# Ion-Sensitive Properties of Organic Electrochemical Transistors

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ARTICLE

**ABSTRACT** Ion-sensitive properties of organic electrochemical transistors (OECT) based on Poly(3,4-ethylenedioxythiophene): poly(styrene sulfonic acid) (PEDOT:PSS) have been systematically studied for the first time. It has been found that the transfer curve ($I_{DS}-V_G$) of an OECT shifts to lower gate voltage horizontally with the increase of the concentration of cations, including $H^+$, $K^+$, $Na^+$, $Ca^{2+}$, and $Al^{3+}$, in the electrolyte. The gate electrode of the OECT plays an important role on its ion-sensitive properties. For devices with Ag/AgCl gate electrode, Nernstian relationships between the shift of the gate voltage and the concentrations of the cations have been obtained. For devices with metal gate electrodes, including Pt and Au, the ion sensitivity is higher than that given by the Nernst equation, which can be attributed to the interface between the metal gate and the electrolyte.

**KEYWORDS:** organic electrochemical transistor • ion sensor

## INTRODUCTION

Organic thin-film transistors (OTFTs) have been explored for sensing applications for several decades due to their many advantages over inorganic counterparts, including easy fabrication, low cost, flexibility, biocompatibility, etc. (1−3). Among these OTFTs, organic electrochemical transistors (OECTs) have attracted a great deal of interest in recent years because the devices can operate stably in aqueous environment with relatively low working voltages and are suitable for applications in chemical and biological sensing (1, 2). The first OECT based on polypyrrole was reported by Wrighton et al. in 1984 (4), who exploited a new direction in the field of OTFTs. From then on, several different conducting polymers were investigated in OECTs, such as poly(3-methylthiophene), polypyrrole, polycarbazole, polyaniline, and poly(3,4-ethylenedioxythiophene) (PEDOT) (5−17). In the OECTs, the conductivity of the active layer can be modulated by the drift of cations between the electrolyte and the organic semiconductor film under an applied gate voltage. Therefore, the transistor characteristics of the devices are normally sensitive to ion concentration in the electrolyte. On the basis of this principle, ion sensors (14−17) and pH sensors (12, 13) have been successfully developed using OECTs. In addition, OECTs have been used to make logic circuits (18), as well as sensors for detecting antibody−antigen interaction (5), DNA (6), glucose (7−9), penicillin (13), humidity (19), etc.

OECTs can be easily integrated into microfluidic channels to realize a "lab on a chip" system. Mabeck et al. first demonstrated a microfluidic-based OECT (16). Yang et al. reported a multianalyte biosensing based on the integration of a surface-directed microfluidic system with OECTs (20). Because the OECTs can be miniaturized without the degradation of the device performance (21), these devices are promising in many sensing application in the future, especially for high-density, high-throughput, low-cost, and disposable sensors.

However, all of the ion sensors have been characterized by simply measuring the channel current at constant operation voltages. The change of ion concentration in the electrolyte is represented by the variation of channel current (12−17). Therefore, a comprehensive description of the ion-sensitive behavior of OECTs from device physics point of view has not been developed, which is very important for the real application of OECT in various types of sensors. In addition, the possible factors that may influence the sensitivity of the ion sensors have never been addressed. Besides the organic semiconductor material used in the device, the design of the device and the gate electrode are also critical issues to the device performance (22), which have not been carefully studied in the ion sensors that have been reported before.

Poly(3,4-ethylenedioxythiophene):poly(styrene sulfonic acid) (PEDOT:PSS) has been widely used in organic devices, including organic solar cells, organic light emission diodes, and OTFTs (23). The doping of PSS in PEDOT not only increases the conductivity of the polymer but also solves the solubility problem (24). Because of the high conductivity and environmental stability, PEDOT:PSS is also an excellent candidate for the active layer in OECTs (1, 16, 17, 22, 25). In this paper, we systematically studied the ion-sensitive behavior of OECTs based on PEDOT:PSS in aqueous solutions with different cations, including $H^+$, $K^+$, $Na^+$, $Ca^{2+}$, and $Al^{3+}$. We have found that a near-Nernstian dependence exists between the effective gate voltage and the concentration of cations in the electrolyte and gate electrode plays an important role on the device performance, which gave us a better understanding for the physical and chemical mechanism of OECTs.

