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Modeling of self-assembled monolayers (SAMs) of Octadecanethiol and Hexadecanethiol on gold (Au) and silver (Ag)

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ABSTRACT

This work proposes a modeling of self-assembled monolayers (SAMs) to assess the quality of SAM layers in terms of their leaky behavior. In the modeling, electrochemical impedance spectroscopy (EIS) and a conceptual and simplified resistance-resistance-capacitance (RRC) electrical model from an electrochemical setup were demonstrated. Based on the model, Matlab-based simulation was carried out to find out the suggestive low frequency measurement for analyzing the defective monolayers. Finally, the developed electrical model facilitated the interpretation of the EIS spectra measured in the range of 10 Hz to 100 kHz and provided the capacitance, resistance and thickness of the monolayer. Thus the proposed model could be considered as a powerful tool to characterize the defective SAMs, which was very specific to determine the resistive behavior of the self-assembled monolayers prepared for shorter adsorption time.

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Introduction

The surface functionalization by molecular assembling in the applications of organic electronics has attracted enormous interest [1,2] during the past decades. The Langmuir-Blodgett (LB) or Self-assembled monolayers (SAMs) are the two promising approaches for the molecular assembling down to nanoscale [3]. Both of the routes provide the means of engineering to integrate the mono or multilayers of the functional molecules onto the electrode surface. In addition to the electrode surface modification, the formed layer either by LB or by SAMs technique also functions as passivation layer [4]. Since the films prepared by LB technique are chemically and mechanically instable and structurally disordered [5], surface functionalization by self-assembled monolayers (SAMs) are mostly preferred than LB.

Self-assembled monolayers (SAMs) provide well-ordered and densely packed films of functional molecules, which are spontaneously chemisorbed on metal surface with a controllable thickness [6] with systematic variation of SAM preparation time and concentration of adsorption solution. Since the molecules have strong affinity to metal surface, patterning of molecules is reported in the applications of organic solar cells for modifying cathode to block holes [7], interfacial electron transfer [8,9], patterning microarray or selective surface patterning for bio-sensors [3,10], and fabricating lipid bilayers on electrodes [11] among others. These applications are closely related to the blocking properties of monolayer that exhibit both the ionic and electronic transfer between the electrolyte and the metal substrate. The blocking properties are eventually deteriorated by the presence of leakage or pinholes in the monolayer. Leakage or pinholes resultant from imperfect adsorption of the self-assembling molecules can be characterized by several electrochemical methods reported [12,13]. However, works on the proper modeling of characterizing the adsorption time dependent leaky behavior of monolayer were rarely found.

In this work, we focus on the formation of time dependent and self-assembled adsorption of Octadecanethiol [$CH_3(CH_2)_{17}$-SH] (ODT), and Hexadecanethiol [$CH_3(CH_2)_{15}$-SH] (HDT) on Au and Ag surface using PET (polyethylene) substrate, which is an excellent thermoplastic polymer in the form of flexible, and semi-rigid to rigid with sufficient lightweight, depending on its thickness [14]. Because of its low-cost and scalable roll-to-roll technique with excellent water and moisture barrier properties, good thermal sta-

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bility, surface inertness, and good spin ability, flexible PET was influential to select it as substrate [15,16]. The chain $(CH_2)_n$ of ODT and HDT is responsible to function as a backbone between the linking group (—SH), which is attached with the Au and Ag metal surface and terminal group ($—CH_3$) for ODT and HDT. The stability of the monolayers on Au and Ag is dependent on the Van der Waals force functioning among the neighboring molecules of the monolayer chains, cleanliness of the Au and Ag surface [17–19] among others. SAMs of ODT and HDT block both the ionic and electronic transfers between the electrolyte solution and the metal layer of Au and Ag during of electrochemical analysis, which is a major concern for many applications. To the extensive study of the blocking properties of the adsorption time dependent monolayer by electrochemical impedance spectroscopy (EIS), a straightforward electrical modeling of the monolayers has been reported in this paper. Both the alkanethiols we selected provide functional end group of ($—CH_3$) and head group of (—SH). The —SH headgroup of the alkanethiol has strong affinity to Au and Ag surface and the methyl-terminated long-chain of alkanethiols form well-defined monolayers on Au and Ag [12,20].

