Layer-by-layer assembly of mica and polyelectrolyte for use in low-humidity sensor

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ARTICLE INFO

ABSTRACT

Mica multilayer thin films were assembled layer-by-layer (LBL) from mica that was modified with carboxylic groups (—COOH) and cationic polyelectrolyte (poly(allylamine hydrochloride)) (PAH) on a quartz-crystal microbalance (QCM) for low-humidity sensing applications. The surface characteristics of the thin films were studied for various assembly conditions of pH and ionic strength of modified mica solutions. The thin films were characterized using QCM and by atomic force microscopy (AFM). The thickness of mica-COOH/PAH multilayer thin films increased with assembly pH of modified mica solution. When salt was added, the multilayer films became thicker, smoother and grew more regularly. The mica-COOH/PAH multilayer films that were deposited from the mica-COOH solution with salt at pH 3 was coated on the gold electrode of the QCM exhibited excellent sensitivity ($0.524 -\Delta Hz/\Delta ppm_v$ at $36.1\ ppm_v$) and linearity and a short response time (13 s at $60.4\ ppm_v$).

1. Introduction

Clay minerals, particularly smectites (such as montmorillonite and mica), have many useful properties, such as high specific surface area and excellent adsorptive capacity. They are also the most common minerals on the surface of the earth. Therefore, they have applications in adsorbents, catalysts and ion exchangers [1,2]. The latter applications have been particularly useful in the development of chemical sensors and biosensors in recent years [3–7].

Mica has been of widespread interest because of its critical role in geochemical and biological processes and sensor elements [3]. However, mica has a low surface cation exchange capacity (CEC), which is typically more than an order of magnitude smaller than that of expandable clay minerals, such as montmorillonite or vermiculite, limiting its usefulness in sensors. Therefore, the modification of the surface properties of mica is potentially useful in extending its range of applications. Many organic polymers, such as dodecyl pyridinium [8], poly(ethylene oxide) [9] and styrene [10] have been reported to modify the surface of mica via ion exchange.

Of all of the methods that have been developed for preparing thin films, the layer-by-layer (LBL) self assembly method based on sequential adsorptions of ionized polyelectrolytes and oppositely charged materials in aqueous solutions has many advantages, including simplicity, low-cost, low temperature of deposition,

controllable thickness of nanometers to the micrometers and the absence of need for any complex equipment [11–13]. Many works have reported on various nanoparticles in LBL assembled polyelectrolyte-based multilayer films, such as Au [14], Fe_3O_4 [15], α-zirconium phosphate [16], Al_{13} Kiggin ions [17], In_2O_3 [18], TiO_2 [19–22] and montmorillonite [23,24]. However, no attempt has been made to construct mica multilayer thin films by layer-by-layer self-assembly. In this study, mica modified with carboxylic groups (—COOH) were LBL self-assembled on a quartz-crystal microbalance (QCM) for low-humidity sensing application. The surface characteristics of the thin films were investigated in relation to assembly conditions, including pH and ionic strength of the modified mica solutions. The low-humidity sensing characteristics of mica-COOH/PAH multilayer films were investigated; they included the sensitivity, linearity, response and recovery times.

2. Experimental

2.1. Materials

A dispersion and exfoliated of organic modified mica (mica-COOH) particles solution was purchased from NanoMica Technology, Taiwan. Anionic poly(4-styrenesulfonic acid-*co*-maleic acid, SS:MA 1:1) sodium salt (PSSMA 1:1; Mw = 20,000) and cationic poly(allylamine hydrochloride) (PAH; Mw = 15,000) were obtained from Aldrich. All used deionized water (DIW) was prepared using a Milli-Q Millipore (Bedford, MA, USA) purification system, and the resistivity of water was above 18.0 MΩ/cm.