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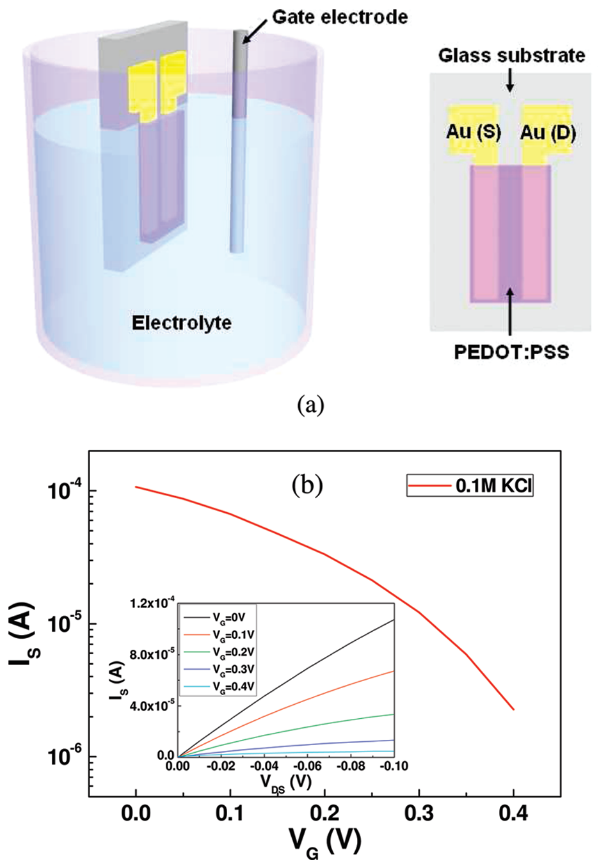

**FIGURE 1. (a) Schematic of an OECT-based on PEDOT:PSS. (b) Transfer characteristics of the OECT measured in 0.1 M KCl solution. A commercial Ag/AgCl (saturated KCl) electrode is used as the gate electrode. The inset shows output characteristics of the OECT measured in 0.1 M KCl solution.**

## EXPERIMENTAL SECTION

As shown in Figure 1a, OECTs are fabricated on glass substrates. The size of the glass substrates was 1 cm ×1 cm. Cr/Au source/drain electrodes were deposited on the top of the glass substrates through a shadow mask with a thermal evaporation (Edwards Auto306). After the substrates were cleaned with DI water, PEDOT:PSS (from Sigma-Aldrich) was spin-coated and patterned on the top of source and drain electrodes. The channel length and width of the devices were 0.2 and 6 mm, respectively. The thickness of PEDOT:PSS characterized by a scanning probe microscopy (SPM, Digital Instruments NanoScope IV) was about 80 nm. Then the devices were annealed at 200 °C for 1 h in a glovebox filled with high-purity $N_2$ (23). Each chip with an OECT was attached to a printed circuit board and the source/drain electrodes were connected to cables for the measurement. The left area of the source/drain electrodes with no PEDOT:PSS was packaged with epoxy and the devices are ready for characterization.

Aqueous solutions of different salts, including potassium chloride (KCl, Aldrich), sodium chloride (NaCl, Aldrich), calcium nitrate ($Ca(NO_3)_2$, Aldrich), and aluminum sulfate ($Al_2(SO_4)_3$, Aldrich), were prepared by dissolving the salts in DI water with different molar concentrations. The aqueous solutions with different pH value were prepared by diluting concentrated hydrochloric acid (HCl, Aldrich) with DI water and the pH values of the solutions were calibrated by a pH meter (HI98103, Hanna Instruments).

