is crucial and dependent on the surface area of the stationary phase. In an LOC device with down-sizing to 1/10 of the original column width, the increased surface to volume ratio is done to reduce all time-dependent factors to 1/100 of the original values, yielding higher efficiency [1]. Moreover, total analysis systems are miniaturized due to the surface to volume ratio increment, thereby reducing sample or reagent consumption for low sample loss [4]. Therefore, the sample preparation and analysis with high multiplicity of the detection process can be integrated on the same LOC device and high-throughput performance of routine chemistry process can be accomplished [5, 6].
At present, LOC device technology has shown high potential for automated sample transfers without enormous amount of equipment space, energy and cost, and labor [7]. For applications in the fields of medicine, chemistry, and environmental science essentially, LOC devices require three tenets, which are to be cheap, durable, and compact [8].

1.2 Thesis Objectives

The main objective of this work is to design and develop micropumps, one of the crucial microfluidic components, for driving the minute amount of fluids using electrokinetic technique, and fluid flow control technology is studied and utilized to manipulate EOF, which is one of the biggest challenges to the extensive development of LOC devices [8].

Briefly, micropumps have a variety of operating principles, which can be divided in two categories: Mechanical and Non-mechanical pumps [9, 10]. The mechanical micropumps are microscale versions of the traditional macroscale pumps with moving parts, such as mechanical valves, oscillation membranes, or turbines, connected to a chamber where an oscillating volume flow is generated to deliver a constant fluid volume [10]. The mechanical micropumps, however, have been shown complications like limited material options, integration restrictions, and reliability issues when piezoelectric or peristaltic components have been employed [11, 12, 13, 14].

The second category is nonmechanical or dynamic micropumps equipped with no moving parts and operated by adding momentum to the fluid to generate pumping effect. Nonmechanical pumps mainly discover its advantages in the microscale [10]. Non-
mechanical micropumping techniques, for instance, include magnetohydrodynamic (MHD) [15], thermal bubble generation [16], transpiration [17], surface tension [18], centrifugal systems [19], and electroosmotic flow (EOF) [20, 21, 22, 23, 24]. At present, nonmechanical micropumps are more promising than the mechanical ones with respect to portability, accurate control of minute amount of samples and reagents, and low power requirement [25].

In the development of micropumps, polymer materials are strongly favored for disposable and analytical LOC devices [26]. The mostly common used polymers in microfluidics include polycarbonate [27], polydimethylsiloxane (PDMS) [28, 29, 30], polyethylene terephthalate [31], poly methyl methacrylate (PMMA) [32], polystyrene [33], and SU-8 photoresist [34].

In this work, the PDMS prepolymer was selected since it has a number of advantages over other polymer materials mentioned above, such as easy fabrication with low cost. Further details on PDMS prepolymer were presented in the later chapter. Moreover, electroosmotic was chosen as our pumping mechanism. Electroosmotic (EO) micropumps are capable of generating high pressure or high flow rate, are easy to fabricate and integrate with other microfluidic components with low cost, and offer accurate control of minute amount of fluids, besides being reliable,. The key dictating parameters of the performance of EO micropumps are (1) the magnitude of the applied electric field, (2) the cross-sectional dimensions of the channel structure where flow is generated, (3) the surface charge density of the solid surface wall in contact with the working liquid and (4) the viscosity and pH of the working fluid [35].
Field effect flow control (FEFC) is proposed as an adjustable, integrated technique to locally manipulate EOF within a microchannel [36]. Figure 1-1 describes the application of FEFC at the bottom wall of a microchannel. An EO pump is implemented by applying a longitudinal electric field over the microchannel to generate EOF, driving fluid from the anodic/inlet reservoir (+) to the cathodic/outlet reservoir (-). A second or transverse electric field is applied over the gate placed at the bottom microchannel wall to modulate the surface charge at the fluid-bottom microchannel wall interface to locally control EOF. A voltage, $V_G$, applied at the gate, generates the transverse electric field, which allows fluid flow to be controllable. The mechanism of electrokinetic (EK) micropumps with a gate control component will be elaborated in a later chapter.
1.3 Thesis Organization

In this thesis, the EK micropump with the FEFC application is presented. The devices are fabricated on two types of substrate, glass slide and ITO glass slide, using standard photolithography and soft lithography technique. The development of electro-fluid models for explanation and prediction of the flow behavior is illustrated using FEMLAB, a finite element analysis (FEA) tool. The model also helps to further investigate the effects of microchannel geometry and gate geometry designs for the future FEFC microfluidic network.

This thesis consists of seven chapters. Chapter 1 contains a brief introduction to the field of high-throughput LOC devices and FEFC application as well as a brief review of micropumps. Chapter 2 explains the mechanisms of the electric double layer and electroosmosis, and FEFC mechanism with a succinct review of previous FEFC work in microfluidic systems. General modeling ideas based on FEMLAB are also included in this chapter. Chapter 3 explores detailed microchannel fabrication procedure, flow visualization investigation, and study on fluid variables and electrochemical effects to EO pump performance along with supporting experimental results. Chapter 4 is focused on channel geometry factors over EO pump performance and microchannels for interfacing application. Chapter 5 is aimed at the EK micropump with gate control application. The electro-fluid models using FEMLAB are developed in this chapter to demonstrate and predict flow behavior and effect. Concluding remarks and possible future work are discussed in Chapter 6 and 7 respectively.
2. BACKGROUND

2.1 Introduction

In general, a potential difference develops between two phases when they are placed in contact. In the case of microfluidics, one of the two phases is often an ionic liquid, such as water, and the other phase is a solid. At the liquid-solid interface, the electric charges tend to distribute in a non-uniform way, thus causing a separation of electric charges between the two phases, referred to as electric double layer (EDL).

Fluid pumping occurs when the solid phase remains stationary and the liquid phase moves under an applied electric field. The counter-ions in the liquid phase, balancing the surface charge at the solid wall, move under the applied electric field thus dragging the bulk liquid with them due to liquid viscosity. This phenomenon is referred to as electroosmotic flow (EOF).

The ion concentration at the fluid-solid wall interface is indicated by a term called zeta potential, which also determines the electroosmotic flow rate of the bulk fluid. Field-Effect Flow Control (FEFC) could be applied to manipulate EOF through application of a transverse electric field at the solid wall. At the interface, the charges in the fluid phase redistribute themselves in accordance to the transverse electric field to increase or decrease the zeta potential. The transverse electric field determines the magnitude and sign of the zeta potential due to changing the ion concentration at the fluid-solid wall interface. Therefore, EOF can be manipulated through the transverse electric field, independent of the longitudinal electric field.
2.2 Formation of Electric Double Layer

When two phases are placed in contact, the charged carriers in each phase are attracted to the opposite charge carriers in the other phase. Figure 2-1 demonstrates an excess of charges of one sign near to or on the solid surface and the balancing charges distributed throughout the adjoining fluid surface. Moreover, the electric field from the separation of charge may also cause polarization effects in neighboring molecules. All of these effects tend to produce an electrical potential difference between the interior of the two phases, called the ‘inner’ or Galvanic potential difference, $\Delta \phi$ [37]. The separation of charge at the fluid-solid interface is called an electrical double (EDL) layer because it consists of two regions of opposite charges.

The simplest model of such a system is where both layers of charges are fixed in parallel planes to form a molecular condenser or parallel plate capacitor. One layer of charges is spread out uniformly over a plane surface of the wall while the charges in the electrolyte solution are distributed with non-uniformity. The wall surface has an inherent surface potential, $\psi_0$, while the balancing ions are regarded as point charges immersed in a dielectric medium.
The thickness of the electrical double layer is customarily defined as the distance \(1/\kappa\) from the fluid-solid wall interface, which is typically on the order of nanometers in thickness. The distance \(1/\kappa\) depends on the ion concentration of the buffer solution as in equation 2.1. From the double layer thickness equation:

\[
\kappa^{-1} = \left( \frac{\varepsilon k T}{2 \varepsilon^2 z^2 n^0} \right)^{1/2}
\]

where \(\varepsilon\) is the dielectric permittivity of the solid channel, \(n^0\) is the counter-ion concentration of bulk solution, and \(z\) is the ion valance. \(\kappa^{-1}\) is obviously seen to decrease inversely as the square root of the counter-ion concentration which is directly proportional to the conductivity of the working fluids. Within the electric double layer, the counter-ion charge distribution decays exponentially from the wall potential, \(\psi_0\), to the potential in the bulk fluid defined to be zero due to the equal concentration of co-ions and counter ions \((\psi(y \to \infty) = 0)\).

2.3 Electroosmotic Flow (EOF)

Electroosmosis is the motion of bulk fluid with respect to a stationary, charged solid surface. The application of an electric field, \(E_x\), parallel to the microchannel wall produces electroosmotic flow in a solution. The direction of the electroosmotic velocity, \(u_{eo}\), is parallel to the wall and its magnitude, which Smoluchowski first expressed, can be written as follows [38]:

\[
u_{eo} = -\frac{\varepsilon \zeta}{\eta} E_x
\]
where \( \varepsilon \) is the fluid permittivity and \( \eta \) is the fluid viscosity. The zeta potential, \( \zeta \), is the average potential at the plane of shear, which is the no-slip boundary at the microchannel wall, due to strongly adsorbed counter-ions. The zeta potential is determined from the wall surface potential, \( \psi_0 \) (an exponential relationship in the Debye-Hückle approximation). Under the influence of \( E_s \), the counter-ions not adsorbed to the fluid-wall interface, because of high concentration in the electric double layer, generate a body force on the liquid phase due to ion drag. The bulk fluid is driven in the direction of \( E_s \), depending on the valency and concentration of the ions. A schematic representation of the ion distribution at the wall surface is shown in Figure 2-2. The surface functional groups of the wall material determine the inherent surface potential, \( \psi_0 \). The electrochemical reaction takes place at the fluid-solid interface, due to Galvanic potential difference between the phases, thus generating these excess ions. The surface functional groups normally define the negative surface charge, \( \psi_0 \) at the solid wall.

![Figure 2-2. Schematic of the electric double layer. The co-ions in the diffuse layer are not shown [36].](image)

For fused silica capillaries, for instance, the surface charge is due to the dissociation of the free hydroxyl groups of the silica surface from the silanol groups (SiO⁻) [39]:

\[
\text{SiOH} \leftrightarrow \text{SiO}^- + \text{H}^+
\]

The dissociation of the silanol groups is the sole ionization reaction of the surface for a resulting negative surface charge as shown in Figure 2-2. Since \( \psi_0 \) determines the magnitude of the zeta potential, modification of the wall chemistry controls EOF. Common modification to the surface charge includes buffer additives [40], ionic strength [41], buffer pH [42], organic solvents [43], or wall coatings [44]. To compensate for the negative wall charge, positive counter-ions are adsorbed to the wall interface forming the immobilized Stern layer or inner Helmholtz plane. The potential decays exponentially between the Stern layer, where the ions are tightly bound due to electrical forces, and the diffuse layer, where the electrical forces compete with diffusive forces [36].

The outer Helmholtz plane marks the plane of counter-ions, which are loosely bound. The zeta potential lies at the outer Helmholtz plane, or plane of shear, where the counter-ions are free to move under the \( E_x \) force. The zeta potential is shown in Figure 2-2 as \( \zeta_0 \). The counter-ions drag the neutral molecules in the direction of the electric field to achieve the bulk flow. On the contrary, the flow is in the opposite direction if the counter-ions are opposite in polarity due to a positive wall potential.

