ANALYSIS OF STATIC BEHAVIOR OF ION SENSITIVE FIELD EFFECT TRANSISTOR FOR PH MEASUREMENTS

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ABSTRACT

The Ion-Sensitive Field-Effect Transistor (ISFET) is one of the most popular pH sensors traditionally using to measure hydrogen ion concentration (pH) of the electrolytic solutions. It is developed from Metal Oxide Semiconductor Field-Effect Transistor (MOSFET) by replacing gate electrode with an electrolytic solution to be tested, and a reference metal electrode immersed in that solution. Basic principle of ISFET operation is based on that of standard NMOS structure in conjunction with the insulator-electrolyte capacitor as described in this paper. The site-binding theory (generalized to two kinds of binding sites), together with the Gouy - Chapman-Stern model for the potential profile in the electrolyte, is coupled to the MOS physics. As a result, an approximate analytical model which completely describes static behavior of the ISFET is obtained. The developed description can serve as useful tool for understanding many contemporary biosensors based on original ISFET structure which has broad application in biomedicine, biological, chemistry and environmental areas.

Keywords: ISFET, pH sensor, MOSFET, Biosensors, Device simulation.

INTRODUCTION

In the last few decades, silicon based biosensors have found broad application in biomedical and environmental monitoring areas, high sensitive chemical detections, the diagnostic field, food industry etc. Main advantages of these biosensors over conventional ones are high sensitivity, rapid response, small size, the possibility of mass production and low cost (Bandiziol, 2015). Among a variety of types of silicon based biosensors, one of the most popular is the Ion-sensitive field-effect transistor (ISFET) based one. The ISFET was first introduced by Bergveld in the 1970 and it soon became the dominant device for measuring ion concentration (H^+ or OH^-) in the electrolytic solutions.

Generally, the ISFET is a type of potentiometric device that operates in a way similar to its purely electronic analogue, the MOSFET (Bergveld, 1981). Namely, the structures of these two transistors are identical except that metal gate in the ISFET is not immediate to the insulating layer. Instead, the gate is replaced by an electrolytic solution to be tested, with a reference metal electrode immersed in this solution. The reference electrode acts as a gate terminal allowing the ISFET to be biased in the same way as the standard MOSFET. When the ISFET is placed in an electrolyte, due to the interaction between the surface of the insulator and hydrogen ions in the electrolyte, a charge layer is created on the insulator surface and potential ψ_0 is generated at the electrolyte -insulator (EI) interface (Massobrio, 1991). Consequently, the threshold voltage and conductivity of the MOSFET channel are changed, and hence the current flowing through the channel. Therefore, to the ISFET could be applied same equations as for MOSFET with exception of the threshold voltage equation which had to be modified in order to include the effect of the EI interaction. In addition, pH sensitivity of the ISFET biosensor can be explained by examination of the effect of pH value of the electrolyte on the charge and potential distributions above the gate insulator (Si, 1979).

Over time, there have been outstanding advances in the modification of basic ISFET structure in accordance with various bio-sensing researching. For example, an enzymatically modified ISFET has been developed for the direct detection of penicillin (Caras, 1980), the enzyme-immobilized FET for detection of hydrogen ion concentration (Lee et al., 2009) and the DNA - modified FET based on deoxyribonucleic acid hybridization detection (Gasparyan, 2019), etc.

In this paper the ISFET is considered as a combination of the electrolyte - insulator capacitor and the standard NMOS transistor. For analyzing the potential changes at the EI interface a site-binding model was used. On the other side, the Gouy-Chapman-Stern model was used for description of the potential profile in the electrolyte. These two models are coupled to the MOSFET physics in order to explain the sensitivity of the ISFET device to hydrogen ion concentration in electrolytic solution. In this way, complete description of the ISFET static behavior is achieved.

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PHYSICS

OPERATING PRINCIPLE OF ISFET

The MOSFET is a basic building block of modern electronics. It has four terminals: source (S), gate (G), drain (D) and body (B). However, in many practical cases the body terminal is in connection with the source one thus forming a three-terminal device such as a field-effect transistor (Kevkić, 2018).