Each OECT has been immersed in an electrolyte, as shown in Figure 1a, and characterized with a semiconductor parameter analyzer (Agilent 4156C). A commercial Ag/AgCl (saturated KCl) electrode or metal wire (Pt, Au) was used as the gate electrode. For transfer characteristics, the channel current $I_S$ between source and drain was measured as a function of gate voltage $V_G$ under a constant drain voltage $V_{DS} = -0.1$ V. For output characteristics, the channel current $I_S$ was measured as a function of drain voltage $V_{DS}$ under a constant gate voltage $V_G$ and different $V_G$ resulted in different curves of $I_S$ versus $V_{DS}$. To obtain a stable result, the channel current at each point was measured after applying the voltage for 10 s.

## RESULTS AND DISCUSSION

We find that the type of gate electrode plays an important role on the performance of an OECT, and details are discussed later in this paper. To study the ion-sensitive property of OECTs, Ag/AgCl (saturated KCl) electrode is chosen as the gate electrode since its electrode potential is constant in different aqueous solutions (26). The OECT in a container, as shown in Figure 1a, is then characterized. Figure 1b shows the transfer characteristics ($I_S-V_G$, $V_{DS} = -0.1$ V) and output characteristics ($I_S-V_{DS}$ at different $V_G$) of an OECT measured in 0.1 M KCl solution. The channel current $I_S$ can be modulated for nearly 2 orders of magnitude by a gate voltage $V_G$ of only 0.4 V. The output characteristics show that the channel conductance of the OECT is dependent on the gate voltage and the current from the gate electrode ($\ll 1$ $\mu$A) is negligible compared with the channel current.

OECTs have been found to show similar performance in electrolytes with many other cations, such as $H^+$, $Na^+$, $Ca^{2+}$, and $Al^{3+}$. These cations including $K^+$ can be injected into the PEDOT:PSS layer under a positive gate voltage and decrease the hole density as well as the channel conductance of the OECTs (21, 27). The injection of cations into the PEDOT:PSS film can be regarded as a dedoping process of the active layer. The dedoping process can be described with the electrochemical reaction as follows (27)

$$n(\mathrm{PEDOT}^+:\mathrm{PSS}^-) + \mathrm{M}^{n+} + n\mathrm{e}^- \underset{\text{Oxidation}}{\overset{\text{Reduction}}{\rightleftharpoons}} n\mathrm{PEDOT}^0 + \mathrm{M}^{n+}:n\mathrm{PSS}^- \quad (1)$$

where $M^{n+}$ denotes a cation from the electrolyte, $n$ is the number of charge of the cation, $e^-$ is an electron from the source electrode. The migration of $M^{n+}$ to the polymer leads to the reduction of PEDOT, which is from the oxidized state ($PEDOT^+$) to the neutral state ($PEDOT^0$). When the gate voltage is removed, $M^{n+}$ diffuses back into the electrolyte and PEDOT recovers to the oxidized state.

The device characteristics of OECTs are sensitive to the density of cations in the electrolyte. Figure 2a shows the transfer characteristics ($I_S-V_G$, $V_{DS} = -0.1$ V) of an OECT measured in KCl aqueous solutions with different concentrations. It can be found that the transfer curve shifts to lower gate voltage with the increase in the concentration of KCl. It is worth noting that all transfer curves can be shifted horizontally to the right and merged into a universal curve, as shown in the inset of Figure 2a. Therefore different concentration of KCl results in a change in effective gate