From a fluid mechanics standpoint, the outer Helmholtz plane indicates the plane of shear. The adsorbed counter-ions are strongly bound to the microchannel surface because of the high electrical force. The nature of the adsorbed counter-ions marks the
no-slip boundary condition. The flow in the diffuse layer is mainly due to the electrokinetic forces from the longitudinal electric field, $E_x$, and viscous forces on the counter-ions. The velocity field exhibits a gradient over a distance of tens of nanometers from the microchannel wall until it reaches the plane of slip with a maximum velocity given by the Smoluchowski equation in equation 2.2.

The flow outside EDL exhibits a plug-like flow for uniform zeta potentials and the absence of pressure gradients. The basic equation describing EOF outside EDL of an incompressible Newtonian fluid is derived from the classic Navier-Stokes equation [45]:

$$\rho \frac{D\vec{u}}{Dt} = \nabla p + \eta \nabla^2 \vec{u}$$  \hspace{1cm} (2.3)

where $\rho$ is the fluid density, $\vec{u}$ is the velocity field, $F$ is the body force, $p$ is the pressure and $\eta$ is the fluid viscosity. Assuming that gravity forces are negligible, the body force in EOF is purely an electrokinetic body force due to the electrical force exerted on the ions [46]:

$$F = \rho_e \vec{E}$$  \hspace{1cm} (2.4)

where $\rho_e$ is the spatial charge density of co-ions and counter-ions and $\vec{E}$ is the electric field in the microchannel. Since typical EOF in microfluidic applications has low Reynolds number (Re) smaller than unity, the flow is a balance of the electrokinetic body force, pressure gradients, and viscous forces as demonstrated in equation 2.3. For low Reynolds numbers (Re $\ll 1$), the left hand side is zero. Thus, for electrosmosis, the flow is a balance of the electrokinetic forces, pressure gradients, and viscous forces.
In typical electroosmotic pumping applications, the pressure gradients are eliminated, \((\nabla p = 0)\) and the applied electric field is parallel to the microchannel wall, \((\vec{E} = E_x)\), so that equation can be written:

\[
0 = \rho_e E_x + \eta \nabla^2 \vec{u} \tag{2.5}
\]

The charge distribution, \(\rho_e\), at EDL is governed by the Poisson's equation:

\[
\nabla^2 \psi = -\frac{\rho_e}{\varepsilon} \tag{2.6}
\]

where \(\psi_0\) is the inherent potential distribution from EDL and assumed to be without tangential gradients. Typically, the longitudinal electric field is on the order of hundreds of volts per centimeter and the inherent potential field at the microchannel wall is on the order of tens of thousands of volts per centimeter.

Since the effect of the inherent potential field is negligible outside EDL, \((\psi(y \to \infty) = 0)\), the longitudinal electric field is without gradients normal to the microchannel wall \((x\)-direction). Likewise, the effect of the longitudinal electric field on the inherent potential distribution is typically negligible due to its low field strength and the high diffusivities of the counter-ions. Substituting for \(\rho_e\), into equation 2.6 yields:

\[
\eta \nabla^2 \vec{u} = \varepsilon \nabla^2 \psi E_x \tag{2.7}
\]

In equation 2.7, EOF results from the competition of electrokinetic and viscous forces. For flow parallel to the microchannel wall in the \(x\)-direction, two conditions are applied. The first condition is that the flow is divergence-free. Since there is no flow in the normal direction, the flow along the microchannel is without a tangential gradient. Second, due
to the negligible interaction of the longitudinal electric field, the inherent potential
distribution, \( \psi_0 \), is assumed to be without a tangential gradient. With these two
conditions, the Navier-Stokes equation is reduced. The boundary conditions at the Stern
layer, \( (u = 0 \text{ and } \psi = \zeta_0 \text{ at } y = 0) \) and in the bulk fluid \( (\partial u / \partial y = 0 \text{ and } \partial \psi / \partial y = 0 \text{ at } y = \infty) \) are applied. Assuming that both \( \eta \) and \( \varepsilon \) keep their bulk value everywhere in the
microchannel, the velocity is proportional to the zeta potential, the applied electric field,
and the inherent potential distribution in the microchannel. The adopted sign convention
is that when \( \zeta_0 \) is negative, the flow is towards the cathode. Then, the Debye-Hückle
approximation is applied to obtain the inherent potential distribution:

\[
\psi = \psi_0 \exp(-\kappa y')
\]  \hspace{2cm} (2.8)

The origin for the Navier-Stokes equation is at the plane of shear (\( y = 0 \)), but the
origin for the inherent potential distribution is at the microchannel wall \((y' = 0)\). Without loss, the potential distribution can be rewritten in terms of the Navier-Stokes
coordinates as:

\[
\psi = \zeta_0 \exp(-\kappa y')
\]  \hspace{2cm} (2.9)

The velocity is uniform at a distance far outside EDL proportional to the value of \( 1/\kappa \).
The values for \( 1/\kappa \) correspond to the low buffer solution concentration, like 1 mM, which
is typical in microfluidics [36]. The velocity is uniform at the same distance for each
value of \( \zeta_0 \) but the maximum velocity varies according to the zeta potential.
Smoluchowski equation is applied when the velocity profile becomes uniform
(equation 2.2) at the plane of slip. The plane of slip marks the distance from the plane of
shear where the flow velocity rises from a value of zero at the wall to a maximum value, $u_{eo}$.

2.4 Field-Effect Flow Control (FEFC)

2.4.1 FEFC Mechanism

FEFC technique is a method to manipulate EOF by modulating zeta potential through a transverse electric field, $E_{FEFC}$, on the microchannel wall. In the previous research, for instance, electrodes surrounded fused silica capillaries to generate the transverse electric field through the capillary wall [47]. Similar to FEFC in capillaries, gate electrodes are embedded under microchannel walls to generate the transverse electric field [48].

The transverse electric field from the gate electrode directly affects the zeta potential in the microfluidic devices. Figure 2-3 shows the change in the surface charge, $\psi_{FEFC}$, due to the FEFC electric field. The induced surface charges are due to

![Figure 2-3. Field-effect flow control in the electric double layer.](image)

the capacitive effect of the gate electrode. Compared to the unmodified EOF in non-conductive solid wall, a larger concentration of counter-ions reside in both the Stern layer and the diffuse layer because of $\psi_{FEFC} > \psi_0$. Increased counter-ion concentration affects the zeta potential magnitude, $\zeta_{FEFC}$, for an increased EOF pumping following the Smoluchowski equation in equation 2.2.

In the case of a positive transverse electric field, the induced surface charges are lower than the inherent surface charge $\psi_{FEFC} < \psi_0$ and fewer counter-ions reside in EDL, thus leading to EOF reduction. If the positive transverse electric field is sufficiently large to drive away the counter-ions, then it can establish negligible EOF, or even a reverse EOF.

To understand the effect of FEFC on EOF, previous researchers have proposed a double capacitor model that considers the change in the zeta potential to be electrostatic in nature [49]. As seen in Figure 2-4, the diffuse layer capacitance, $C_D$, the Stern layer capacitance, $C_{SL}$, and the microchannel wall capacitance, or gate capacitance in this case, $C_W$ are in series. The zeta potential change, $\Delta \zeta$, can be determined from the equivalent capacitor circuit where

$$\zeta_{FEFC} = \zeta_0 + \Delta \zeta$$

(2.10)
As in a voltage divider, $\Delta \zeta$ measures from the voltage drop across $C_D$ due to an applied gate voltage, $V_G$:

$$\Delta \zeta = \frac{I}{C_D}$$  \hspace{1cm} (2.11)

The theoretical current, $I$, through the circuit is:

$$I = \frac{(V_G - V_i)}{Z_T}$$  \hspace{1cm} (2.12)

where $V_i$ is the voltage potential above the gate region due to the longitudinal electric field for EOF and $Z_T$ is the total impedance of the three capacitors.

Since the thickness of microchannel or gate wall in this case is much larger than the Stern layer and diffuse layer, the total impedance can be approximated as:
Substituting equations 2.11 to 2.12 into 2.13, the zeta potential change for a given gate voltage is:

$$
\zeta_{\text{FEFC}} = \zeta_0 + \frac{C_w}{C_D} (V_G - V_i)
$$

(2.14)

The degree of control over the zeta potential can be determined by ratio of wall and diffuse layer capacitance, $C_w/C_D$, and magnitude and direction of the transverse electric field.

2.4.2 Design Factors for FEFC

There are several ways to enhance the degree of control over EOF. First, increasing the double capacitor ratio ($C_w/C_D$) can be a way to increase zeta potential control at a low transverse electric field. The capacitance of the diffuse layer is:

$$
C_D = 2.285 \varepsilon c^{1/2} \cosh(19.4z\psi_0)
$$

Also, changes to the buffer solution or wall surface chemistry can decrease $C_D$ for an improved FEFC performance. Reducing the buffer ion concentration (c) in the equation of $C_D$ can shrink the double layer thickness for an increased $C_D$ [49]. Lowering the buffer pH drives the dissociation reaction of surface functional groups for a decreased $\psi_0$ [39]. Similarly, surface coatings can be employed to deactivate the surface functional groups [50]. The thickness of the microchannel wall or gate wall, $d$, can be reduced to increase
the wall capacitance in F/m\textsuperscript{2} [47]:

\[ C_w = \frac{\varepsilon}{d} \]  

Equation (2.15)

Furthermore, the wall or gate material with a high dielectric constant yields an increased wall capacitance for an improved FEFC. In the previous work, controlling these factors has enabled an improved FEFC and validated the double capacitor model. However, recent criticism of the double capacitor model is its inadequate encompassing of all reported experimental results [51]

2.4.3 Review of Microchannel FEFC

Polson \textit{et al.}

The first demonstration of FEFC in Figure 2-5 was performed in a microfabricated glass substrate where a channel (30 \( \mu \)m wide and 10 \( \mu \)m deep) was paralleled with two gate electrodes positioned 50 \( \mu \)m away from the microchannel [24]. The applied gate voltage of \( \leq 120 \) V from the power supply was smaller than the kilovolts typically applied in capillary FEFC work. The speed of fluorescent dye was used to

Figure 2-5. The first demonstration of FEFC on glass substrate by Polson [24].
measure the EOF velocity. The improved control is attributed to the thin channel wall thickness, possible with microfabrication.

Schasfoort et al.

The second FEFC LOC device was a silicon-glass microdevice that used silicon nitride as microchannel walls, shown in Figure 2-6 [48]. Microchannel dimensions were 400/100 µm wide and 25 µm high. A silicon wafer was etched in KOH to form cavities to serve as a mold. Chemical vapor deposition was employed to grow silicon nitride to a 390 nm thickness on the mold walls. This deposition step formed the three walls of the microchannel. Anodic bonding of a glass substrate to the silicon nitride formed the bottom wall of the microchannel, where its transparency allowed for flow visualization. The backside of the silicon wafer was patterned and etched all the way down to the silicon nitride layer. The un-etched silicon structures served as the gate electrodes and as the fluid reservoirs. The device had high control over EOF due to the ultra-thin silicon nitride microchannel wall. The applied gate voltages were -37.5 - +25V for experiments. However, using +50 V gate voltages, the microdevice demonstrated a reverse EOF.