The abbreviation MOS refers to the three-layer transverse structure of the device. Namely, the substrate consists of a semiconductor, usually silicon p or n type. A thin insulating layer of silicon dioxide, briefly called gate insulator, is applied to the surface of the substrate. A metal gate electrode is placed above the insulator having the role of a control electrode. A positive voltage applied to the gate of MOSFET with p substrate generates a transverse electric field which leads to the repulsion of the holes present under the gate insulator. That results in creation of a depletion region which is populated by the bound negative charges. Simultaneously, the positive gate voltage pulls electrons from the substrate into the surface region under the gate insulator. When sufficient electrons are induced there, the substrate surface is inverted from p-type to n-type creating the thin inversion layer, i.e. the conduction channel. The gate voltage at which the channel forms is so-called the threshold voltage. Further, by applying voltage between the drain and source terminals V_{DS}, the current flows freely through the channel and its amount is controlled by the gate voltage (Kevkić, 2016; Kevkić 2018).
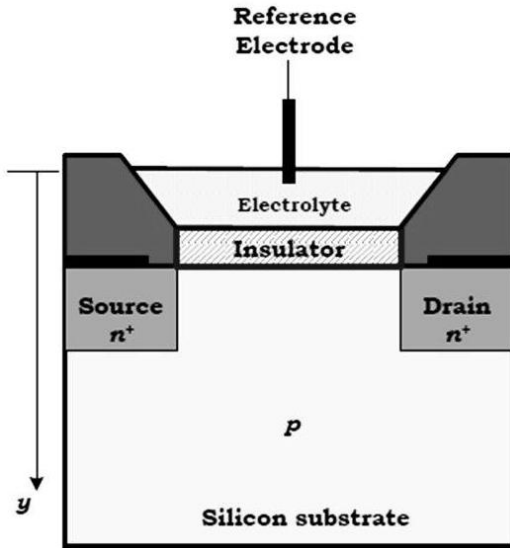

Fig. 1. Illustration of the ISFET structure.

The relative simple MOSFET structure has been served as a base for construction of the ISFET one which cross-section is schematically illustrated in Fig. 1. From figure is obvious that metal electrode in ISFET becomes a remote gate which is exposed to an electrolytic solution together to the gate insulator. The charge distribution at the EI interface is affects by any change of the hydrogen ion concentration of the solution. That change can be a consequence of the interaction of the gate insulator with the ions in the solution, as well as a horizontal electric field in the electrolyte when the voltage V_{DS} is applied (Tarasov, 2012).

In following will be described that basic mechanism of a pH-sensitive ISFET operation consists in the change of potential between the electrolytic solution and the gate insulator surface. As a consequence of that change the output current of ISFET I_d is increasing or decreasing. That is why, during the pH measurement, at a fixed input voltage at reference electrode of ISFET the change in I_d is observed. Therefore, the sensitivity of the ISFET device is relating to change in the output current, i.e. it can be expressed as I_d change per pH unit change (Heidari, 2018).

THE ISFET CONCEPT

During normal operation, the ISFETs as well as MOSFETs are usually biased in non-saturated mode. In this mode the threshold voltage V_T exhibits a linear relation with drain current (Dutta, 2012):

$$I_d = C_{ox}\mu\frac{W}{L}\left[(V_{GS} - V_T)V_{DS} - \frac{V_{DS}^2}{2}\right] \quad (1)$$

where C_{ox} is the gate insulator capacitance per unit area, μ is the effective surface mobility, W and L are the channel width and length, respectively, V_{GS} and V_{DS} are the gate-to-source and drain-to-source voltage, respectively.

The general threshold voltage of basic MOSFET is given by:

$$V_T = V_{FB} + 2\phi_F - \frac{Q_{dep}}{C_{ox}} \quad (2)$$

Here V_{FB} is flat-band voltage, Q_{dep} is the semiconductor surface depletion region charge density, ϕ_F is the Fermi potential of bulk silicon p-type given by $\phi_F = u_T \ln N_A/n_i$ where N_A is the doping acceptor concentration, n_i is the intrinsic carrier concentration in bulk, $u_T = kT/q$ is the thermal voltage. If we take into account relation for V_{FB}, the Eq. (2) can be rewritten in following form:

$$V_T = \phi_M - \phi_S + 2\phi_F - \left(\frac{Q_{ss} + Q_{ox} + Q_{dep}}{C_{ox}}\right) \quad (3)$$

Where ϕ_M and ϕ_S are the work function difference of the metal and semiconductor respectively, Q_{ss} is the surface state density at the substrate, Q_{ox} is fixed oxide charges.