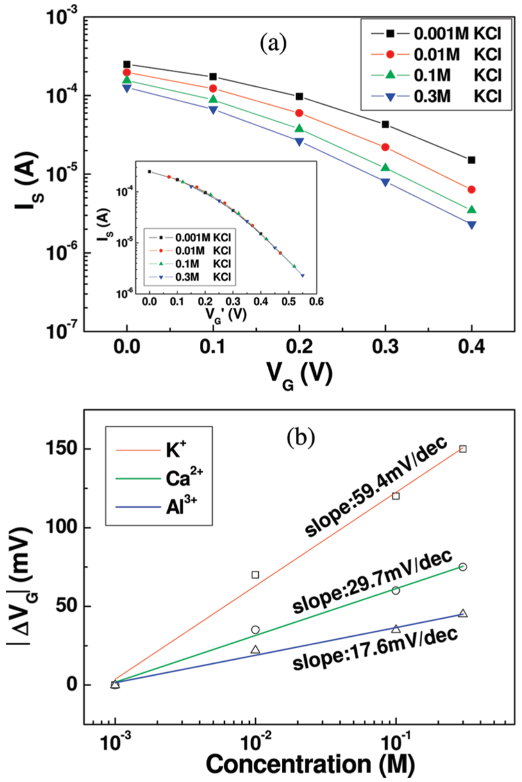

**FIGURE 2. (a) Transfer characteristics ($I_S-V_G$, $V_{DS} = -0.1$ V) of an OECT measured in KCl solutions with different concentrations. Inset shows transfer characteristics of the OECT where the curves are scaled in *x*-axis to yield a universal curve. The extent of the shift of the gate voltage is dependent on the concentrations of KCl solution. (b) Dependence of $|\Delta V_G|$ on the concentration of cations in the aqueous solutions of KCl, $Ca(NO_3)_2$, and $Al_2(SO_4)_3$.**

voltage, which will be discussed in more details later. Similar effect has been observed for other cations ($H^+$, $Na^+$, $Ca^{2+}$, and $Al^{3+}$).

Figure 2b shows the relative change of the gate voltage $|\Delta V_G|$ as functions of the concentration of different cations ($K^+$, $Ca^{2+}$, and $Al^{3+}$). It can be found that $|\Delta V_G|$ shows a logarithmic dependence on the concentration of the electrolyte. The slope obtained in KCl solution is 59.4 ± 0.8 mV/decade, which is very close to the theoretical value (59.2 mV/decade, $n = 1$, temperature is 300 K) given by the Nernst equation. Similar results can be obtained in HCl and NaCl solutions. The response of the device to bivalent or trivalent cations also indicates near-Nernstian dependences, as shown in Table 1 and Figure 2b.

Next, we will discuss the mechanism for the ion-sensitive behavior of the OECTs. The channel current of an OECT is given by the following equation (9, 21):

$$I_{DS} = \frac{q\mu p_0 tW}{LV_p}\left(V_p - V_g^{eff} + \frac{V_{DS}}{2}\right)V_{DS} \quad (\text{when}|V_{DS}| \ll |V_p - V_g^{eff}|)$$
$$V_p = qp_0t/c_i$$
$$V_g^{eff} = V_G + V_{offset} \tag{2}$$

where $q$ is electronic charge, $\mu$ the hole mobility, $p_0$ the initial hole density in the organic semiconductor before the application of a gate voltage, $t$ the thickness of the organic semiconductor film, $W$ and $L$ the width and length of the OECT device, respectively, $V_p$ the pinch-off voltage, $V_g^{eff}$ the effective gate voltage applied, and $V_{offset}$ an offset voltage related to ion concentration (9). It is worth noting that $c_i$ is the effective capacitance per unit area of the transistor, which is not only related to the capacitance of the interface between the electrolyte and organic semiconductor but also the capacitance of the interface between the electrolyte and the gate electrode. We consider that $c_i$ is approximately equal to the total capacitance of the two interfaces connected in series divided by the area of the channel.