![Top View](Figure 2-6. The second FEFC LOC device by Schasfoort [48].)
2.5 EOF Modeling in Microchannels

Micropumps using electroosmotic flow (EOF) are of high promise to be essential in microfluidic devices because they consist of no moving parts and are easy to fabricate as well as integrate with other components in the microfluidic system. To develop the EOF micropumps, FEMLAB/COMSOL multiphysics as finite element analysis (FEA) tool is utilized to model and demonstrate the behaviors and effects of EOF in the microchannel. FEMLAB offers a complete environment that enables to perform all steps in the modeling process.

The typical modeling steps basically include (1) definition of the geometry, (2) definition of the physics in the volume and at the boundaries, (3) meshing, (4) solving, (5) visualization, and (6) postprocessing. 2-D models of the microchannel are considered to model and simulate in this work. The model uses the Conductive Media DC application modes and Incompressible Navier-Stokes application modes to numerically solve the problems under particular conditions. The models to be solved are the microchannels with and without gate electrodes or fluid flow control components. The particular geometry microchannel is selected to perform simulation. This section demonstrates on only general cases and ideas of modeling performed in this work; however, all specific conditions will be specified later along with the supporting experimental results. The Smoluchowski equation in equation 2.2 mainly comes into play to model the microchannel system:

\[ u_{eo} = -\frac{\varepsilon \zeta}{\eta} E_x \]
There are two modules used to solve for electric potential and flow velocity as follows:

1. Conductive Media DC Application Mode Definition

   From FEMLAB, the equation applied to solve for current and electric potential in microchannel system is:
   \[
   -\nabla \cdot (\sigma \nabla V - J^e) = Q_j
   \]
   where \(\sigma\) is the fluid conductivity (S/m), \(J^e\) is the current density, and \(Q_j\) is the amount of power generated per unit volume. Microchannel walls are defined to be electrically insulated, meaning no current can flow in or out through them. The inlet reservoir electrode is defined as electric potential \((V_0)\). The outlet reservoir electrode is specified as being ground or \(V=0\), meaning current escapes through this boundary. The conductivity of fluid, DI water in this case is 2.8 \(\mu\)S/cm.

   However, electrostatics application mode also can be utilized to solve in this case. Because the flow has no effect on the electric field, the model solves for the electric potential and then uses that solution to solve the velocity field. The electric potential can be calculated from:
   \[
   -\nabla \cdot \varepsilon_0 \varepsilon_r \nabla V = \rho
   \]
   \[
   -\nabla \cdot \varepsilon_0 \varepsilon_r \nabla V = 0
   \]
   Where \(\varepsilon_0\) is the electric permittivity of vacuum, and \(\varepsilon_r\) is the relative electric permittivity of the microchannel. The electric potential \((V_0)\) boundary condition on the inlet reservoir electrode is kept as low as possible. The outlet reservoir is defined as ground. In all other boundaries, the model uses zero charge/electric insulation boundary condition. The model
uses a relative permittivity of 78.5 for water in the subdomain.

(2) Navier-Stokes Application Mode Definition

For modeling laminar steady-state flow or Stoke’s flow, the density in the Navier-Stokes equation is set to zero. To analyze the free-flow condition, there is no pressure gradient in consideration. So, the equations can be written:
\[
\nabla \cdot [-p I + \eta (\nabla u + (\nabla u)^T)] + F = \rho (u \cdot \nabla)u
\]
\[
\nabla \cdot u = 0
\]
\[
-\nabla \cdot \eta (\nabla u + (\nabla u)^T)] + F = 0
\]

Where \( u \) is the flow velocity, \( \eta \) is the fluid viscosity, and \( p \) is the pressure inside the bulk fluid. The inlet and outlet reservoir are defined as normal flow/pressure and outflow/pressure boundary condition, respectively. The microchannal walls are set as inflow/outflow velocity. The velocity is tied to the electric potential by using the tangential electric field components on the velocity components to model the electroosmotic effect. The model uses water with a value of 0.001 N.s.m\(^{-2}\) or 0.001 Kg.m\(^{-1}\).s for the viscosity and 1000 kg.m\(^{-3}\) for the density.

2.6 Conclusion

The features of EDL, EOF, and FEFC technology were presented in this chapter. EDL results from the charge separation at the fluid-wall interface. As a result of the non-uniform distribution of counter-ions near the interface, there exists a plane of shear, which has a potential known as zeta potential. The counter-ions in the fluid move under the influence of the longitudinal electrical field and drag the neutral molecules with them,
thus generating EOF. FEFC is a technique to adjust the counter-ion concentration at the plane of shear with a transverse electric field for direct control over EOF without adjusting the longitudinal electric field. The modeling procedure using FEMLAB was also demonstrated in this chapter.
3. FABRICATION, MEASUREMENT, FLUID VARIABLES AND THEIR EFFECTS IN MICROFLUIDIC DEVICES

3.1 Fabrication of Microfluidic Devices

3.1.1 Brief Review of Polydimethsiloxane (PDMS)

Polydimethsiloxane (PDMS), called Silicone Encapsulants in general, has been a popular material used to fabricate microfluidic devices due to many advantages over other materials. First, PDMS is a low-cost material comparing to such materials as silicon and glass in traditional microfabrication materials. Second, the material needs to have suitable properties for microfluidic applications; for instance, sustaining their physical and electrical properties beyond a broad range operating conditions, good chemical stability, long-term and reliable protection, and resistance to ozone and ultraviolet degradation [53, 54].

Other advantages of PDMS [55] are as follows: (1) No waste products such as water generated from the chemical reaction (2) suitable for electrical/electronic potting and encapsulating applications (3) durable dielectric insulation, and (4) available in a variety of useful forms as conformal coating, encapsulants and adhesives. Physical properties and appearance of PDMS [55] are transparent, colorless, odorless and flowable liquid. They have cures to flexible elastomers, constant cure rate regardless of sectional thickness, service range of -45 to 200 °C (- 49 to 392 °F) and no post cure required.
3.1.2 Fabrication Procedure

The conventional photolithography and soft lithography technique were applied in this work to metal substrate to produce a channel pattern. Soft lithography technique is a technique using a patterned elastomer as a mask or mold, which is straightforward and time-effective when compared to traditional etching and bonding approaches. It can provide means to fast prototype of repeated patterns and structures with low cost [56]. The fabrication steps are illustrated in Figure 3-1. Photoresist patterning method could be referred to ref [57]. The explanation of the fabrication procedure is detailed in this section.

Figure 3-1. PDMS patterning procedure using conventional photolithography and soft lithography technique.
A) Mold Making using Conventional Photolithography and Soft Lithography Technique

Mask Design and Making

1. Mask layout is designed using drawing software, i.e., MS Powerpoint. It is then printed out on a laser transparency with high quality resolution to serve as masks as shown in Figure 3-2. Any black areas on masks will be etched away. Mask size is no greater than 4”x 4” for this particular case.

Material Preparation and Photoresist Patterning

2. Metal is cut with size of ½ inch larger than every side of the designed mask. Meanwhile, laminator is turn on.

3. 2 pieces of laminator carrier is prepared with both of which are ½ inch larger than every side of the metal.

4. Photoresist film is cut from its large piece with ¼ inch size larger than every side

Figure 3-2. Mask layouts created by drawing software.
surrounding the metal. Photoresist film is sensitive to light, so yellow light happens to help when working with this material.

5. The metal is cleansed before placing the photoresist film.

6. The clean metal is soaked with water to help the photoresist film well seal on the metal. Before placing the photoresist film, one side of the two protective layers of the photoresist film is first removed. The photoresist film with no protective layer side is then put on the metal soaked with water. It needs to make sure whether the photoresist film is well on the metal and no air bubbles or water beads are introduced between the metal and photoresist film.

**Lamination**

7. The metal with the photoresist film is sandwiched between 2 pieces of the carrier sheet. The gross side of the two carrier sheets are faced each other and the photoresist film. The sandwiched photoresisted metal is inserted into laminator. It needs to check the condition after laminating. Further laminating may be needed if necessary. The photoresist film should be well on the metal without spots or trails. If photoresist film condition is unfavorable, stripper step in Step 12 is needed to eliminate the photoresist film. Steps 4-7 are repeated.

**Mask Alignment and Exposure**

8. Plexiglass is used to help align the designed mask on the laminated photoresisted metal.

9. The laminated photoresisted metal with the aligned mask is exposed to light generated from 150 watt lamp for 8 minutes. The distance between them is around 4”.
The pattern as the designed mask should be visible after exposure.

**Developing**

10. ¼ oz of Sodium Hydroxide (NaOH) is diluted with 8 oz of warm water for developing.

11. The remaining protective layer is removed before developing. The metal with the pattern after exposure is immersed in the developing mixture for 30 seconds. The unhardened area should be eliminated and pattern forming should be visible. The material is pulled out and stopped developing with rinsing it under running water. It needs to examine if further developing is needed. However, if the material is over-developed, the photoresist film may be too soft, flexible and readily coming off the metal. Brushing and stirring may be helpful while developing if necessary.

**Stripper**

12. If the photoresist pattern is unfavorable, the undiluted NaOH is employed to strip the photoresist film off the metal. The steps 4-12 are repeated to create photoresist pattern.

**Mold Assembly**

13. Walls with a higher thickness than the photoresist pattern are placed to enclose the photoresist patterns to create molds as shown in Figure 3-3.

**B) PDMS Preparation and Patterning**

1. PDMS prepolymer is supplied 2-part silicone elastomers: Base and Curing Agent (SYLGARD 184 Silicone Elastomer Kit, Dow Corning, Midland, MI). Two parts are thoroughly and smoothly mixed at ratio of 10:1 v/v or w/w. Mixing time depends on
Figure 3-3. Molds with the photoresist patterns surrounding walls.

amounts of used base and curing agent. The more base and curing agent amount, the longer mixing time.

2. The mixture in Step1 is allowed to set and reduce air bubbles introduced during mixing for around 30 minutes or until there are no visible bubbles.

3. The PDMS mixture is poured over the mold with the designed pattern. A flat and smooth blade is used to smoothly flatten the PDMS mixture on the mold surface while maintaining contact between them.

4. Thermal cure at 125°C (257 °F) for 20 minutes is applied to allow the PDMS mixture to polymerize. It needs to ensure that well-cured PDMS film is accomplished, which can be checked if it is easily removed from the metal and well dry when touching the PDMS film after cure.
C) Microchannel Assembly

1. The patterned PDMS film is removed from the mold and placed on the substrate. The substrates used in this work are glass slide and ITO glass slide. The PDMS film sealed to the substrate is reversible so that the devices are easily cleaned between tests. The reversible seal of PDMS is due to its elasticity which allows a high degree of contact. The seal is fast, watertight, and occurs at room temperature. However, it always needs to ensure that there are no bubbles between substrate and PDMS film.

2. Inlet/outlet reservoir accesses are drilled. PVC tubes are employed to serve as Inlet/outlet reservoirs. 5-min epoxy glue is used to prevent leakage between reservoirs and patterned PDMS film and to strengthen other connections if necessary. Figure 3-4 shows finished microchannel assembly. It needs to ensure if fluids can go all the way through from inlet to outlet reservoir prior to operation. Applying pressure may be helpful at either one of reservoirs if necessary to allow fluids to occupy throughout the microchannel; however, it needs to be sure not to introduce any further air bubbles.