For an ISFET fabricated on the same substrate and by using the same technology, since the electrolyte is in direct contact with the insulator, the flat band voltage changes due to the chemical changes that occur at the EI interface. Therefore, the expression for the threshold voltage of ISFET $V_T^{'}$ should include also terms which reflect the interfaces between the electrolyte and the gate insulator as well as the electrolyte and the reference electrode. Thus the threshold voltage of the ISFET is given by (Ytteralal, 2003):

$$V_T^{'} = \left(V_{ref} + \psi_{ij}\right) - \left(\psi_0 - \chi_{sol}\right) + V_T - \phi_M \qquad (4)$$

where V_{ref} - the reference electrode potential with respect to vacuum and it is constant; ψ_{ij} - the potential difference between reference solution and electrolyte with typical value of 3 mV; χ_{sol} - surface dipole potential of the solution which is also constant and ψ_0 - potential of the electrolyte-insulator interface which is usually governed by the dissociation and association of the oxide surface groups (Bard, 1980).

According to Eq. (4), the threshold voltage of an ISFET is the sum of the threshold voltage of the MOSFET and the chemical part in which all terms are constant except ψ_0 which represents a chemical input parameter that depends on pH of electrolytic solution. It is clear that the main goal in ISFET applications is to represent the relation between ψ_0 and pH of the solution. In that purpose is utilizing the site- binding model considering silanol and amine groups as two possible kinds of binding sites (Bergveld, 2003).

Let us point out that for an ISFET, gate to source voltage V_{GS} is kept constant while the threshold voltage contains the variable input signal. On the other side, in the case of MOSFET, V_T is assumed to be constant and V_{GS} is the variable. However, in the basic electronic conception this difference will give no complications, because the so-called effective voltage defined as $V_{GS} - V_T$ can be seen in both cases as the actual input variable.

SITE – BINDING THEORY AND ELECTRICAL DOUBLE LAYER

Due to relative large radiuses and thermal motion, the ions of the dissolved species of the electrolyte cannot approach to the insulator surface (Yates, 1974). Opposite, hydrogen ions H^+ can approach to the surface of the insulator because they are small and not hydrated by water molecules. These ions can be accepted by amphoteric $Si - OH$ sites that exist at the surface of the sensing SiO_2 insulator. As a result, the $Si - OH$ sites became protonated, positively charged surface sites, i.e. $\left[Si - OH_2^+\right]$. On the other side, the surface amphoteric

sites can also donate H^+ ions to the electrolyte becoming deprotonated, negatively charged ones $\left[Si - O^-\right]$. The exchange of H^+ ions between the electrolyte and the reactive surface sites can be described by following chemical reaction's equations (Bandizol, 2015):

$$\begin{aligned} Si - OH &\xleftarrow{K_a} Si - O^- + H_s^-; \\ Si - OH + H_s^+ &\xleftarrow{K_b} Si - OH_2^+ \end{aligned} \qquad (5)$$
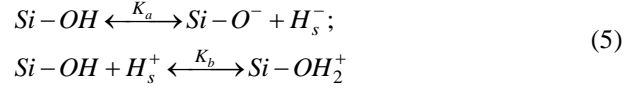
Here K_a and K_b are respectively the acidic and basic equilibrium constants of the surface reactions, given by:

$$K_a = \frac{\left[Si - O^-\right]\left[H_s^+\right]}{\left[Si - OH\right]}; K_b = \frac{\left[Si - OH_2^+\right]}{\left[Si - OH\right]\left[H_s^+\right]} \qquad (6)$$

Where $\left[H_s^+\right]$ is the surface concentration of hydrogen ions. It is related to the bulk concentration $\left[H_B^+\right]$ through the Boltzmann relationship given by:

$$H_s^+ = H_B^+ \cdot \exp\left(-\frac{\psi_0}{u_T}\right) \qquad (7)$$

Since the pH of the solution is $-\log H_B^+$, taking the minus logarithm of both sides of equation (7) results in key equation for formulating the relation between the ψ_0 to changes in the bulk pH:

$$pH_s = pH_B + \frac{\psi_0}{2.3 \cdot u_T} \qquad (8)$$

According site – binding theory, the reactions of the ions present in the electrolyte and positively or negatively charged active sites present at the insulator surface result in the change of the total value of the active site charge at the insulator surface. This further means that insulator surface is charged with the surface charge density σ_0 which depends on the ion concentration in solution, i.e. on pH value of the solution. Due to charge neutrality, σ_0 is balanced by an equal but opposite charge σ_{dl} in the electrolyte. The charge σ_{dl} originated from ions which form the electrical double layer. According to the Gouy-Chapman-Stern model, the double layer which is formed at the vicinity of the EI interface consists of the Stern layer and the Gouy-Chapman diffuse layer (Tarasov, 2012; Lee, 2009). The Stern layer contains the adsorbed ions and is further divided into Inner Helmholtz Plane (IHP) and Outer Helmholtz Plane (OHP). The name of these planes originated from the Helmholtz condenser model used as a first approximation of the double layer which is very close to the interface. The IHP comprises of the counter ions specifically adsorb on the EI interface. The potential drop in IHP is

considerably sharp depending on the ions concentration present there. On the other side, the OHP is plane that passes through the centers of the hydrated non-specifically adsorbed ions at their distance of closest approach to the solid (Nakamura, 2011). The potential drop in OHP is less steep than in the IHP. Beyond the OHP the Gouy-Chapman diffuse layer extends into bulk of solution to the distance termed as Debye length, and represents the point to which the effect of the sensing surface is felt by the ions in the electrolyte.

It is clear that double layer may be considered as two parallel plate capacitors with equivalent capacitance given by:

$$C_e = \frac{C_{st} + C_{dl}}{C_{st} \cdot C_{dl}} \tag{9}$$

here C_{st} is constant capacitance called Stern capacitance and C_{dl} is the Gouy-Chapman diffuse layer capacitance. The value of C_{st} is 20 µF/cm², while C_{dl} is an order of magnitude greater than that. On the other side, the capacitance of the insulator layer C_{ox} is very low so the series of capacitances C_{st}, C_{dl} and C_{ox} results in a lower equivalent capacitance of the overall electrolyte-insulator-semiconductor (EIS) structure. As it is mentioned above, for electrolyte-insulator system holds the charge neutrality equation:

$$\sigma_0 = -\psi_0 C_e = -\sigma_{dl} \tag{10}$$

MODELING ELECTROLYTE – INSULATOR - SEMICONDUCTOR (EIS) STRUCTURE

Based onsite – binding theory and the Gouy-Chapman-Stern model, Bousse et al have modeled EIS system by introducing two parameters, pH_{pzc} and β (Hazarika, 2017). The parameter pH_{pzc} represents the point of zero charge that means the electrical neutral insulator surface. In this case the number of positive $\left[Si-OH_2^+ \right]$ and negative $[Si-O^-]$ surface sites per unit area must be equal, and according to Eq. (6) and Eq. (7) we can get:

$$\left[H_S^+ \right] = \left[H_B^+ \right] = \sqrt{\frac{K_a}{K_b}} \tag{11}$$

Taking the minus logarithm of both sides of Eq. (11) we can obtain:

$$pH_{pzc} = -log\left(\sqrt{\frac{K_a}{K_b}} \right) \tag{12}$$

The second parameter β is the buffer capacity defined as the quantity of a strong acid or strong base that is added to one liter of the solution for changing it by one pH unit. For better understanding, buffer is a compound that resists change in pH when a limited amount of acid or base is added to it. Based on that, the β can be expressed as the ratio of the change in the number of the charged surface groups to the change in the pH_s:

$$\beta = \frac{d[B]}{dpH_S} \tag{13}$$

where $[B] = -\sigma_0 / q$.

The resulting equation for the potential drop ψ_0 between the electrolyte and the surface of the insulator, for relative large value of β is given by:

$$\psi_0 = 2.3 u_T \frac{\beta}{\beta+1} \left(pH_{pzc} - pH \right) \tag{14}$$

In the case where the pH of the bulk solution is different from pH_{pzc} which usual value is 3, the insulator surface responses to that difference indicating how pH sensitive it is. Further, by using Eq. (10) for charge neutrality of EI system the effect of a small change in the surface pH on the change in the potential ψ_0 can be expressed as:

$$\frac{\partial \psi_0}{\partial pH_s} = \frac{\partial \psi_0}{\partial \sigma_0} \frac{\partial \sigma_0}{\partial pH_s} = -\frac{q\beta}{C_e} \tag{15}$$

By combining Eq. (15) with Eq. (7) the general expression for the pH sensitivity of insulator surface and, therefore ISFET device is obtained in following form:

$$\frac{\partial \psi_0}{\partial pH_B} = -2.3 \frac{kT}{q} \cdot \alpha \tag{16}$$

with

$$\alpha = \left(2.3 \frac{kTC_e}{q^2\beta} + 1 \right)^{-1} \tag{17}$$

The parameter α is a dimensionless sensitivity parameter of the ISFET gate insulator to pH of the bulk solution. Value of α varies between 0 and 1, depending on the β and the effective capacitance C_e. If $\alpha = 1$, the ISFET has a Nernstian sensitivity which is also the maximum achievable sensitivity (Lowe, 2015; Pijanowska, 2005).