**Table 1. Change of the Effect Gate Voltage Induced by the Change of Cation Concentration for One Order of Magnitude**

| | slope (mV/decade) | |
|---|---|---|
| cations | experimental value | theoretical value |
| $H^+$ | 64.2 ± 2.2 | 59.2 ($n = 1$) |
| $K^+$ | 59.4 ± 0.8 | 59.2 ($n = 1$) |
| $Na^+$ | 57.7 ± 1.7 | 59.2 ($n = 1$) |
| $Ca^{2+}$ | 29.7 ± 1.2 | 29.6 ($n = 2$) |
| $Al^{3+}$ | 17.6 ± 1.5 | 19.7 ($n = 3$) |

For Ag/AgCl gate electrode, the electrode potential is constant in different electrolytes and thus the offset voltage is mainly related to the potential applied on the interface (ionic double layer) between the electrolyte and the PEDOT:PSS layer. Although the PEDOT:PSS film is porous, a double layer on the surface still exists since the PEDOT:PSS film is insoluble in aqueous solution. However, the effective area of the double layer is much bigger than the physical area of the film and thus the PEDOT:PSS film has been used to modify electrodes for super capacitors (28).

For the electrochemical reaction shown in eq 1, the potential applied on the double layer near PEDOT:PSS is given by Nernst equation as follows

$$E_{Nernst} = E^0 + \frac{kT}{ne}\ln Q$$
$$Q = \frac{[PEDOT^0]^n[M^{n+}{:}nPSS^-]}{[PEDOT^+{:}PSS^-]^n[M^{n+}]} \tag{3}$$

where $E^0$ is a constant, $k$ Boltzmann's constant, $T$ temperature, $n$ the number of electrons transferred, $e$ the electron charge, and Q the reaction quotient. $[PEDOT^+{:}PSS^-]$, $[M^{n+}]$, $[PEDOT^0]$ and $[M^{n+}{:}nPSS^-]$ are the densities/concentrations of each species in eq 1, respectively. To achieve the same channel current of an OECT measured in electrolytes with different ion concentrations, the doping level in the PEDOT:PSS film should be kept at the same value and thus the only parameter in eq 3 that can influence the potential $E_{Nernst}$ value is $[M^{n+}]$. For higher $[M^{n+}]$, a lower $E_{Nernst}$ value is required to obtain the same channel current, which can be regarded as an increase in $V_{offset}$ in eq 1. Therefore, the offset voltage related to ion concentration is given by

$$V_{\mathrm{offset}} = -E_{\mathrm{Nernst}} = A + \frac{kT}{ne}\ln[\mathrm{M}^{n+}] \qquad (4)$$

, where $A$ is a constant. Therefore, for the same effective gate voltage $V_g^{\mathrm{eff}}$, the applied gate voltage $V_G$ decreases with the increase in the concentration of cations and shows a Nernstian relationship given by eq 4. It is worth noting that an OECT based on a different organic semiconductor may not show Nernstian relationship since the solid/electrolyte interface may have different properties.

Normally, a metal (Au or Pt) electrode instead of an Ag/AgCl will be used as the gate electrode of an OECT because a metal electrode can be easily fabricated, patterned and miniaturized, especially for the integration of the OECTs into a microfluidic system. Therefore Pt and Au gate electrodes have been used to study the ion-sensitive behavior of the OECTs. Figure 3a shows the transfer characteristics ($I_S-V_G$, $V_{DS} = -0.1$ V) of an OECT measured in 0.1 M KCl aqueous solutions with three different gate electrodes: Ag/AgCl, Pt and Au wires. The three electrodes were chosen to have the same surface area of 0.6 cm$^2$. However, the transfer characteristics show very big differences, which can be attributed to the different properties of the interface between the gate electrode and the electrolyte.

For Ag/AgCl electrode, there is no capacitance at the gate electrode due to faradic current at the interface. But the electrode potential (+197 mV) (29) needs to be considered since it changes the $V_{\mathrm{offset}}$ and induce a horizontal shift of the transfer curve of the OECT to lower gate voltage. For Pt and Au gate electrodes, we find that there is no charge transfer reaction (nonfaradic region) at the electrodes when the applied voltage is relatively low (<1.25 V). Therefore the double layer between the gate electrode (Pt or Au) and electrolyte can be regarded as a capacitor $C_g$, which is connected in series with the capacitor $C_d$ of the interface between PEDOT:PSS and the electrolyte, as shown in the equivalent circuit in Figure 3a. Thus the effective capacitance of the OECT is $c_i = (C_gC_d)/(WL(C_g + C_d))$. The capacitance $C_g$ is different for Pt and Au electrodes because the devices with the two different gate electrodes show big difference in transfer curves, although the two metal electrodes have the same area.