Figure 3-4. A single PDMS-based microchannel with inlet/outlet reservoirs on glass
3.2 Characterization of Microfluidic Devices

3.2.1 Variation in Experimental Results

Figure 3-5 shows an example of the flow measurements with variance. The measurements were performed on a single microchannel with DI water of 2.8 \( \mu \text{S/cm} \) at a range of applied electric fields of 10-50 V/cm. The flow shows linear relationship between applied voltage and flow rate.

The results exhibited variance due to many reasons [58]. The difference in surface curvature between injections with the manual filling process and surface roughness of the reservoir walls might cause the variance. The wetting of the buffer solution along the reservoir walls varied because of the manual injection of the solution from a pipette. For

![Graph](image)

Figure 3-5. Applied voltage versus measured flow rate of a single microchannel with dimensions of 1 cm in length, 1 mm in width, and 50 \( \mu \text{m} \) in height.
this reason, human error was a significant factor in running the experiment since the same surface curvature could not be repeated between injections. As a result of the different fluid curvatures, the surface tension forces were different between the reservoirs, yielding net pressure forces at the reservoirs. The different pressure forces pumped the buffer solution through the microchannel, even without the application of the longitudinal electric field. With EOF, the pressure flow impeded or sped up the bulk flow rate, so the measured velocity was not purely EOF. Characterizing the pressure flow component would be required characterizing the fluid curvature at both reservoirs.

Additionally, the fluid was removed from the microchannel between tests with vacuum force from a pipette-aid so that the microchannel could be filled with fresh buffer solution. It is likely to assume that contamination of the microchannel wall occurred during this removal process due to the air flow through the microchannel. The surface chemistry that generates the zeta potential is sensitive to adsorption chemistry at the microchannel wall and was possible to have changed between tests, yielding variance in the zeta potential and diffuse layer capacitances.

3.2.2 Flow Visualization

Current monitoring method is only able to measure the velocity in a single microchannel. The microchannel or capillary acts as a variable resistor as the buffer solutions with different concentrations flow through and replace each other, thus resulting in current dropped due to the change in conductivity of the buffer solutions. The EOF velocity is calculated from the measured microchannel length and the time for the current to drop to a new steady-state value. Current monitoring cannot be used to visualize and
measure the flow in two or more interconnected microchannels and FEFC microchannels in a network design because the current splits or sums at the intersection.

In order to study the control of EOF in microfluidic device, flow visualization techniques are significant to investigate. For characterization of EOF in FEFC microfluidic devices, an imaging technique needs to be developed to enable velocity measurements and visualization in microchannels. Flow visualization is critical for FEFC since the change in zeta potentials will induce a pressure that disrupts the plug-like flow of EOF. The methods of caged fluorescence, alternative fluorescent dyes, Shah convolution detections, and micro-particle image velocimetry (PIV) were investigated in this section

A) Caged Fluorescence

Caged fluorescence [59] is a method to measure EOF in a microchannel. The caged dyes are fluorescent dyes made non-fluorescent through the binding of chemical groups, which are also present in the solution. A pulse of focused UV light (365 nm) unlocks a small volume of the dye for illumination. The illuminated spot traveling in the microchannel with EOF serves as a flow marker for recording the flow. The method is not suited for FEFC because of a high pH of the caged fluorescent dye and buffer solution. Recent studies report that the inclusion of the caged fluorescent dye changes the zeta potential at the microchannel wall, thus increasing the EO [60].
B) Fluorescent Dye

The mobility of a sample plug or dye is another method to measure and visualize EOF; however, the choice of neutral markers at low pH is limited [61]. For instance, Umbelliferone (Fisher Scientific, Pittsburgh, PA) with an excitation at 330 nm wavelength and an emission at 390 nm has been used in polymer capillaries for the EOF velocity measurement at low pH [62]. Umbelliferone was dissolved to a 10mM concentration in 20 mM acetic acid buffer solution at pH 4. The dye illuminated with poor intensity into the microchannel as seen in Figure 3-6. Higher concentrations of Umbelliferone were not possible without the onset of salt precipitation.

Additional investigated dyes were BODIPY FL and Oregon Green 488 (Molecular Probes, Eugene, OR), which have excitation at 504 nm and 490 nm and emission at 511 nm and 514 nm, respectively. The dyes were prepared separately with 20 mM acetic acid buffer at pH 4. In the microchannel, the fluorescent intensity of the dyes

![Figure 3-6. Microchannel T-intersection with Umbelliferone dye used to characterize](image)
EOF [61].
illuminations was too low for observation due to the low pH of the buffer solution. The
dyes illumination intensity falls off at pH < 7 and no fluorescence is possible at pH < 5[63].

C) Shah Convolution Detection

Shah convolution Fourier transform is another technique for the velocity measurement in microfluidic devices [64]. In this method, a mask with a periodic array of slits is fabricated on the microchannel or superimposed onto the video. The slits spatially modulate the excitation beam aimed at the microchannel. Fluorescent microparticles with the diameters of micrometer to nanometer are introduced into the microchannel. Once the microparticles flow across the slits, the spatial modulation is converted into a temporal modulation. The distribution of velocities is found with a Fourier transform of the temporal signal with the peaks identifying the frequency of the microparticles after transformation. The known length of the slit spacing and the microparticle frequency are needed to obtain the velocity for flow characterization. This method was tested for FEFC flow measurements. A masking grid was superimposed onto the recorded flow video to act as the periodic array of slits. A distinct frequency peak after the Fourier transformation of the video was not distinct for the velocity measurements, but large scatter was observed yielding in conclusive measurement instead.

D) Micro-Particle Image Velocimetry

A promising flow imaging method for the velocity measurement is particle
Figure 3-7. Microparticles in a microchannel with velocity vectors under an excitation of 465-495 nm [65].

imaging velocimetry (micro-PIV) [65]. This work utilized this method for velocity measurement and flow observation. Video imaging is used to record the displacement of the microparticles moving with the EOF within a known time interval to obtain the velocity fields in a microchannel. This technique can measure planar flow fields [65] and 3D flow fields [66].

Moreover, micro-PIV of the Brownian motion of nanometer diameter microparticles can be used to measure the temperature in a microchannel [67]. Their displacements between subsequent video frames were measured for the velocity of the individual microparticles since the neutralized microparticles move due to EOF and pressure. With this technique developed, the characterization of flow in microchannel networks could be effectively studied. Standard PIV techniques use a cross correlation algorithm to extract velocity vectors. Each frame was recorded for the displacement measurements. The center position for each microparticle was obtained with a Sobel edge detection subroutine. For example, fluorescent polystyrene microparticles flowed in the microchannel under EOF in the experiment of FEFC. A sample image with velocity vectors is shown in Figure 3-7.
3.3 Fluid Variables and Their Effects on EO Performance

There are some basic variables dictating EOF performance. In this thesis, some relevant factors were demonstrated with supporting experimental results, conductivity and viscosity of the fluids. Also, undesirable effects normally unavoidable in the pumping experiment, like electrolytic reaction and thermal effect, were also presented in this section.

3.3.1. Fluid Conductivity

A) Effect on Electric Double Layer Thickness

From the double layer thickness equation in equation 2.1:

\[ \kappa^{-1} = \left( \frac{\sigma k T}{2e^2 z^2 n^0} \right)^{1/2} \]

\( \kappa^{-1} \) is obviously seen to decrease inversely as the square root of the counter-ion concentration which is directly proportional to the fluid conductivity. The higher fluid conductivity, the thinner electric double layer thickness. For instance, DI water with bulk conductivity ~10\(^{-4}\) S/m has estimated double layer thickness of ~100 nm, and acetonitrile with bulk conductivity of ~10\(^{-6}\) S/m has estimated double layer thickness of ~1000 nm [68].

B) Effect on Current

Another significant matter due to the working fluid conductivity is the total current, \( I \), which is the sum of the bulk conduction current, \( I_b \) and surface current, \( I_s \), which is carried by the motion of ions inside the charged double layer. As in Figure 3-8a
Figure 3-8. (a) Applied voltage versus current and (b) Applied voltage versus measured flow rate, in a single microchannel using different fluid conductivities.
especially for fluid conductivity of 14.63 \( \mu \text{S/cm} \), the double layer thickness is much thinner than the channel dimensions (1-cm long, 1-mm wide, and 50-\( \mu \text{m} \) high) due to its high fluid conductivity, so most of the current is carried by \( I_b \) in the bulk fluid, where a net Maxwell force that drives the fluid does not exist. Surface current should dominate bulk conduction current to generate the work of useful EOF, hereby creating high EO pump efficiency [68]. According to Figure 3-8b, as only small current is carried by thin EDL due to high conductivity, 14.63\( \mu \text{S/cm} \) in this case, low flow rate can be produced.

C) Effect on EO Pump Efficiency

Pumping efficiency is one of the important measures for liquid-driving devices. EO micropumps are very inefficient if the working fluid is relatively conductive. EOF relies on a net Maxwell force when an electric field is applied to a region containing mobile ions which have a collective net charge. This net charge exists only within the EDL surface, typically around several nanometers in thickness of mostly used electrolyte buffer solutions. Thus, the EO pump efficiency is obviously confined to the thin double layer [68]. Pumping efficiency can be defined as the useful pressure work that the working fluid can supply over total power consumption, called the thermodynamic efficiency as:

\[
\eta = \frac{\Delta P Q}{VI}
\]

The pressure work is \( \Delta P Q \) while the total power consumption is \( VI \). In the microchannel system, the hydraulic diameter of the channel cannot be smaller than the EDL thickness as polarization, Maxwell force and flow would diminish significantly.
Figure 3-9. Flow rate versus total power consumption using different conductivities of fluids.

Figure 3-9 is the experimental results performed in a single microchannel with dimensions of 1-cm in length, 1-mm in width, and 50μm in height using different fluid conductivities, 2.8µS/cm and 14.63 µS/cm. The results clearly show that the high conductivity of the fluid consumes high input power to generate the flow since most of the power is consumed in the bulk fluid, thus generating no pumping work thus decreasing pump efficiency.

Therefore, due to the importance of the EDL thickness to EO pump performance, low ionic concentration is generally preferred for less power input and high pump efficiency. Lower power consumption of the EO micropumps is mainly due to its low conductivity of the fluid, like DI-water as obviously proved from the results in Figure 3-9. DI-water is widely used due to its low conductivity, high latent heat capacity.
for heat transfer applications and its compatibility with many biological buffers in wide applications the μ-TAS [68].

3.3.2 Fluid Viscosity

From the Smoluchowski equation in equation 2.2:

\[ u_{eo} = -\frac{\varepsilon \zeta}{\eta} E_x \]

Obviously, viscosity, \( \eta \), comes into play as one of the dictating factors of the fluid property to pump performance that is inversely proportional to the EOF velocity [69]. This factor limits the EOF pump performance particularly when the working fluid has a significant magnitude of viscosity. In Figure 3-10, using a single PDMS-based microchannel with different fluid viscosities, 0.001 and 0.007 Pa.s, at the same level of applied voltages, serum growth media moves slower than DI water, approximately 5-7 times due to its high viscosity. This implies that serum growth media requires higher input power to generate the same level of flow rates as DI water.