Materials and methods

Chemicals and solutions

Octadecanethiol $[CH_3(CH_2)_{17}\text{-}SH]$ (ODT) and Hexadecanethiol $[CH_3(CH_2)_{15}\text{-}SH]$ (HDT) obtained from Sigma Aldrich was used without further purification. The adsorption solutions containing thiol of 5 mM were prepared in ethanol separately for ODT and HDT. An electrolyte solution of 10 mM of Tris-buffer ($C_4H_{11}NO_3$) with pH 7.4 was received from Sigma Aldrich. pH of 7.4 for Tris-buffer was selected since its non-reactive to most of the organic molecules.

Preparation of monolayer on Au and Ag on PET substrate

In the preparation of monolayer on Au and Ag, cost-effective, flexible PET substrate of 1 cm^2 was utilized. Prior to the preparation of SAM layer of alkanethiols, PET substrate properly cleaned with acetone was exposed to UV ozone cleaner to get rid of unexpected residues.

Then Au and Ag of 120 nm were sputtered on the PET substrates to prepare the metal surface for self-assembling. A SAM layer of ODT and HDT was formed by immersing the Au and Ag substrate into the adsorption solutions with systematic variation of time for 30 s, 10 min, 1 h and 1 day. Thereafter the substrates were rinsed 5 times with ethanol and water and dried by N_2 to remove the unbound and weakly bound assembling molecules from the metal surface. The preparation scheme of SAM layer of ODT and HDT on Au and Ag coated PET substrate is shown in Fig. 1. Also. the 2D topographic view of ODT and HDT SAM layer prepared for 30 s on Au is shown in Fig. 2(a) and (b) respectively. The topographic view of the AFM images shows considerably smoother surface for HDT SAM on Au than ODT, which means the higher resistive monolayer of HDT SAM than ODT on Au layer.

Preparation of PDMS mold for holding solution

Sylgard polymer and curing agent in 10:1 ratio inside a plastic container was mixed homogenously and placed inside a desiccator until the air-bubble of the mixture were gone. The degassed mixer was then transferred to a petri dish carefully avoiding any air-bubble formation and baked inside an oven for 1 h at 70 °C. Then the soft mold was taken out and allowed to cool down for a few minutes. Finally, it was shaped and punched to create hole of 5 mm diameter in the middle of the PDMS mold to hold solution during EIS experiment as shown in inset of Fig. 3(a).

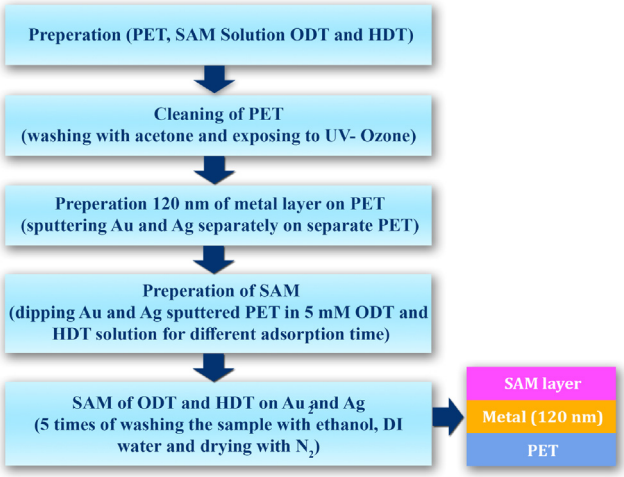


Fig. 1. Schematic flowchart of SAM preparation process.

Principles and electrical modeling

Electrochemical impedance spectroscopy (EIS) is a standard tool for analyzing interfacial impedance [21] of a thin layer. The EIS measurement provides the frequency dependent impedance data of a monolayer. A proper modeling of the electrochemical measurement setup used in EIS can quantify the capacitive and resistive value of the monolayers from the frequency dependent impedance data. The resistive and capacitive data achieved from the proper modeling favor to estimate the blocking and leaky properties of the monolayers. The schematic block diagram of the electrochemical setup for EIS [22,23] and proposed equivalent circuit to model the measurement setup has been sketched in Fig. 3.