ISFET STATIC MODEL

In Fig. 2 is shown the potential distribution in the EIS system along the y direction, normal to the interfaces. Here, by ψ_d, ψ_0 and ψ_s are denote the electric potentials at the edge of diffuse layer (OHP), at the electrolyte - insulator interface, and at the semiconductor - insulator interface, respectively.
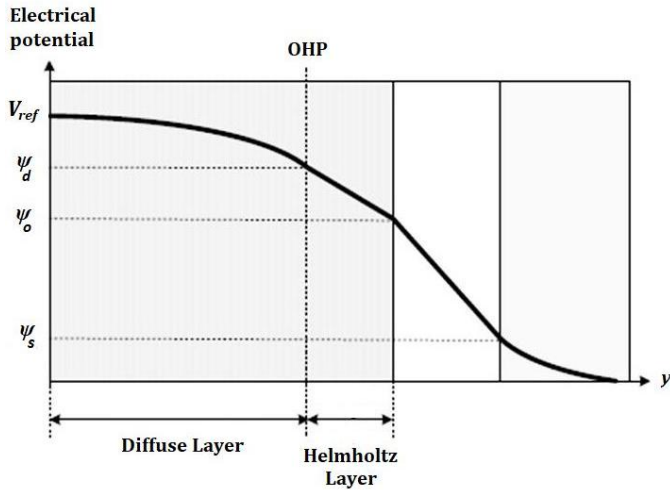
Fig. 2. Potential distribution in an EIS system along the direction normal to the interface.

Equation of the charge neutrality of the EIS system can be expressed by following sum:

$$\sigma_0 + \sigma_{dl} + Q_s = 0 \qquad (18)$$

where σ_0 and σ_{dl} are the charge on the electrolyte - insulator interface and through the diffuse layer, respectively, while Q_s is the charge density in the semiconductor and is given by:

$$Q_s = \pm\sqrt{(2\varepsilon_s kTp_0)} \times$$
$$\left[\frac{\psi_s}{u_T} - 1 + exp(-\frac{\psi_s}{u_T}) + \frac{n_0}{p_0}(-\frac{\psi_s}{u_T} - 1 + exp\left(\frac{\psi_s}{u_T}\right))\right]^{\frac{1}{2}} \qquad (19)$$

Here ε_s is dielectric permittivity of semiconductor; p_0 and n_0 are the equilibrium concentrations of holes and electrons, respectively. Sign „plus" is taken for $\psi_s \prec 0$ and "minus" for $\psi_s \succ 0$.

Otherwise the charge density Q_s is related to ψ_0 and ψ_s through Gauss law (Passeri, 2015):

$$Q_s = C_{ox}\left(\psi_s - \psi_0\right) \qquad (20)$$

On the other hand, using a Gaussian surface which encompasses the charge σ_{dl} and passing through the Stern layer it holds (Wu, 2015):

$$\sigma_{dl} = C_{st}\left(\psi_s - \psi_0\right) \qquad (21)$$

Further, if the Stern layer and the diffuse layer are considered as two capacitors in series, the potential drop ψ_0 between the solution and the insulator surface can be expressed as:

$$\psi_0 = \frac{\sigma_0 C_{st} C_{dl}}{C_{st} + C_{dl}} \qquad (22)$$

where C_{dl} is the diffuse layer unit-area capacitance given by:

$$C_{dl} = \left(\frac{q^2 c_0 \varepsilon_w \varepsilon_0}{kT}\right)^{1/2} \qquad (23)$$

here ε_w is dielectric constant of water and c_0 is the solution concentration.