3.3.3 Electrolytic Reaction

A) pH Gradient

A high field is not suitable in biological analysis since evidence of a pH gradient develops between the electrodes at inlet and outlet reservoir because of electrode reactions releasing water electrolysis products, proton and hydroxide ions. pH gradient affects a \( \zeta \) potential gradient which is sensitive to pH [70]. To visualize the pH gradient, phenolphthalein indicator can be employed to observe the concentration profiles
Figure 3-10. Applied voltage versus measured flow rate in a single microchannel using two different viscosities of solutions.

of the hydroxide ions. The indicator is colorless under neutral and acidic conditions (pH ≤ 7) but becomes pink to purple in a basic condition, ranging pH 8.2-10 as seen in Figure 3-11.

Based on the accomplished experiment, to investigate on pH gradient, before applying voltage, a few drops of phenolphthalein were dropped in both inlet and outlet reservoir already filled with DI water. After applying voltage of 3 KV for a while, DI water in the cathode reservoir was gradually turned pink. Therefore, the result clearly indicated that pH of DI water was increasingly changed since the electrode reservoir released hydroxide ions after applying high voltage.
B) Fluid Damage

In addition to pH gradient effect when applying high voltage to the microchannel, the working fluid can be also changed its physical properties, like color, or even destroyed. For example in Figure 3-12, the voltage at 40 V was applied over Pt wire electrodes of both reservoirs, the serum growth media solution utilized as a working fluid in this case was changed its color from orange to pink, indicating obviously that electrochemical reaction took place. The vial of serum growth media solution designated its real color in Figure 3-12.
C) Bubble Generation

Once voltages are applied over inlet and outlet reservoir of the DCEO micropumps, electrolytic reaction takes place at contact between electrodes and fluid. Protons and hydroxide ions released from the reaction result in bubble generation. Figure 3-13 shows bubble generation at Pt wire electrodes in DI-water after applying 3 KV. Bubble generation reaction at electrodes is fatal for the microchannel flow because bubbles can aggregate and block the electrical pathway of the microchannel, hereby decreasing EO pump efficiency. However, Palladium (Pd) wire can be employed in place of Pt wire to absorb H ions, thus leading to bubble reduction.
Figure 3-13. Bubble generation from the electrolytic reaction at Pt wire electrodes after applying 3 KV.

3.3.4 Thermal Effect

When EO micropumps were designed, the effect of fluid property variation on EOF under non-isothermal conditions is an important consideration. The temperature gradient in the flow channel could be attributed to Joule heating or to the thermal inequilibrium due to external factors. It was found that increasing the applied voltage significantly, thus generating Joule heating, could improve the EO mobility. When Joule heating was generated, a viscosity gradient within the channel was created and EDL was influenced [71]. Thus, Joule heating or temperature gradient has a significant effect on EO pump performance.
3.4 Conclusion

The primary purpose of this chapter is to develop research strategies for the development of micropumps. Detailed fabrication procedure was described. It was proved that PDMS-based microchannels could be easily fabricated and implemented with low cost using conventional photolithography and soft lithography technique. Flow velocity measurement and flow visualization techniques were investigated to find the best technique for the FEFC application. Micro-PIV was best suited since measurement of the EOF velocity could be done without operating limitations at particular pH levels, changing any flow properties, or introducing unfavorable effects to EOF. Moreover, relevant fluid factors and electrochemical reactions were demonstrated with supporting experimental results along with succinct principles.
4. PDMS-BASED MICROCHANNEL PUMP DESIGN

4.1 Introduction

A major aspect of microfluidics refers to the manipulation of minute amount of fluid flows in a microchannel. Mechanical pumps that use a membrane actuated by various forces, e.g., piezoelectric, electromagnetic, pneumatic, are capable of pumping fluids of various physicochemical properties; however, the flow is pulsed, and the fabrication of the micropump is relatively complicated. Nonmechanical pumps, with no moving parts that operate in a variety of principles, e.g., electrokinetic, electrohydrodynamic, etc., have been implemented. The flow by these pumps is generally pulse-free and varies from a few nanoliters to hundred microliters per minute.

The utilization of electroosmosis for pumping was demonstrated in open and packed capillary columns as well as for open-channel microchip platforms [71]. The generated flow uses an application of electric field to generate electroosmotic flow or EOF. EOF is dependent on the surface charge of the channel walls and is sensitive to the physicochemical properties of the fluid, such as pH, ionic strength, and organic content. EOF can be easily changed if the channel surface is altered by contact with specific liquid types [71].

Alternatively, for some other techniques, such as micro-liquid chromatography (µLC) or flow injection analysis (FIA), or for some samples that require zero electric field conditions for their manipulation, fluid flows may or must be generated by differential pressure. Typically, this is accomplished by connecting external devices to
the microchip; for example, gas pressure generators, or syringe pumps. However, the connection of external devices to microchips can increase the system complexity, and integration and multiplexing capabilities may be compromised [71].

The major goal of this thesis is to develop a simple, miniaturized pumping unit capable of stable fluid delivery at a range of flow rates compatible with common analytical applications on LOC devices, i.e., 0.05-1 µL/min, and sufficiently small to enable multiplexing of individual pumps. A number of PDMS-based micropumps using electroosmosis principle were built with a single open-channel configuration. Varying channel geometries in width, length, and height was accomplished to investigate on channel geometry factors to pump performance in term of flow rate. With this channel configuration, a range of flow rates of 0.018-3.42 µL/min was generated. The microchannel was also designed to easily interface with other operational elements in a microfluidic device using Nafion membrane, a conductive polymer or a charge-selective salt bridge that permits diffusive and electrophoretic transport of ions, to serve as both a liquid junction unit and an interfacing junction [68].

This pump design offers several advantages. First, it can be easily fabricated and integrated on microfluidic substrates for analysis scheme to utilize for fluid propulsion of LOC devices, or micro-Total Analysis Systems (µ-TAS). Second, the ability to control reproducibly very low flow rates enables the utilization of the micropumps for many other µ-TAS applications, such as sample transfer, liquid chromatography [71]. Third, its fabrication using standard photolithography and soft lithography ensures high manufacturing reproducibility. Lastly, the simplicity of the design ensures robustness,
reliability, and trouble-free operation. The fundamental principles behind the operation of the pump design and its implementation on glass substrate are presented.

4.2 Fundamentals of an Open Channel Electroosmotic (EO) Micropump

4.2.1 Introduction

Ideally, EO micropumps should be capable of delivering a large range of fluid flow rates independent of backpressure imposed by an existing network of microfluidic channels on the system. An optimum pump design is required to ensure that EOF can create pressurized flow in the system without flow leakage. The approach is to design a simple, miniaturized pump with a single open microchannel configuration with high aspect ratio, i.e., large width to height, to produce EOF and result in a necessary hydraulic resistance to pressurized backflow leakage. The single, small-diameter open-channel EO micropump could generate flow if the backpressure is small, and the flow is independent of the backpressure [71].

4.2.2 EOF Generation in Open Channel Configuration

To illustrate the principle of the open-channel EO micropump, an ideal system composed of rectangular PDMS-based microchannel and cylindrical capillaries is considered and examined on the conditions required for proper pumping. For the discussion, contributions of local hydraulic resistances to total pressure drop will be neglected. The standard equations for pressure-driven flow (PDF) and velocity, \( Q_{\Delta p} \) and \( v_{\Delta p} \), and electroosmotic flow (EOF) and velocity, \( Q_{\text{EOF}} \) and \( v_{\text{EOF}} \), in open channel
systems are as follows [71]:

In general cases,

\[ Q_{\Delta \rho} = \frac{\pi \Delta \rho}{128 \eta} \frac{d_h^4}{L} \]  
\[ V_{\Delta \rho} = \frac{1}{32 \eta} \frac{\Delta \rho}{L} d_h^2 \]  
\[ Q_{EOF} = \frac{\pi \varepsilon_0 \varepsilon_r \zeta}{4 \eta} \frac{U}{L} d_h^2 \]  
\[ V_{EOF} = \frac{\varepsilon_0 \varepsilon_r \zeta}{\eta} \frac{U}{L} \]

where \( \Delta \rho \) is the pressure drop across the microchannel of hydraulic diameter, \( d_h \) and length, \( L \), \( \eta \) is the fluid viscosity, \( \varepsilon_0 \) is the electrical permittivity of the vacuum, \( \varepsilon_r \) is the relative permittivity of the medium or dielectric constant, \( \zeta \) is the zeta potential of the microchannel wall, and \( U \) the voltage applied across the microchannel of length \( L \), and \( d_h \) is the hydraulic diameter of the microchannel with the relationship:

\[ d_h = \frac{4A}{P} \]

where \( A \) is the cross-sectional area of the microchannel and \( P \) is the perimeter of the cross-sectional area of the microchannel.

From equations 4.1 and 4.3, it is obviously seen that \( Q_{\Delta \rho} \) and \( Q_{EOF} \) are dependent on \( d_h^4 \) and on \( d_h^2 \) respectively. Also, \( V_{\Delta \rho} \) is dependent on \( d_h^2 \) while \( V_{EOF} \) is independent of \( d_h^2 \). Therefore, if fluid flow generation and distribution in a microfluidic
device occurs by both pressure and EO mechanisms, the fluid flow can be expected to be induced in preferential directions.

Figure 4-1 is used to explain the fundamental for interfacing a pumping section to other microfluidic section. The system consists of a single small microchannel (Section I) with dimensions $d_{h1}$ and $L_1$ connected to a large microchannel with dimensions $d_{h2}$ and $L_2$.

If a potential is applied only to the Section I to generate EOF, dependent on $\sim d_{h1}^2$, the flow will be redistributed in both microchannels through a pressure-driven mechanism, dependent on $\sim d_{h1}^4$ and $d_{h2}^4$. Pressure generation is due to the fact that the field-free microchannel in Section II acts as a restrictor for the $Q_{EOF}$ produced in Section I under the influence of the electric field. The $Q_{EOF}$ will distribute into a forward flow through the large channel ($Q$) and a backflow through the narrow channel ($Q_b$). Pressurized fluid flow will be proportional to $(d_{h1}/d_{h2})^4$. Practically, for a ratio of $d_{h1}/d_{h2}$ of only 1:10 with $L_1$ being equal to $L_2$, the flow will be distributed in a ratio $Q_b/Q$ of 1:10000.

![Diagram of flow distribution in an interfacing system with a single microchannel.](image)

Figure 4-1. Diagram of flow distribution in an interfacing system with a single microchannel.
When all the flows are directed through the large channel, and when both pressure and a potential gradient are applied, the net flow is a sum of PDF and EOF. By balancing the EO input flow with the pressurized output flows, it is shown that the pressurized forward flow in the large channel is dependent on EOF in Section I according to equation 4.5. The $Q/Q_{EOF}$ ratio is dependent on channel dimensions but independent of actual EOF in the system, can be written as follows:

$$\frac{Q}{Q_{EOF}} = \frac{L_1/L_2}{L_1/L_2 + (d_{1h}/d_{2h})^4}$$

(4.5)

$Q/Q_{EOF}$ could be defined as an efficiency coefficient for a given pump since it will determine what fraction of the originally produced EOF is actually pumped forward in the system. $Q/Q_{EOF}$ values can be calculated from equation 4.5 and represented as a function of $d_{h1}/d_{h2}$ and $L_1/L_2$.