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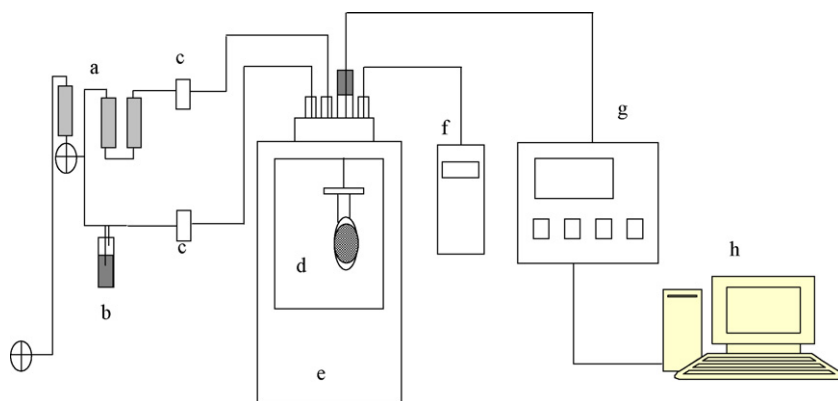*E-mail address:* spg@faculty.pccu.edu.tw (P.-G. Su).

Fig. 1. Schematic diagram of experimental set-up for the QCM sensor measurement and low humidity atmosphere controller. (a) Molecular sieve and desiccating agent; (b) water; (c) mass flow controller; (d) detecting chamber and QCM; (e) thermostat; (f) low humidity hygrometer; (g) oscillator and frequency counter; (h) PC.

2.2. Preparation of LBL mica multilayer thin films

AT-cut quartz crystal substrates with a fundamental resonance frequency of 9 MHz were obtained from ANT Technologies Corp. Negatively charged QCM substrates with gold-coated were obtained using an H_2O_2/H_2SO_4 mixture (1:2, 15 ml) that was treated and then washed using deionized water and successively cleaned ultrasonically in acetone. Before mica-COOH/PAH multilayered coating systems were constructed, two-cycle PSSMA/PAH bilayers were deposited on the substrate to improve the combinability of the surface and the first monolayer of the film of mica. A cycle PSSMA/PAH bilayer film architecture was produced by alternately depositing aqueous PAH (0.06 mM at pH 4) and PSSMA (0.05 mM at pH 2) aqueous solution onto the negatively charged QCM substrate. For each layer produced, the immersion time was about 5 min and was followed by further rinsing and drying. Therefore, a two-cycle PSSMA/PAH bilayer film was fabricated by repeating the above processes. Then, a mica multilayered thin film that comprised (mica-COOH/PAH)$_3$ was deposited on the (PSSMA/PAH)$_2$/QCM substrate in the same manner as the PSSMA/PAH multilayers.

2.3. Instruments and analysis

A divided humidity generator was used as the principal facility for producing the testing gases. The required water vapor concentration was produced by adjusting the proportion of dry and humid air generated by the divided flow humidity generator. The lowest testing point is limited by the dryness of the gas. A low humidity hygrometer (HYGROCLIP IC-3, Rotronic Inc.) which measurement range and precision are 0–100% RH and 0.1%RH, respectively, and a QCM sensor were connected to an outlet of the divided flow humidity generator, and the low humidity hygrometer was used as the reference standard for the calibration of the QCM sensor.
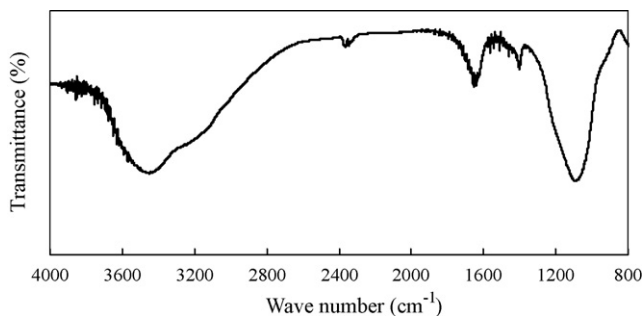

Fig. 2. IR spectra of organic modified mica particles.