Fig. 3(a) shows the typical electrochemical set up of lock-in amplifier (SR830 DSP) used in EIS and Fig. 3(b) and (c) show the corresponding equivalent circuits proposed. Customized PDMS mold with hole is placed on the SAM layer and is poured with electrolyte. An Ag/AgCl reference electrode has been placed in the electrolyte as shown in Fig. 3(a) to apply a 15 $mV_{p\text{-}p}$ ac signal in frequency range 1 Hz to 100 kHz to the monolayer. The complex electrical impedance $Z(\omega)$ due to the resistance and capacitance of the electrolyte and the SAM layer considered as a function of frequency is given by Eq. (1), where $\omega = 2\pi f$ with frequency f and ϕ is the phase shift between the applied voltage V and resultant current I.

$$Z(\omega) = \frac{V(\omega)}{I(\omega)} = Z_o(\omega).e^{i\theta(\omega)} \tag{1}$$

In analyzing, each component of the SAM layer is assumed in parallel to be equivalent to impedance, whereas these parallel components of SAM layer are in series additive with the electrolytes. In the case of leakage free highly blocking monolayer, electrolyte resistance ($R_{electrolyte}$) should be considered in series with capacitance of SAM (C_{SAM}) and an equivalent RC circuit could be modeled as in Fig. 3(b). Therefore, the total absolute impedance of the modeled circuit due to $R_{electrolyte}$ and C_{SAM} in series shown in Fig. 3(b) is given by,

$$Z_{total}(\omega) = abs[R_{electrolyte}(1 + 1/j.\omega.R_{electrolyte}.C_{SAM})] \tag{2}$$

Furthermore, the study of the leaky monolayer adds an additional resistance (R_{SAM}) of lower magnitude in parallel to C_{SAM} shown in the RRC model of Fig. 3(c). The contribution from the low resistive R_{SAM} to the total impedance following the RRC model could be given by,

$$Z_{total}(\omega) = abs[R_{electrolyte} + \{R_{SAM}/(1 + j.\omega.R_{SAM}.C_{SAM})\}] \tag{3}$$