However, in the real microfluidic microchannel, channel hydraulic resistance ($R_f$) always comes into play for microchannel design, the equation can be defined as follows:

$$R_f = \frac{\Delta P}{Q_{\Delta P}}$$

$$R_f = \frac{\Delta P}{\pi \Delta P} \frac{d_h^4}{128 \eta L}$$

$$R_f = \frac{128 \eta L}{\pi d_h^4}$$

(4.6)
From equation 4.6, the channel hydraulic resistance is a function of $d_h^4$ and $L$. The short length and large hydraulic diameter are desirable to reduce the hydraulic resistance of the channel to enhance the flow rate. When the interfacing unit is considered as Figure 4-1, the relationship can be written by balancing pressure gradients at the channel interfacing connection, i.e., $\Delta P_1 = \Delta P_2$:

\[
\frac{Q_{\Delta P^2}}{Q_{\Delta P^1}} = \frac{L_1}{L_2} \cdot \frac{d_{h2}^4}{d_{h1}^4}
\]  (4.7)

To obtain an efficient fluid pumping for the interfacing system, based on equation 4.7, the dimensions of the Section II channel should have a large $d_{h2}$ and short $L_2$ to reduce the hydraulic resistance and be able to drive the fluid through the interfacing microchannel.

Pumping efficiency is an important measure for liquid-driving devices, which can be defined as the pressure work that the fluid can supply over total power consumption, called the thermodynamic efficiency in equation 4.8 [68]:

\[
\eta = \frac{\Delta PQ}{VI}
\]  (4.8)

The pressure work is $\Delta PQ$ while the total power consumption is $VI$. 

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4.3 A Single Open Channel Configuration with Different Channel Geometries and No Gate Component

4.3.1 Instrumentation and Methods.

The scheme of the EOF pumping system, based on a single PDMS-based microchannel is presented in Figure 4-2. The main objective is to investigate on channel geometry factors with DI water of 2.80 µS/cm, low conductivity used in a wide application of microfluidic area. Channel geometries with a single microchannel configuration are built on glass substrate with dimensions of 1 and 2 cm in length, 0.5, 1, 2 mm in width, and 50 and 250 µm in height. The fabrication procedure was illustrated in details in Section 3.1. All pumping microchannels have an inlet and outlet reservoir. The voltage is applied between reservoirs through Pt electrodes to generate a longitudinal electric field for EOF generation.

The flow velocity is measured using micro-Particle Imaging Velocimetry (micro-PIV) technique. Micro-PIV procedure was explained in Section 3.2.2. Briefly, the motion of 3-µm tracer particles in the microchannel is recorded for velocity measurement of EOF. The image recording is performed on Nikon LV 100D BF/DF/DIC/FLUOR Microscope equipped with CCD camera and Image Pro AMS 6.0 software. The velocity of the microparticles in the microchannel is recorded to find displacement within 10 frames per second. The flow rate is a function of measured flow velocity and channel cross-sectional area. Current is done using Keithley 2700 DMM/Data Acquisition System to measure voltage drop over 1 KΩ of resistant. Pressure work is not in consideration in this work. Effects during the experiments are observed and recorded for further development. The experimental results well agree with principles and are presented in this section.
Figure 4-2. (a) Schematic of the EOF PDMS-based pumping system on glass substrate, (b) Experimental setup and (c) Velocity measurement setup, for a single PDMS-based microchannel with equipment for testing.
4.3.2 Experimental Results

A) Channel Length Factor

The circuit analysis system is applied to clearly investigate on the current behavior in the microchannel system. The microchannel acts as a resistor with a length (L), width (W), and height (H) and the current can pass through when applied voltage. The measured current is very low using multimeter since channel impedance is normally high. Generally, resistance is directly proportional to L, but inversely proportional to cross-sectional area (A) of the microchannel (WxH). Also, the Smoluchowski equation in equation 2.2 comes into play to analyze the microchannel performance in regard to flow velocity, associated with the applied electric field, V/L, applied voltage over microchannel length. The flow velocity is measured directly from the microchannel using micro-PIV procedure. Moreover, power consumption is also considered to determine pump performance and efficiency following equation 4.8.

In Figure 4-3a, the measured current in the microchannel with 1-cm length is approximately 1.2 greater than that of the microchannel with 2-cm length as the resistance of the latter microchannel dimension increases, the channel current decreases. Theoretically, when the channel length increases by 2 times, the current should decrease by magnitude of 2 instead of 1.2 times as seen in the experimental results. The reason of the variation may be due to contamination when changing the working fluid between tests. Also, the fluid is removed from the microchannel between tests from a pipette-aid so that the microchannel could be filled with fresh buffer solution. It is assumed that contamination of the microchannel wall may occur during this removal process because
of the air flow through the microchannel. The zeta potential at channel surface is sensitive to adsorption chemistry and is possible to have changed between tests, thus yielding variance in the zeta potential and diffuse layer capacitances. However, liner relation between applied voltage and generating current is achieved.

The flow rate is dependent on a set of three factors, applied voltage, microchannel length and cross-sectional area. In case of the same cross-sectional area of the microchannel, the flow rate only relies on applied electric field, applied voltage over the microchannel length varied from 1 cm to 2 cm in this case. For a fixed voltage, the longer the microchannel is, the lower the electric field strength over the microchannel is. Comparing with 1-cm microchannel length, the applied electric field strength over the microchannel is reduced by 2, hereby resulting in reduction by magnitude of 2 of the flow rate for 2-cm microchannel length. As demonstrated in Figure 4-3b, the flow rate of the 1-cm microchannel length is about 1.9 times greater than that of the 2-cm microchannel length. The overall results are quite in good agreement with theory with a linear relationship between applied voltage and flow rate.

Figure 4-3c shows pumping capability of the micropump with power input. It happens that at the same amount of power consumption, the microchannel with length of 2 cm produces lower flow rate than that of length of 1 cm. The hydraulic resistance is directly proportional to the microchannel length, so with the increment of microchannel length, the flow rate is reduced. The microchannel with a short length is desirable for high pump efficiency in case of the same cross-sectional area of the microchannel.
Figure 4-3. Plots with the factor of the microchannel length, 1 cm and 2 cm: (a) applied voltage versus current, (b) applied voltage versus flow rate, and (c) flow rate versus power consumption.
Figure 4-3, continued.
B) Channel Width Factor

To analyze the effect of variation of microchannel width, the same approach as described earlier for analyzing the effect of microchannel length is applied. When the microchannel width increases, the microchannel resistance decreases and thus the microchannel current increases. With 4- time increment of microchannel width in Figure 4-4a, the measured current is around 3.8 times greater. With the same condition of the microchannel geometry, the measured flow rate is greater with magnitude of 3.58 times in Figure 4-4b. Based on Figure 4-4c showing flow rate versus power consumption relationship, the microchannel with higher width in case of the same length and height require low power input to generate the pumping effect. By increasing microchannel width, the hydraulic diameter of the microchannel increases, hydraulic resistance decreases, and thus the flow rate increases. Pump efficiency can be improved by the reduction of power consumption with increment of the microchannel width.

C) Channel Height Factor

The height variable affects the microchannel current in the same way as the width. With 5 times greater of the microchannel height, the microchannel resistance is reduced to increase the microchannel current, approximately 4.6 times based on the experimental results in Figure 4-5a. The hydraulic diameter of the microchannel increases with the height, thus resulting in higher flow rate and pump efficiency improvement as in Figure 4.5b-c. The micropump with a high height can produce a high flow rate with low power consumption as the hydraulic diameter of the microchannel increases while the microchannel load decreases.
Figure 4-4. Plots with the factor of the microchannel width, 0.5 mm and 2 mm: (a) applied voltage versus current, (b) applied voltage versus flow rate, and (c) flow rate versus power consumption.
Figure 4-4, continued.
Figure 4-5. Plots with the factor of the microchannel height, 50 µm and 250 µm: (a) applied voltage versus current, (b) applied voltage versus flow rate, and (c) flow rate versus power consumption.
Figure 4-5, continued.
4.3.3 Simulation Results with a Single Microchannel with No Gate Component

FEMLAB is utilized to model in this section and the general modeling method was explained in Section 2.5. The objective for this section is to simulate and study electric field and velocity distribution in the microchannel with a real geometry of length (L) to height (H), 2cm to 50µm or 400:1. DI water is employed as working fluid with viscosity of 0.001 N.s.m\(^{-2}\) or 0.001 Kg.m\(^{-1}\).s and density of 1000 kg.m\(^{-3}\). A relative permittivity of 78.5 for DI water is applied in the subdomain of the model. To simulate only EOF in the microchannel, no pressure gradient is considered. The electric potential of 40 V is applied over the microchannel. The Smoluchowski equation as in equation 2.2 comes into play to model the microchannel system. The zeta potential of all channel surfaces is approximately 0.146 V obtained from calculation based on experimental results. In the model, since EOF is the surface flow, only velocity in \(x\) direction (\(x\)-velocity) is in consideration. Gat voltage is not applied in the model.

Figure 4-6a shows the real channel geometry the same as using in experiment using drawing tool provided in FEMLAB. The electric field strength, 2000 V/m shown in Figure 4-6b is constant throughout the microchannel with direction from positive to negative polarity. The color bar scale on the right of the figure indicates a magnitude of electric field strength. The velocity field distribution shown in Figure 4-6c is uniform throughout the microchannel with a flow velocity, 202 µm/s. and with flow direction from inlet to outlet reservoir indicated by arrows. The velocity field profile at center point from inlet to outlet reservoir (2 cm in length) and at channel cross section (50 µm in height) is constant all over the microchannel, 202 µm/s, as in Figures 4-6d and 4-6e. The simulation results support the experimental results and in good agreement with EOF principle.
Figure 4-6. A single microchannel modeled using FEMLAB: (a) schematic of channel geometry, (b) electric field strength and distribution using arrows indicating field direction, (c) velocity field distribution using arrows demonstrating field direction, (d) velocity field profile at center point of the microchannel starting from inlet to outlet reservoir, and (e) velocity field profile of channel cross section.
Figure 4-6, continued.
Figure 4-6, continued.
Figure 4-6, continued.
Figure 4-6, continued.
4.4 Micropump Interfacing with External Load

4.4.1 Experimental Procedure

The instrumentation and methods and fabrication procedure are similar to that of the previous setup in Section 4.3 except for inclusion of an interfacing section to the pumping system using another PDMS-based microchannel with dimensions of 1-cm in length, 0.5-mm in width, and 50-µm in height to serve as electric-field-free flow channel or pressure-driven flow channel as schematically shown in Figure 4-7 to test fluid delivery capability of the designed micropump. The pumping unit utilizes the microchannel of the same geometry as the interfacing unit. All microchannel units are mounted and integrated on glass substrate, which is well suited for LOC application.

To connect a field pumping to field-free pumping section, the Nafion membrane (Perma Pure, NJ) is employed to serve as both a liquid junction unit and as an interfacing junction. The Nafion membrane allows electric current penetration by counter ions to sustain electrolytic reaction on the cathode electrode outside of the flow channel. It also helps prevent electrolytic bubble and co-ion interference in the flow channels since bubble generation reaction is fatal for the microchannel flow. Once gas bubbles get into the channel, it would increase the effective viscosity of liquid dramatically and aggregate as well as block the electrical pathway. Moreover, the Nafion membrane helps prevent flow leakage into electrode reservoir and hence maximize the hydrodynamic pressure build-up created by EOF inside the channel. As the operation electric current in the experiment is very small (µA), concentration polarization near the Nafion surface could be neglected [68].
4.4.2 Experimental Results and Discussion

The pump was tested and proved that it is capable of driving DI water of 2.8 µS/cm conductivity and could produce a range of flow rates, 0.017-0.097 µL/min under the application of electric field, 10-50 V/cm. The micropump consumes around 2.3 µW to produce about 0.1 µL/min under 50 V/cm applied electric field with a portable size, which is well compatible for analytical application on LOC device. There exists no visible bubble invasion in the flow channels. Figure 4-8 illustrates plots of comparison between the pump with no interfacing unit and with interfacing unit.