The volume ratio of the moist air would be adjusted according to the reading of the low humidity hygrometer calibrating to the CMS/NML (Center for Measurement Standards/National Measurement Laboratory) humidity laboratory. The volume ratio of the moist air was calculated by the following equation:

$$\text{ppm}_v\,(\mu L/L) = \frac{V_v}{V} \times 10^6 \tag{1}$$

$$\text{ppm}_v\,(\mu L/L) = \frac{e}{P - e} \times 10^6 \quad \text{(ideal gas)} \tag{2}$$

where V_v is the volume of water vapor, V is the total volume, e is the partial pressure of water vapor and P is the total pressure. Measurement procedures were recorded as below: firstly the synthetic dry air was passed through the detection chamber until the frequency of QCM became stable and then the required water vapor concentration was flowed into the detection chamber, finally the synthetic dry air was passed through the detection chamber until the frequency of QCM recovered to its initial value. The initial vol-
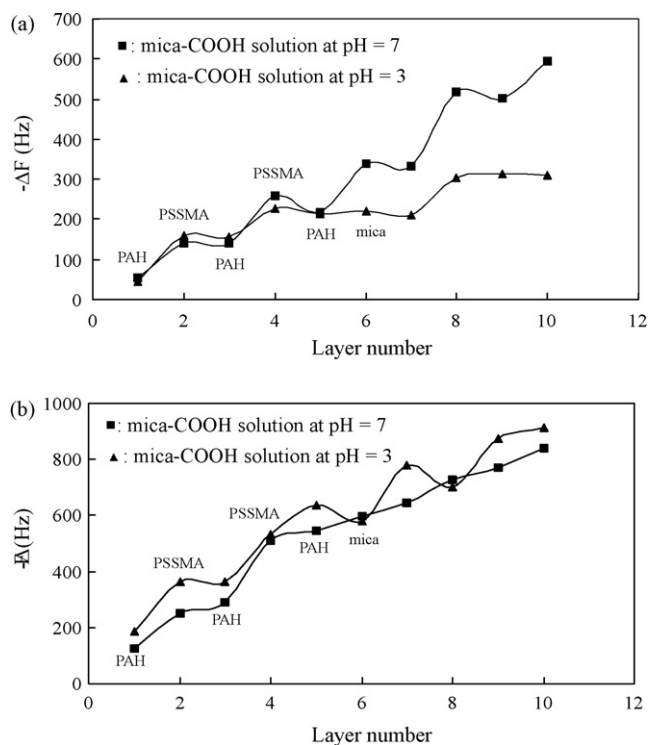

Fig. 3. Frequency shifts of mica-COOH/PAH multilayer thin films from (a) deposited solutions without salt and (b) deposited solutions with 0.5 M NaCl.
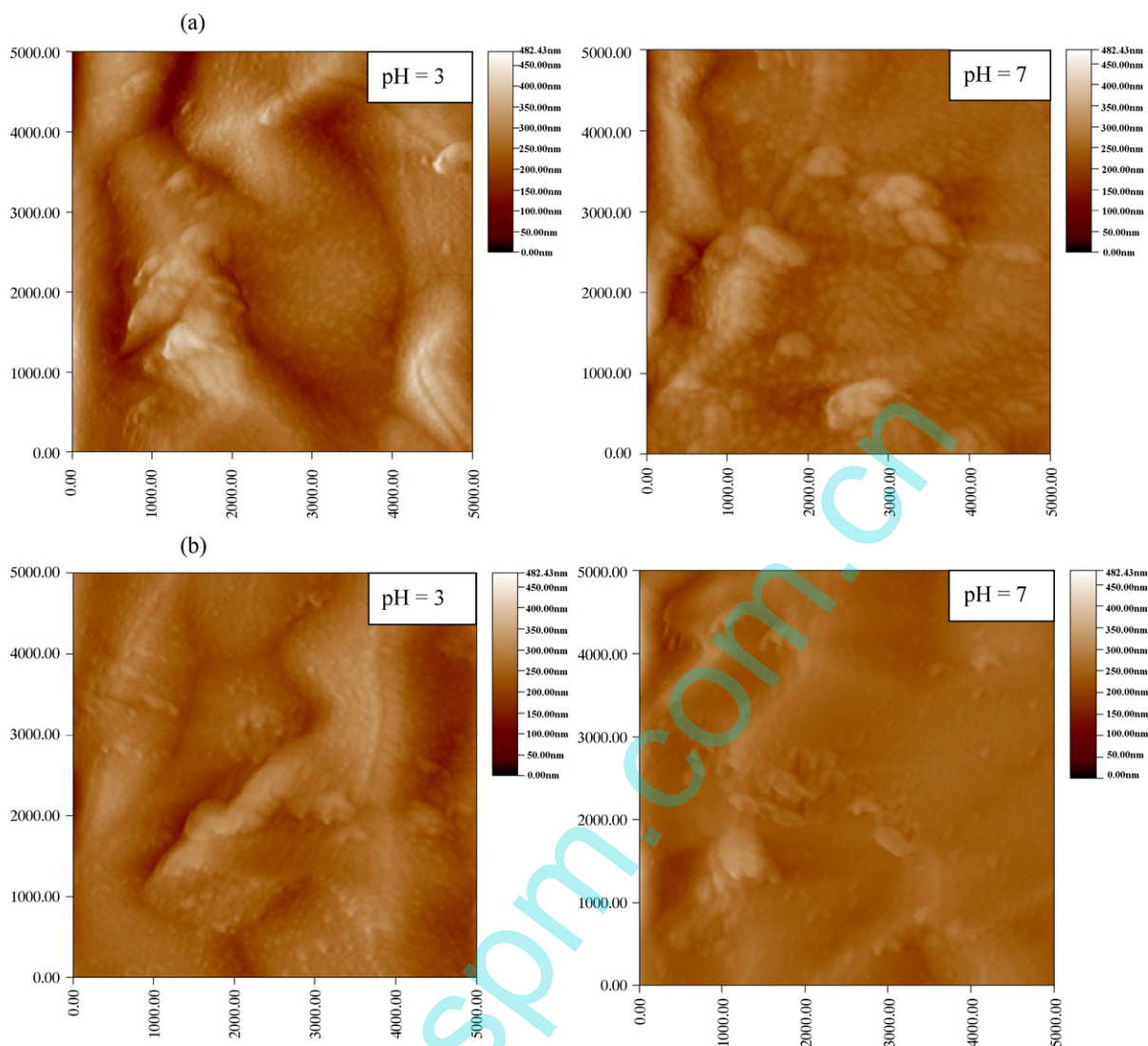
Fig. 4. AFM images of mica-COOH/PAH multilayer thin films from (a) deposited solutions without salt and (b) deposited solutions with 0.5 M NaCl.