Also, relationship between charge σ_{dl} and potential ψ_d can be obtained by solving the Poisson equation in the diffuse layer, i.e.

$$\sigma_{dl} = -\sqrt{8kT\varepsilon_w\varepsilon_0 c_0}\, sinh\left(\frac{V_{ref} - \psi_d}{2u_T}\right) \qquad (24)$$

Second, by considering two kinds of the binding sites on the insulator surface the surface charge density is given by:

$$\sigma_0 = qN_s \left[\left(\frac{[H_S^+]^2 - K_a K_b}{[H_S^+]^2 + K_a\left[H_S^+\right] + K_a K_b}\right)N_{sil} + \right.$$
$$\left. + \left(\frac{[H_S^+]}{\left[H_S^+\right] + K_N^+}\right)N_{nit}\right] \qquad (25)$$

Where N_s is the total number of available surface binding sites per unit area, while N_{sil} and N_{nit} are that of silanol and primary amine sites and K_N^+ is dissociation constants for positively charged amine sites.

Combining the above equations give the potential ψ_0 as function of hydrogen ion concentration in the solution (Jiao, 2012):

$$\psi_0(H_s) = \frac{qN_s}{C_{dl}}\left(\frac{[H_S^+]^2 - K_a K_b}{[H_S^+]^2 + K_a\left[H_S^+\right] + K_a K_b}\right) +$$
$$+ 2u_T sinh^{-1}\left[qN_s S\left(\frac{[H_S^+]^2 - K_a K_b}{[H_S^+]^2 + K_a\left[H_S^+\right] + K_a K_b}\right)\right]\frac{1}{\sqrt{8kTc_0\varepsilon_w}} \qquad (26)$$

This equation together with equation (7) show that the change in the pH of electrolyte leads to the change in the surface potential ψ_0 which in turn leads to the change in the threshold voltage. In this way is obtained a set of equation that presents the base of the ISFET static model with two kinds of binding sites.

RESULTS AND DISSCUSION

In this paper is consider an ISFET pH sensor with channel length $L = 1.3 \cdot 10^{-6} \, m$; channel width $W = 10^{-6} \, m$ and effective insulator thickness $t_{ins} = 4 \cdot 10^{-8} \, m$. The concentration of acceptor atoms in region of channel is $N_A = 2 \cdot 10^{-18} \, cm^{-3}$. The other parameter values used for simulation are shown in Table 1.

Table 1. The parameter value used for simulation.

K_a	$15.8 \, mol/l$
K_b	$63.1 \cdot 10^{-9} \, mol/l$
C_{st}	$2 \cdot 10^{-5} \, F/cm^2$
$n_0 (inv) \approx p_0$	$2 \cdot 10^{15} \, cm^{-3}$
T	$300 \, K$
n_i	$1.43 \cdot 10^{10} \, cm^{-3}$
ε_w	50
c_0	$0.015 \, mol/l$
χ_{soi}	$3 \cdot 10^{-3} \, V$
d_{OHP}	$3 \cdot 10^{-10} \, m$
u_T	$26 \, mV$

Set of above equations was implemented in software package MATHEMATICA 11.0 to simulate the static behavior of ISFETs. Fig. 3. shows the drain current of SiO$_2$-gate ISFET when the reference electrode was exposed to different pH electrolytes. The reference voltage and the drain voltage were kept at 2 V and 0.8 V, respectively.
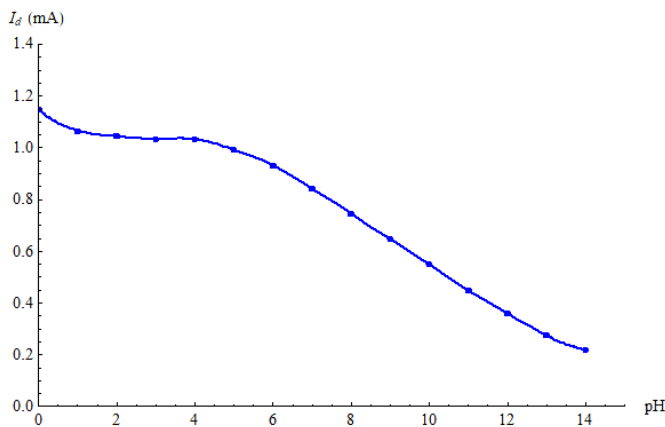

Fig. 3. The drain current of ISFET as a function of pH at $V_{ref} = 2V$ and $V_{DS} = 0.8V$.