Figure 4-8a shows applied voltage versus current. The current of the microchannel with the interfacing unit is reduced a little comparing to the one with no interfacing unit since the pump probably works against increased hydrodynamic load. Theoretically, by balancing the EO input flow with the pressurized output flows, it is shown that the pressurized forward flow in the microchannel Section 2 is dependent on EOF in Section I according to equation 4.5. The \( Q/Q_{EOF} \) ratio is dependent on channel dimensions but independent of actual EOF in
the system. $Q/Q_{EOF}$ could be defined as an efficiency coefficient for the pump to determine what fraction of the originally produced EOF is actually pumped forward in the system. Therefore, as the field pumping unit and field-free pumping unit has the same geometry, 1 cm in length, 0.5 mm in width, and 50 µm in height, the fraction of $Q/Q_{EOF}$ is down to 0.5.

The experimental results demonstrate about 0.54 time reduced in flow rate for the interfacing case, which approximately agree with the theory. Also, because backpressure generation may result from the work against channel hydrodynamic resistance, the flow rate can be reduced, thus generating slight variation to the flow rate in experiments differing from the theoretical results as seen in plots of experimental results in Figure 4-8b. More power input based on Figure 4-8c is required for the interfacing unit case to overcome the microchannel hydraulic resistance to generate the forward fluid flow.

4.5 Conclusion

This chapter has demonstrated the effect of microchannel geometries on EO pump performance. A single, simple, miniaturized pump configuration with high aspect ratio (W/H) was built in order to reduce the microchannel hydrodynamic resistance, thus resulting in flow rate improvement and power input reduction. No bubbles obviously were observed in all experiments. The micropump with no interfacing section can provide a range of flow rates, 0.018-3.42 µL/min, sufficient for micro-total-analysis systems (µ-TAS). The designed microchannel was simply easy to be fabricated and integrated on glass substrate with high manufacturing reproducibility and low cost using traditional photolithography and soft lithography technique.
Figure 4-3. Plots of a pumping unit interfaced to a single PDMS-based microchannel using Nafion membrane: (a) applied voltage versus current, (b) applied voltage versus flow rate, and (c) flow rate versus power consumption.
Figure 4-8, continued.
Also, the interfacing section using a single PDMS-based microchannel was linked to the pumping system and integrated on glass substrate to generate driven field-free flow for the application requiring no electric field over sample manipulation. The Nafion film was utilized to serve as a channel interfacing junction and liquid junction unit to help prevent bubble invasion into the pump channel, prevent flow leakage and maximize the hydrodynamic pressure generated by EOF. A range of flow rates, 0.017-0.097 µL/min, can be produced under the application of electric field, 10-50 V/cm with a portable size to deliver fluids when interfaced with other microfluidic devices.
5. FIELD-EFFECT-FLOW CONTROL IN A PDMS-BASED OPEN MICROCHANNEL

5.1 Introduction

EOF is generally controlled by manipulating the chemical composition of the buffer solution, like conductivity, pH solution, buffer additives ionic strength, organic content, and solution pH, or by coating of the inner wall of the fluid channel [72]. In this thesis, the coating of the inner wall of the fluid channel is applied to modulate the zeta potential at the channel surface, hereby resulting in flow manipulation.

From the Smoluchowski equation in equation 2.2, the equation shows EOF can be manipulated by the electric field and the zeta-potential [73]. In a microchannel network, fluid flows can be controlled by adjusting the electric fields along the different channels via switching of the voltages at electrodes at the outlets of the microchannels. A drawback of this method happens in more complex networks of the microchannels independent control of the flow in the different paths and difficult to control due to all resistively linked channels of the electrical voltages [73]. Applying a transverse electric field to locally control over EOF is the effective way by modulating in the zeta potential, $\zeta$, that can be achieved in FEFC since it is dependent on the potential distribution at the channel wall-electrolyte interface.

A single open microchannel configuration is applied to investigate the manipulation of fluid flow. With many advantages beyond other materials mentioned in Section 3.1, PDMS is selected to fabricate a single microchannel on ITO glass substrate
serving as gate electrode. The FEFC is limited because of theirs dielectric breakdown of the coating of tin on the ITO glass. The maximum electric field that a material can withstand without breakdown is called its dielectric strength, \((E_b)\). The capacitance of the coating material of the channel wall determines the degree of EOF control in the double capacitor model as equation 2.14:

\[
\zeta_{\text{FEFC}} = \zeta_0 + \frac{C_w}{C_D}(V_G - V_i)
\]

For effective control over EOF, the wall capacitance must be increased. Selecting a material with a high dielectric constant \((\varepsilon)\) will increase the wall capacitance as equation 2.15:

\[
C_w = \frac{\varepsilon}{d}
\]

Additionally, the thickness of the coating, \(d\), is inversely proportional to the wall capacitance. The coating thickness and applied gate voltage are factors that counterbalance each other. Reducing the coating thickness will increase the wall capacitance for the improved FEFC, but the applied gate voltage must also be reduced in order to avoid dielectric breakdown. However, operation under high gate voltages will lead to electrical breakdown of the coating material. Therefore, the dielectric constant and dielectric strength are the two key factors in the coating material selection for FEFC.
5.2 A PDMS-Based Microchannel with Gate Component

5.2.1 Introduction

FEFC is demonstrated in a microfluidic device where the transverse electric field is applied through the microchannel wall to control over EOF. The application of microfluidic device is also aimed at the realization of an inexpensive, disposable microfluidic device. In this work, the FEFC device utilize ITO glass slide as gate electrode mounted by a single PDMS-based microchannel. The objective is to control over EOF in terms of flow velocity improvement and reduction as well as flow direction switching when gate voltage is applied at the gate electrode occupying from the microchannel midpoint to outlet reservoir. The FEMLAB is utilized to model the microchannel to explain and predict flow behavior and effect as well as to support experimental results.

5.2.2 Experimental Procedure

Instrumentation and methods for the experiment are similar to Chapter 4 investigation on a single open channel with no gate component. The fabrication procedure of the microchannel was fully explained in details in Section 3.1, except for the substrate. The gate electrode in which the tin layer on the ITO glass is partially etched away using Tin etchant to serve as gate electrode on half side of cathode reservoir. Micromolded PDMS forms the remaining three walls of the single microchannel with dimensions of 2 cm in length, 1 mm in width, and 50 µm in height.
The gate voltage is directly applied to the ITO glass substrate, which acts as the gate electrode on the bottom of microchannel to generate a transverse electric field to modulate counter ions at the surface channel, resulting in EOF manipulation. Contact is made to the ITO glass using Cu tape. The FEFC testing set-up is shown in Figure 5-1. The 20 V/cm electric field is applied between reservoirs through Pt electrodes to generate a longitudinal electric field for the EOF generation. The applied gate voltages to the ITO glass are ranged from ±0.1, ±0.2, ±0.3, ±0.4, ±0.5, and 0. The flow velocity and current are measured using micro-PIV technique and multimeter, respectively.

5.2.2 Experimental Results

A number of tests are conducted for each of the applied gate voltages. The comparison plot of gate voltage versus measured flow rate between experimental and simulation results under the applied electric field of 20 V/cm or 2000 V/m is shown in Figure 5-2. Overall, the micropump with gate control component can produce a range of flow rates, 0.063-0.236 µL/min with gate voltage application of -0.5 to -0.1, 0, and +0.1 to +0.5 V. First, when applying negative voltages to the gate, more counter ions are injected to EDL, hereby leading to flow velocity improvement. A range of flow rates is improved in relation with increment of magnitude in negative polarity trend, 0.170 up to 0.236 µL/min.

On the other hand, a range of fluid flow rates is reduced when varied positive gate voltages are applied since more co ions are injected in EDL and counter ions are driven away in the fluid bulk. A range of reduced flow rates is 0.163 down to 0.063 µL/min.
Figure 5-1. Microchannel setup with gate control component: (a) schematic of experimental setup for gate control study, and (b) a single microchannel equipped with measurement instrument for velocity measurement.
In Figure 5-2, the results of experiment are in good agreement to the results of simulation. However, the variation in the results may be attributed to pressure-driven flow in the microchannel due to a pressure gradient from unequal dispensed volumes in the fluid reservoirs between tests. The contamination to the channel surface as consequence of manual filling process of the fluid may cause zeta potential change and thus flow variance.

With the range of positive gate voltages in this work, reversal of EOF is not seen; however, the trend of gate voltage versus flow rate implies that if positive gate voltages increase until a particular voltage level, anions in EDL increase, so the fluid flow could
stop or even reverse its direction to the opposite side. EOF can be manipulated through gate control component to modulate the channel surface charge.

5.2.3 Modeling Procedure

The modeling method was explained in Section 2.6. The microchannel modeling layout is shown in Figure 5-3 using FEMLAB with real dimensions of H/L, 2cm/50µm, or 400/1. Briefly for modeling condition settings, Conductive Media DC and Navier-Stokes application modes are used to model the microchannel with gate component. In Conductive Media DC application modes, inlet electrode and outlet electrode are defined as electric potential (V₀ = 40 V) and ground, respectively to generate a longitudinal electric field. The gate element and the rest of microchannel walls are set as zero charge/electric insulation. In Navier-Stokes application mode to solve for velocity profile, inlet and outlet electrode are defined as normal flow/pressure and outflow/pressure, respectively. The gate voltage element is set as inflow/outflow velocity to generate a transverse electric field to locally control EOF. Since EOF is the surface flow, only x-velocity is in consideration to observe flow behavior at different locations in the microchannel, inlet, midpoint (the point at joint between no gate and gate control region), and outlet.

5.2.4 Simulation Results

With 40 V applied to reservoirs over electrodes, electric field strength distribution, 2000 V/m, is generated and distributed consistently throughout the microchannel, which is similar to those with no gate control component. Applied
Figure 5-3. Modeling layout using drawing tool in FEMLAB simulation software with real dimensions, H/L = 2cm/50μm or ratio of 400/1.

Gate voltages are applied, -5 V and + 5 V to modulate microchannel surface charge. Velocity field distribution obtained in Figures 5-4 and 5-5.

Based on the EDL mechanism, negative charges are induced on the surface wall electrostatically when in contact with fluids, so positive ions or counter ions are attracted towards the channel surface wall, hereby leading to EDL generation. With application of negative gate voltage, -0.5V, more negative ions or co-ions are adding in gate surface, thus attracting more counter ions in EDL. Under positive electric field application, counter ions or positive ions in EDL will be induced, move, and drag the bulk fluid with them due to viscous force of the fluid towards the outlet reservoir or in regard with electric field direction following Coulomb’s laws, to the right side in this case. The
Figure 5-4. Velocity field distribution results of -0.5 V of gate voltage case using FEMLAB: (a) demonstration of velocity distribution with arrows indicating direction and flow behavior, (b) illustration of velocity distribution profile at center point from inlet to outlet, (c-e) clarity of cross sectional velocity distribution profiles at inlet, midpoint, and outlet, respectively.
Figure 5-4, continued.
Figure 5-4, continued.
Figure 5-4, continued.
Figure 5-4, continued.
Figure 5-5. Velocity field distribution results of +0.5 V of gate voltage case using FEMLAB: (a) demonstration of velocity distribution with arrows indicating direction and flow behavior, (b) illustration of velocity distribution profile at center point from inlet to outlet, (c-e) clarity of cross sectional velocity distribution profiles at inlet, midpoint, and outlet, respectively.
Figure 5-5, continued.
Figure 5-5, continued.
Figure 5-5, continued.
Figure 5-5, continued.
velocity field strength becomes stronger over the gate region as more counter ions are induced in EDL than in the bulk fluid as seen in Figure 5-4a with arrows indicating field direction. However, there are also some co-ions or negative ions in EDL and in bulk of fluid, tending to move towards the inlet reservoir, so the flow curvature is seen demonstrating that flow velocity distribution is not uniform throughout the microchannel. As the counter-ions are majority with more induced counter ions due to applied gate, the induced counter ions movement dominates. The final direction of velocity field is towards the cathode reservoir.