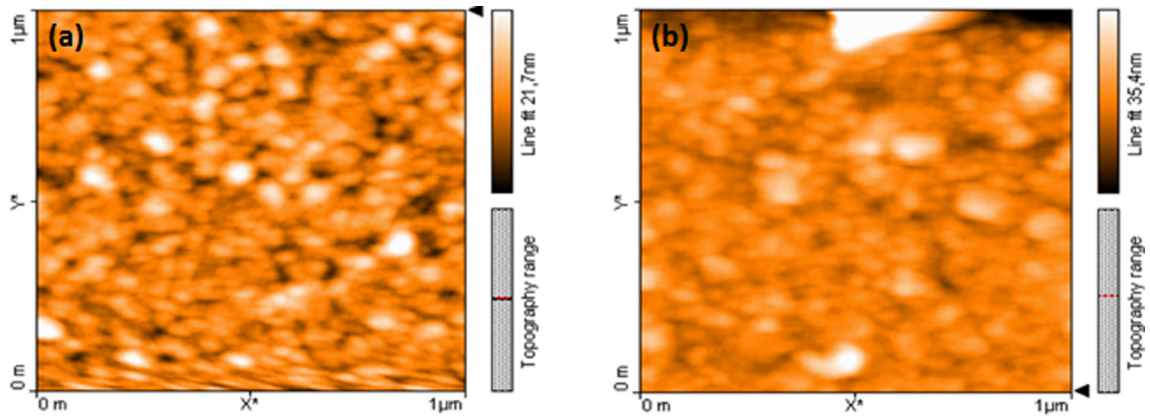
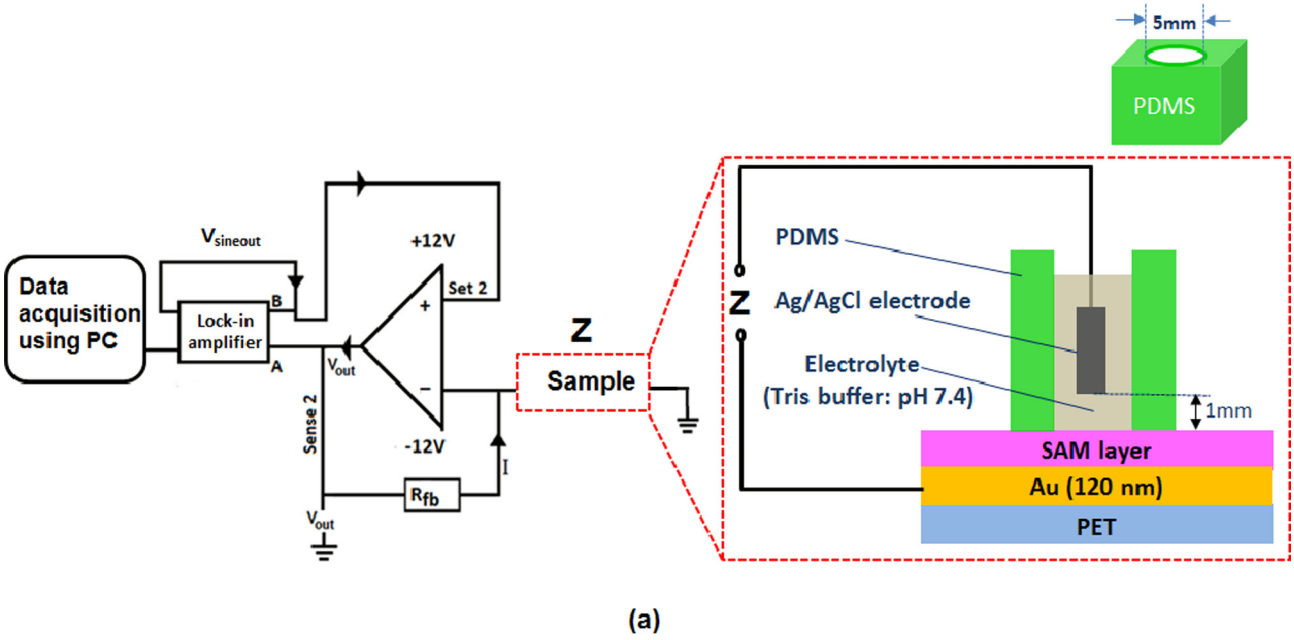


Fig. 2. AFM images of an area of $1 \times 1\ \mu m^2$ for SAM layer of (a) ODT and (b) HDT on Au.



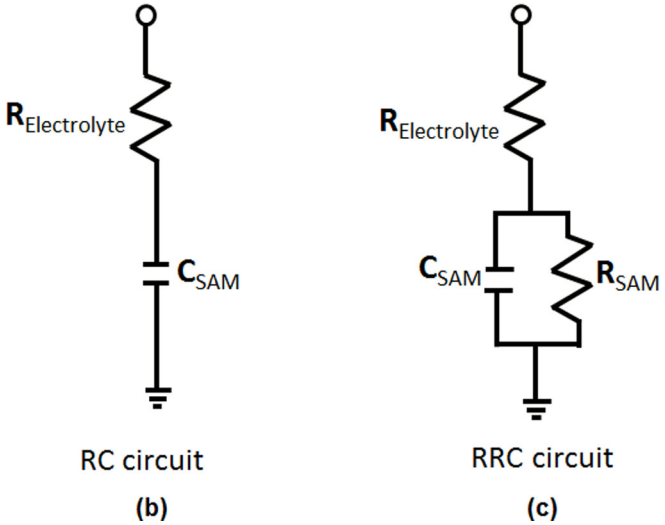


Fig. 3. (a) Schematic of measurement setup and corresponding modeled (b) RC, and (c) RRC circuits.

For the complex impedance in Eq. (2) for RC model, when frequency $\omega \to \infty$, $|Z_{total}(\omega)| \approx R_{electrolyte}$, and with frequency $\omega \to 0$, $|Z_{total}(\omega)| \approx 1/\omega.C_{SAM}$. These considerations correspond only to the C_{SAM} and $R_{electrolyte}$ at respective lower and higher frequency. To explore the leakage behavior of the monolayer studying R_{SAM}, simulation was executed for both models using Eqs. (2) and (3) approximating some reasonable values for C_{SAM}, $R_{electrolyte}$ and R_{SAM}.