Then the ion-sensitive behavior of the OECTs with metal electrodes has been studied. Figure 3b shows the transfer characteristics ($I_S-V_G$, $V_{DS} = -0.1$ V) of an OECT measured in KCl solutions with different concentrations when a Pt wire is used as the gate electrode. The transfer curve shows a horizontal shift to lower gate voltage with the increase of ion concentration. Similarly, a logarithmic relationship between the shift of the gate voltage and ion concentration with a slope of 79.6 mV/decade has been obtained. Therefore the ion sensitivity of the OECT with the Pt electrode is larger than that with an Ag/AgCl gate electrode. Similar effect has been observed in an OECT with an Au electrode measured at the same conditions, as shown in Figure 3c. The transfer curve also shows a horizontal shift to lower gate voltage with the increase of the ion concentration in the electrolyte, which gives a sensitivity of 135 mV/decade.

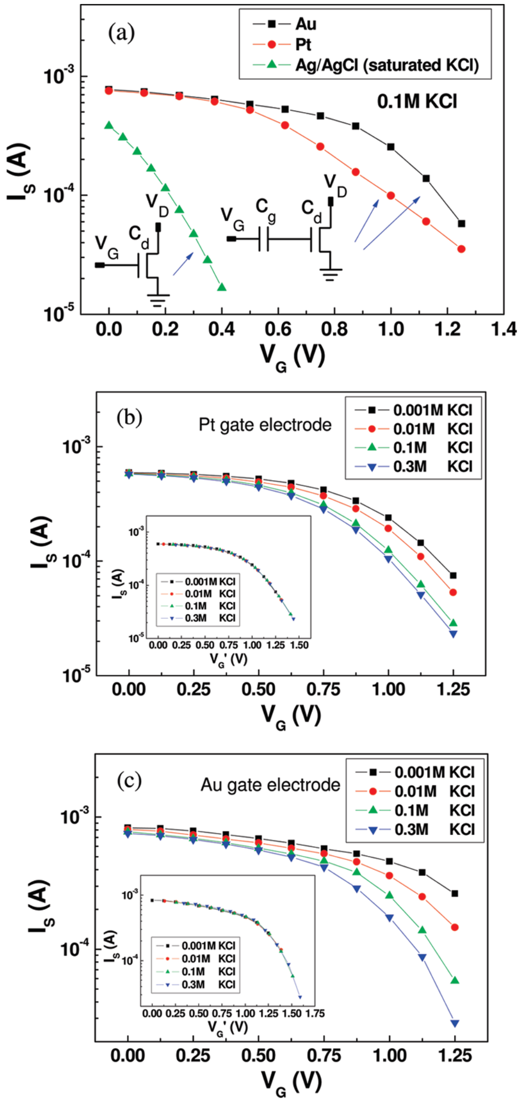

**FIGURE 3. (a) Transfer characteristics ($I_S-V_G$, $V_{DS} = -0.1$ V) of the OECT measured in KCl solutions with different gate electrodes. (b, c) Transfer characteristics ($I_S-V_G$, $V_{DS} = -0.1$ V) of OECTs with Pt and Au gate electrodes, respectively. The OECTs are measured in KCl solutions with different concentrations. Insets show transfer characteristics of the OECTs where the curves are scaled in $x$-axis to yield a universal curve.**