Figure 5-4b shows non-uniform velocity field profile at center point of the microchannel starting from inlet to outlet boundary. Figure 5-4b shows the effect of gate voltages, causing the velocity not constant everywhere, 386 μm/s over first half of the microchannel and dropped down to 264 μm/s over the gate region in the microchannel since zeta potential at the channel surface is modulated. In this case of positive gate voltages, the velocity at the outlet is improved, from 202 μm/s with no gate voltage to 264 μm/s with -0.5 V gate voltages.

Figures 5-4c – 5-4e demonstrates cross sectional velocity distribution profiles at particular locations: inlet, midpoint, and outlet, for further clarity of distribution of the velocity field in the microchannel. At the inlet, Figure 5-4c, the velocity increases from the bottom microchannel to the center point and then dropped down to the same velocity as at the bottom at the top channel wall. This profile implies the possible pressure gradient taking place in the microchannel. Also, at the midpoint of the microchannel location, Figure 5-4d, as counter ions are increasingly adjusted as consequence of applied
positive gate voltages, the velocity becomes stronger over the gate surface and linearly decreases in the fluid bulk. In Figure 5-4e, the velocity distribution still shows non-uniformity; nevertheless, the overall velocity is improved due to the applied negative gate voltage.

On the other hand, when applying +0.5 V of gate voltage, electric field distribution and strength profiles are still the same as the negative gate voltage case. Since positive gate voltage of +0.5 V is applied, adding more positive ions in microchannel surface, more negative ions or co ions are attracted in EDL over gate region. In this case co-ions in EDL over the gate region come into play for dominance. Once positive electric field is applied, co ions or negative ions in EDL of the gate will be induced, move, towards the anodic reservoir following Coulomb’s law and drag the bulk fluid with them due to viscous force of the fluid, to the left side in this case. The velocity field strength becomes stronger over the gate region as more co ions are induced dominating counter ions in EDL than in the bulk fluid as seen in Figure 5-4a with arrows indicating field direction to the left side.

Particularly, at the joint between no gate region (left side) and gate region (right side), the competition of the flow direction is in action, thus leading to the clockwise circulating flow generation. The flow curvature is seen demonstrating that flow velocity distribution is not uniform throughout the microchannel. However, the counter-ions are majority, the induced counter ions movement dominates. The final direction of velocity field is still towards the cathode reservoir with lower velocity. Figure 5-4b shows non-uniform velocity field profile at center point of the microchannel starting from inlet to
outlet boundary. Figure 5-4b shows the effect of gate voltages, causing the velocity not constant everywhere. The negative velocity profile observed demonstrates the reversing flow direction. Since zeta potential is modulated through the positive gate voltage application, the velocity at the outlet is reduced, from 202 μm/s with no gate voltage to 89.5 μm/s with +0.5 V gate voltages.

Figures 5-4c – 5-4e demonstrates cross sectional velocity distribution profiles at particular locations: inlet, midpoint, and outlet, for further clarity of distribution of the velocity field in the microchannel. The profiles in the case are mostly opposite to the case for the negative gate voltage case. At the inlet, Figure 5-4c, the velocity decreases from the bottom microchannel to the center point and then goes up to the same velocity as at the bottom channel wall at the upper channel wall. The profile shows some reversing flow velocity takes place as seen the negative velocity profile. Also, at the midpoint of the microchannel location, Figure 5-4d, as co ions are increasingly adjusted as consequence of applied positive gate voltages, the velocity becomes negatively stronger over the gate surface and linearly increases in the fluid bulk as it relies on induced counter ions. In Figure 5-4e, the velocity distribution still shows non-uniformity and the overall velocity is reduced due to the applied negative gate voltage. However, with this width of these applied positive voltages, EOF reversal is not seen for the net flow velocity; nevertheless, if applied positive voltages increase until a particular voltage level, a reversing flow could be achieved.
5.3 Conclusion

The micropump with gate control component can produce a range of flow rates, 0.063-0.236 µL/min under applied voltage of 40 V over 2 cm length of the microchannel. FEFC experiments were demonstrated using a single PDMS-based microchannel with enabling a cost reduction in the implementation with effortless fabrication. The ITO glass slide was selected to serve as gate electrode. However, the critical properties of the gate coating material were the dielectric constant and dielectric strength, which determined the change in EOF for the applied gate voltage in the FEFC system.

Comparing with the micropump setup with no gate produces flow rate of 0.169 µL/min at 20 V/cm electric field application, flow rate range was improved with -Vg (0.170 up to 0.236 µL/min) and reduced with +Vg, (0.163 down to 0.063 µL/min). Overall flow rate range is 0.063-0.236 µL/min with Vg = ±0.1 to ±0.5 and 0. The experimental and simulation results well agreed with theory. Thus, EOF obviously can be locally manipulated with gate voltage application. However, reversal of EOF was not seen for the net flow in this work; however, it is possible with increment of applied positive voltages until a particular voltage level as can be predicted from the trend of the comparison plot in Figure 5-2.
6. CONCLUSION

The micropump with FEFC, a technique adjusting the counter-ion concentration in EDL with a transverse electric field, has been developed in this thesis. Modulating the zeta potential through the gate voltage on the bottom microchannel wall allows for a locally dynamic control over EOF without adjusting the longitudinal electric field, which is difficult in the microfluidic network with many microchannel paths. The experimental results were supported by simulation using FEBLAB, and both experiments and simulation were in good agreement with physical principles.

The effort to develop research strategies to enhance the FEFC micropump was described in Chapter 3. Detailed fabrication procedure was fully illustrated using traditional photolithography and soft lithography technique, which were proved effective and at low cost. Micro-PIV is well suited for this FEFC application since the measurement of EOF velocity could be done without operating at the device limitations that may introduce unfavorable effects to the EOF. Relevant fluid factors and electrochemical reactions associated with pump performance were included with supporting experimental results along with succinct principles.

The geometry variables to EO pump performance have been investigated to develop the micropump design. A single, simple, miniaturized pump configuration with high aspect ratio was applied to build micropumps to reduce hydrodynamic resistance of the microchannel for flow rate improvement and power input reduction. Also, the interfacing section using a single PDMS-based microchannel was linked to the pumping system and integrated on glass substrate to generate pressure driven field-free flow for
the application requiring no electric field over sample manipulation. The Nafion film was utilized to serve as a channel interfacing junction and liquid junction unit to help prevent bubble invasion into the pump channel, prevent flow leakage and maximize the hydrodynamic pressure generated by EOF. The results indicated that the micropump could provide a range of flow rates, 0.018-3.42 µL/min, sufficient for micro-total-analysis systems (u-TAS) under a low range of operating voltage of 10-50 V. The designed microchannel was simply easy to be fabricated and integrated on glass substrate with high manufacturing reproducibility and low cost.

The ITO glass was used as a gate electrode and substrate to apply a transverse electric field. All tests were done under 20 V/cm electric field. The micropump with gate control component can produce a range of flow rates, 0.063-0.236 µL/min, with gate voltages of ±0.1, ±0.2, ±0.3, ±0.4, ±0.5, and 0. The local control over EOF has been achieved. When negative gate voltages were applied, the flow rate was increased; on the other hand, fluid flow rate was reduced when positive gate voltages were applied. Simulation results using FEBLAB have well supported to the experimental results and further demonstrated the flow behavior and effect in the microchannel. The reversal of flow did not obtain in the experiments but is possible based on experimental and simulation results if the positive gate voltage increases until a particular gate voltage, the flow could be stopped and even reversed.
7. FUTURE WORK

The development of the DCEO micropump with FEFC control component in this thesis has been accomplished to locally manipulate EOF under 20 V/cm electric field applications and produce a range of flow rates suited for microfluidic applications. However, there are some drawbacks needed to solve. First, the operating voltages are still too high for LOC devices since some particular samples could not tolerate. As seen in the experiment of driving serum growth media solution, at 40 V, the fluid has changed its physical property, color in this case. Also, with high voltage applications, electrolysis occurs at the electrodes, which is undesirable for the microfluidic application. Also, a reservoir electrode configuration with high voltage application is another drawback for portable issue.

On the other hand, EO technique with AC voltage operation, ACEO technique, for micropumps can effectively deal with those problems. ACEO can operate at a few volts, which automatically enable to minimize electrochemical and electrolytic reaction and to be portable. The ACEO can be used for not only pumping unit but mixing and trapping unit as it produces recirculating flows. Moreover, electrodes can be integrated with microchannel with planar electrode configuration, suitable for local flow control for LOC applications.

The first focus for the future work is to design a pumping or driving unit utilizing ACEO technique. As ACEO tends to produce recirculating flows demonstrated in Figure 7-1, so to produce a net flow, or uni-directional flow, it is necessary to break AC electric field symmetry which can be achieved in two ways. (1) asymmetric electrode geometries, and (2) asymmetric polarizations.
Figure 7-1. Representation of counter recirculating vortices over a couple of electrodes due to capacitive charging polarization using ACEO technique.

The first way is to design a set of asymmetric electrode array with symmetric AC signal application to produce a small and large recirculating flow over each pair of electrodes with small separation between a pair. Large recirculating flows would dominate the small ones, so net flow could be achieved with this technique as illustrated in Figure 7-2.

Also, the latter way is to use a set of symmetric electrode array with DC offset application over AC signal. With a set of symmetric electrode array, electrodes are biased with AC signal with DC offset to approach asymmetric polarizations. There are two main polarizations dominating at different voltage applications. Capacitive-charging polarization dominates at 1-2 V and Faradaic charging polarization dominates at 2-4 V. The two different kinds of polarization have different direction of recirculating flows, coexist and tend to compete for dominance. So, in order to achieve asymmetric
polarizations, because these polarizations have different voltage dependence, one polarization can dominate another to generate the net flow or uni-directional flow by adjusting AC signal with DC offset as seen in Figure 7-3.

Though ACEO technique has many advantages beyond DCEO, however, one of its drawbacks is ACEO can be best suited for working fluids with a low conductivity as it relies on ion concentration in EDL. As mentioned in Chapter 2, fluids with low ionic strength would reduce double layer thickness containing mobile ions or counter ions which will be induced by electric field application to generate Maxwell force. Also, in the world of biological and chemical analysis, it demands a technique to drive samples with high conductivity, like human blood, serum. Thus, electro-thermal effect technique comes into attention for the future work to utilize for driving microfluidic samples.
Figure 7-3. Schematic diagram of ACEO physical mechanism using asymmetric polarization technique to generate a net flow or unidirectional flow.


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VITA

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