ume ratio of the moist air was 2.9 ppm$_v$ for all experiments. The analysis system was illustrated in Fig. 1. An infrared spectrometer (Nicolet Magna-IR 380) was adopted to characterize the purchased modified mica. The surface microstructure of the thin film that was coated on a substrate was investigated using an atomic force microscope (AFM, Ben-Yuan, CSPM 4000) in tapping mode. Unless otherwise mentioned, all the measurements were performed at the room temperature about $23.0 \pm 1.5\,^\circ C$.

3. Results and discussion

3.1. IR spectra of modified mica

The purchased organic modified mica was analyzed using an FTIR spectrometer, as presented in Fig. 2. Mica (ideal formula $K_2Al_4(Al_2Si_6O_{20})(OH)_4$) is a chemically inert aluminosilicate mineral and its crystal structure consists of a negatively charged 2:1 layer of two tetrahedral sheets that sandwich one octahedral sheet. As presented in Fig. 2, the characteristic absorption peaks of the mica were the asymmetric stretching mode of O—Si—O at 1000–1250 cm^{-1} and Si—O—C at 1450–1500 cm^{-1}, respectively. Additionally, a characteristic absorption peak of the carboxylic groups modified on the surface of mica was observed at 1664 cm^{-1}

associated with the stretching vibration mode of the C=O group. The particular properties of mica-COOH depend on the particular carboxylic groups, as well as on the nature of the layered material itself.

3.2. Frequency shift of QCM

The growth of a multilayer film from PAH and mica-COOH solutions with and without 0.5 M NaCl at pH 3 and pH 7 was monitored using QCM to determine the mass adsorbed. Fig. 3(a) plots the frequency shift of the mica multilayer film that was deposited from the mica-COOH solution at pH 3 and pH 7 without salt. The mass loading increased with the pH of the mica-COOH solution. This observed growth behavior can be rationalized as follows. At high pH, more of the carboxylic acid groups in the mica became ionized, increasing the effective charge density of the mica. As the charge density of the mica increased, electrostatic attraction forces between the charged groups increased, causing the polyelectrolyte to adsorb more mica and to become thicker than at low pH. At low pH, an increasing proportion of the carboxylic acid groups become protonated, reducing the charge density of the mica, causing little adsorption to occur.