From Fig. 3. is clear that the drain current is almost constant for pH value from 2 to 4. It means that I_d is less sensitive to that pH region, which is corresponding to the point

of zero charge pH_{pzc} at which the insulator surface is neutral, i.e. there is no net charge $(\sigma_0 = 0)$.

The drain current of considered ISFET device as a function of the applied gate (reference) voltage is presented in Fig. 4. The $I_d(V_{GS})$ curve in Fig. 4. has similar shape as at a regular MOSFET. In the other words, this curve shows that the ISFET is working same as a regular MOSFET but the threshold voltage is increased in the ISFET case what is expected in accordance with Eq. (4).
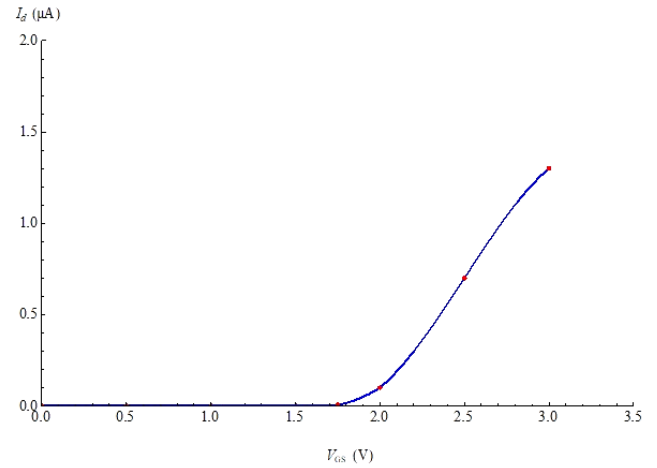

Fig. 4. The drain current of ISFET as a function of the reference electrode voltage V_{GS}.

Fig. 5. shows the output $I_d(V_{DS})$ characteristics of considered ISFET device for three different pH values of the electrolytic solution. The reference voltage V_{GS} was kept to 2.6 V. It is obvious that the drain current of ISFET device decreases such as the pH value of tested electrolyte increases.
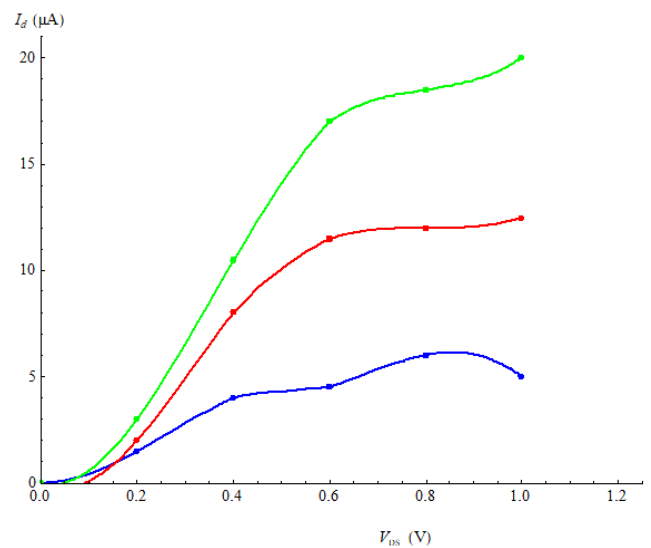

Fig.5. Drain current as a function of the drain to source voltage for pH value of 3 (green line), 7 (red line) and 11 (blue line).

CONCLUSION

In this work, the static behavior of ISFET which is using as a pH sensor is studied. The considered device includes a SiO_2-gate ISFET biased by the AgCl reference electrode, an n-channel MOSFET whose gate is controlled by a controllable external voltage. Statistical behavior of analyzing device is described by the approximate analytical model based on charge and potential equations of an electrolyte – insulator - semiconductor structure. By solving these equations in regions of semiconductor channel, the insulator - electrolyte interface and the diffuse layer respectively, the drain current related of each pH is obtained. The results obtained by implementation of the derived sets of equations in MATHEMATICA 11.0. confirm the prediction of sensitivity study that the drain current is less sensitive to pH of the electrolyte near the point of zero charge, 2<pH<4, in the case of SiO_2-gate ISFETs.

The described model can be applied to the ISFET with gate insulator and reference electrode made of the materials that differ from considered, standard case. Moreover, it can be broad to the ISFET device with appropriate selective membranes what the subject of some future paper. Further, the structure of the fundamental set of equations could be easily modified in order to describe, at the physical level, more complex phenomena.

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