Fig. 4 shows the simulated impedance spectra for both the RC and RRC models and demonstrates the contribution of C_{SAM}, $R_{electrolyte}$ and R_{SAM} to the total impedance at different frequency regimes. In impedance spectra of Fig. 4(a), the linear region at lower frequency reveals C_{SAM}, where the constant impedance at higher frequency region acknowledges $R_{electrolyte}$. The leaky behavior of the monolayer could be realized by the steady impedance in the frequency range of 10–100 Hz from the RRC model as shown in Fig. 4(b). Thus the simulation demonstrated that low frequency measurement was quite sound in sensing the leakage properties of the monolayer only using the RRC model, whereas the RC model gave information of $R_{electrolyte}$ and C_{SAM}. And it also revealed that the monolayer was highly blocking when it behaved like a capacitive surface.

Results and discussions

A series of EIS measurements were carried on the SAM layers formed by different adsorption times of ODT and HDT both on the Au and Ag substrates. Adsorption times considered were 30 s, 10 min, 1 h and 1 day. For convenience, the impedance data for the SAM layer of ODT on Au were fitted to the model shown in Fig. 3(c) using Eq. (3) and the monolayer capacitances, resistances and electrolyte resistances were obtained. The thickness of the SAM layers was estimated from the capacitance of the SAM layers obtained from the fitted data using the following relation [24,25],

$$d = \frac{\varepsilon_o \varepsilon_r A}{C} \tag{4}$$

The plot of measured and simulated impedance curve for SAM of ODT on Au for different adsorption time was presented in Fig. 5(a), where the Fig. 5(b) represented the corresponding phase data. Within the 100 to 1000 Hz of the applied signal as shown in Fig. 5(a), the impedance of C_{SAM} changed linearly. Beyond 100 Hz, the impedance was almost steady for $R_{electrolyte}$ but significant changes were marked for R_{SAM} below 100 Hz. It was noteworthy that impedance of C_{SAM} was found to increase with increasing adsorption time, which was uninterrupted for longer adsorption time of 1 h and 1 day. But below 100 Hz, notably decreased impedance for R_{SAM} was observed for 30 s and 10 min, which was again consistent for longer adsorption time of 1 h and 1 day as reported for C_{SAM}.

In Fig. 5(b), the phase that corresponds to the zero degree at higher frequencies was due to the electrolyte resistance. At lower frequencies, the interrelating phase for 1 h and 1 day adsorption time were around 90 degrees, and it was clear identification of the capacitive behavior of the SAM layer. But for the 30 s and 10 min adsorption time, the phases were inclined towards zero degree, which was due to resistive behavior SAM layer dominating over the C_{SAM}. Thus the overall study of phase spectra against the measurement frequency was subjected to the capacitive and resistive behavior of SAM layer. This indicated that the SAM layer has a tendency to form leaky monolayers on metal surface for their shorter adsorption time, which was in good agreement with time dependent impedance and literature [26,27].

The experimental data retrieved from fittings of the impedance spectra for ODT and HDT SAM layer on Au and Ag for different adsorption time were summarized as shown in Tables 1 and 2. The thickness of the SAM layer (d_{SAM}) was figured out using the corresponding values of C_{SAM}, substrate area (A) under investigation according to the Eq. (4). The relative permittivity (ε_r) value of 2.5, which is usually considered for most of the organic molecules [28,29], was used. Based on the summarized data in Tables 1 and 2, achieved C_{SAM} and d_{SAM} for ODT and HDT SAM layer on Au and Ag for different adsorption time were plotted in Fig. 6.

Fig. 6(a) and (b) plot the respective data for resistance R_{SAM} and thickness d_{SAM} of the SAM layer against the different adsorption time. The data were retrieved and estimated from fittings of the experimental impedance spectra. In the following Fig. 6, both the R_{SAM} and d_{SAM} for all of the ODT and HDT SAM on Au and Ag were found to increase with increasing adsorption time, but they became steady after 1 h. The resistance and the thickness of the SAM layer for 30 s and 10 min were smaller enough than the longer adsorption time.