According to the equivalent circuit for the devices with Pt or Au electrode in Figure 3a, the gate voltage only partially applied on the interface between the electrolyte and PEDOT:PSS. Therefore higher gate voltage is needed to compensate the potential change at the interface between the electrolyte and PEDOT:PSS film induced by the change of cation concentration. As a result, the offset voltage for the device with metal gate electrode is given by

$$V_{\mathrm{offset}} = A' + (1 + \gamma)\frac{kT}{ne}\ln[\mathrm{M}^{n+}]$$

$$\gamma = C_d/C_g \qquad (5)$$

where $A'$ is a constant, $\gamma$ the ratio between $C_d$ and $C_g$. This result is very similar to OECT-based $H_2O_2$ sensor reported

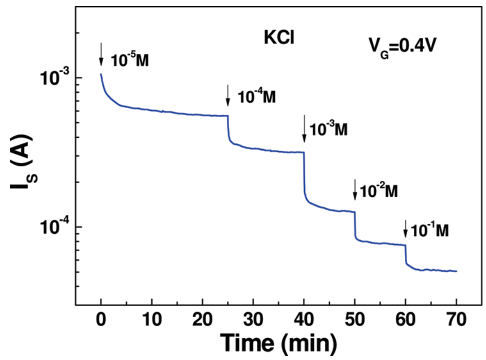

**FIGURE 4. Channel current $I_S$ of an OECT with Ag/AgCl electrode measured at the voltage of $V_G = 0.4$ V and $V_{DS} = -0.1$ V. Each step indicates the response of the channel current to an increase in KCl concentration in the electrolyte.**

by Bernards et al. (9, 22) It is worth noting that the capacitances of the two double layers, $C_d$ and $C_g$, are not constant and are dependent on the voltage applied on the double layers (26). Therefore, $\gamma$ is not a constant value if the applied gate voltage changes too much. In our experiments, $\gamma$ is approximately a constant value since the experimental results can be fitted very well with eq 5. It can be easily calculated that $\gamma$ is 0.34 and 1.3 for the two devices with Pt and Au electrode, respectively.

One convenient way to use the OECT as an ion sensor is to measure the change of channel current at constant gate voltage $V_G$ and drain voltages $V_{DS}$. Figure 4 shows that the channel current of an OECT (Ag/AgCl gate electrode) decreases with the increase of ion concentration. The device can be used for sensing the concentration of $K^+$ down to $1 \times 10^{-6}$ M. However, lower concentration cannot be detected by using this device since $H^+$ in the aqueous electrolyte will play the dominant effect on the OECT when the concentration of other cations is close to $1 \times 10^{-7}$ M. To achieve higher ion sensitivity, one feasible method is to modify the PEDOT:PSS film with an ion-selective layer, which will be studied in the future.

Silicon-based ion-sensitive field-effect transistors (ISFET) have been popularly used for pH sensors and biosensors (30, 31). The relationship between the shift of the gate voltage and the ion concentration for OECTs is very similar to that for ISFETs. But it is worth noting that the working voltage of the OECTs is much lower than that of silicon-based ISFETs (30). Therefore, the sensitivity of OECTs can be much higher than that of silicon-based ISFETs. In addition, OECTs based on PEDOT:PSS are very stable in aqueous solutions. We find that the OECTs show little change in device performance when they are continuously characterized in aqueous solutions for more than 1 week. Therefore, the OECTs based on PEDOT:PSS are suitable for the applications as highly sensitive chemical and biological sensors.

## CONCLUSIONS

In summary, the ion-sensitive mechanism of OECTs based on PEDOT:PSS has been systematically studied. The transfer curves of the OECTs shift to lower gate voltage horizontally with the increase of the concentration of cations in the electrolyte. We have found that the gate electrode of the device is critical to its ion-sensitive properties. For the device with an Ag/AgCl gate electrode, the shift of the gate voltage shows a Nernstian relationship to the concentrations of the cations. For devices with Pt and Au gate electrodes, different ion sensitivities have been obtained, which are both higher than that given by the Nernst equation. It is expected that the OECTs will find important applications in various type of sensors.

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