This indicates that the SAMs layer formed for longer adsorption time provides thick, low-leakage and highly blocking layer of alkanethiol on metal surface than the shorter adsorption time.

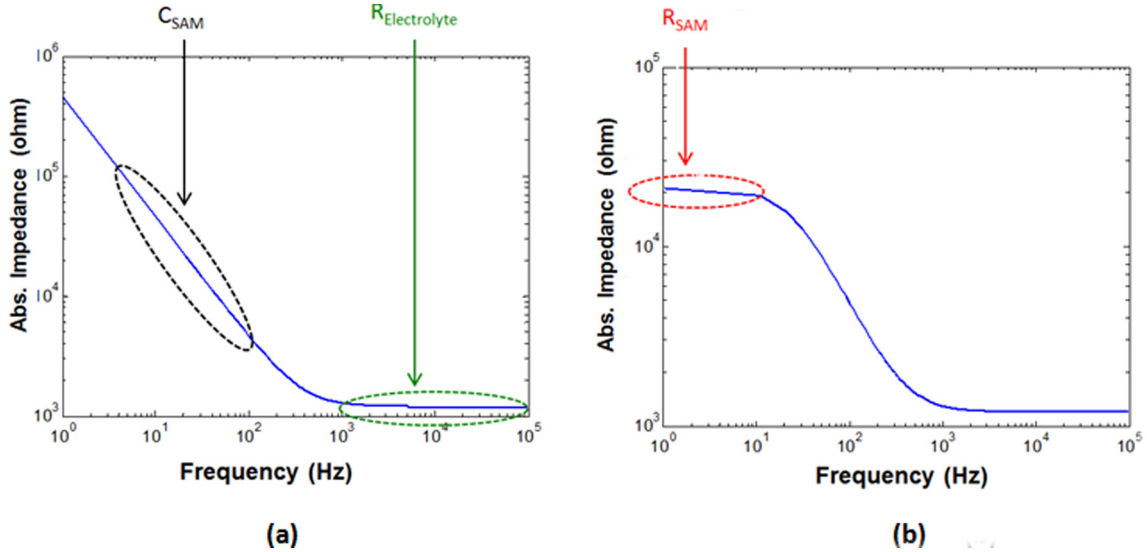


Fig. 4. Simulated impedance spectra based on the equivalent (a) RC, and (b) RRC model.

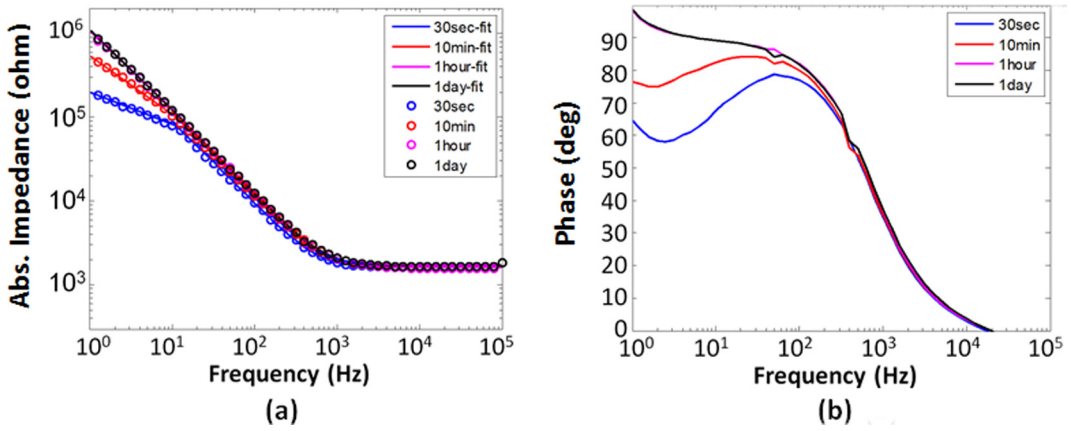


Fig. 5. (a) Measured and simulated impedance, and (b) corresponding phase spectra for SAM of ODT on Au for different adsorption time.

Table 1
Data of SAM parameters of ODT and HDT on Au substrate.

Adsorption time	ODT			HDT		
	C_{SAM} (nF)	d_{SAM} (nm)	R_{SAM} (kΩ)	C_{SAM} (nF)	d_{SAM} (nm)	R_{SAM} (kΩ)
30 s	351	1.88	90	381	1.73	330
10 min	331	1.99	260	341	1.93	350
1 h	320	2.06	780	301	2.19	470
1 day	316	2.09	800	290	2.27	550

Table 2
Data of SAM parameters of ODT and HDT on Ag substrate.

Adsorption time	ODT			HDT		
	C_{SAM} (nF)	d_{SAM} (nm)	R_{SAM} (kΩ)	C_{SAM} (nF)	d_{SAM} (nm)	R_{SAM} (kΩ)
30 s	357	1.84	350	266	2.48	250
10 min	331	1.99	800	201	3.28	850
1 h	320	2.06	820	200	3.29	950
1 day	289	2.28	840	126	5.24	1500

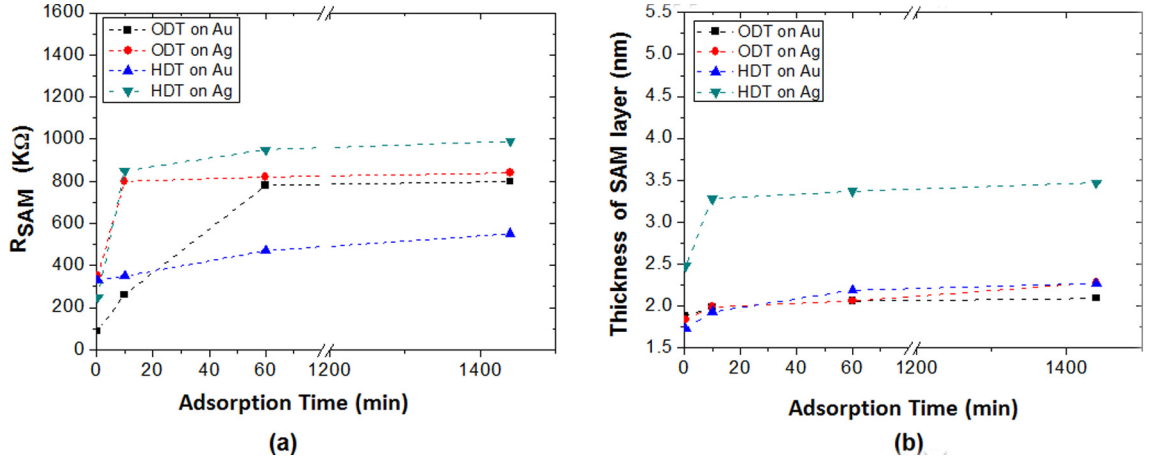


Fig. 6. (a) Resistance of SAM (R_{SAM}), and (b) thickness of SAM (d_{SAM}) for ODT and HDT SAM on Au and Ag for different adsorption time.

Quantification of the leakage, structures and stability of the monolayers of ODT and HDT of SAM on Au and Ag might be the other aspects to explore. Fig. 6(a) and (b) showed that our proposed RRC model of measuring capacitance, resistance, and thickness of SAM layers by EIS was feasible to study the blocking or leakage properties of the monolayer.

Conclusion

In summary, EIS is a useful tool to measure the complex impedance of monolayer. RRC model we proposed allows the capacitance, resistance and thickness of the SAM to be estimated from the impedance data studied by EIS. Furthermore, our RRC model

suggests a low frequency measurement to quantify the leaky behavior of the monolayer. Based on the RRC model, it is concluded that leakage and blocking properties of SAM layer is adsorption time dependent. The monolayer is extremely leaky for shorter preparation time of 30 s to 10 min. To explore the leaky behavior by EIS, it requires the measurement frequency in the range of 10 to 100 Hz. In our modeling, we find the highly blocking and highly leaky monolayers for HDT SAM on Ag and ODT on Au respectively, which is well-agreed with the topographic view of AFM image in Fig. 2. Thus, the model we propose could be used to assess the electrical properties such as resistance, capacitance and physical properties including thickness of the thin monolayers.

Conflict of interest

The author(s) declare(s) no conflict of interest regarding the publication of this paper.

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