Fig. 3(b) plots the frequency shift of the mica multilayer film that was deposited from the mica-COOH solution at pH 3 and pH

Table 1
Roughness and thickness of prepared mica-COOH/PAH multilayer films in the deposition solutions containing with and without salt at pH 3 and pH 7.

	Without salt		With salt	
	pH 3	pH 7	pH 3	pH 7
RMS (nm)	53.1	29.1	35.2	21.1
Thickness (nm)	5.87	11.26	16.11	15.87

7 with salt. The mass loadings were significantly higher than when no salt was added to the mica-COOH and polyelectrolyte solutions. The increased screening of PAH and mica charges in salt-containing solutions is probably necessary to cause sufficient polyelectrolyte and mica to be adsorbed to reverse the charge [25]. Additionally, a linear film growth was observed at pH 7. This observation reveals that, when mica was adsorbed under highly charged conditions, salt is required to promote regular multilayer growth. Another possibility is that adding salt increased the proportion of the mica-COOH that was exfoliated in deposition solution at pH 7, increasing the dispersion of the mica, causing more regular multilayer growth.

3.3. Surface microstructure characteristics

The surface morphology of the mica-COOH/PAH multilayer films in the deposition solutions with and without salt at pH 3 and pH 7 was analyzed using tapping mode AFM. Fig. 4(a) shows surface images of mica multilayer film deposited from the mica-COOH solution at pH 3 and pH 7 without salt. A film deposited at pH 3 had a greater root mean square (RMS) values and larger clusters than the film deposited at pH 7. The features of the film at pH 7 were more obvious and homogeneous than those of the film deposited at pH 3, perhaps because the charge density of the mica-COOH was higher, indicating that an increased proportion of the mica-COOH was exfoliated in deposition solution, increasing the amount of mica adsorbed at pH 7. Fig. 4(b) presents the surface images of mica multilayer film deposited from the mica-COOH solution at pH 3 and pH 7 with salt. The surface roughness was significantly lower than when salt was not added in the mica-COOH and polyelectrolyte solutions. Salt probably promotes interpenetration of both polyelectrolyte and mica absorbed into earlier deposited layers, enabling more effective charge compensation and facilitating multilayer formation [26]. The film deposited at pH 7 is smoother than that deposited at pH 3, because the proportion of added salt that screened the electrostatic charges of mica-COOH solution at pH 7 was lower than that at pH 3. Table 1 presents the surface roughness and thickness of prepared mica-COOH/PAH multilayer films in the deposition solutions at pH 3 and pH 7 with and without salt.

3.4. Low-humidity sensing characteristics

Fig. 5 plots the frequency shifts of mica-COOH/PAH multilayer films in the deposition solutions at pH 3 and pH 7 with and without salt, as functions of time for various volume ratios of moist air
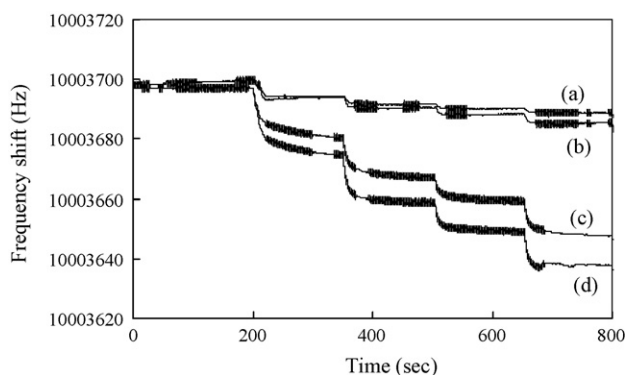

Fig. 5. Frequency shift (Hz) as a function of time (s) for various volume ratios of moist air on mica-COOH/PAH multilayer thin film from deposited solutions without salt at (a) pH 3 and (b) pH 7, and from deposited solutions with salt at (c) pH 7 and (d) pH 3.

from 37.6 to 1642.5 ppm$_v$ (0.1–5.6% RH). Table 2 presents the corresponding sensitivities. When the volume ratio of moist air was 37.6 ppm$_v$, the sensitivities of the mica-COOH/PAH multilayer films that were deposited without salt from the mica-COOH solution at pH 3 and pH 7 were 0.173 and 0.204, respectively. As described in Section 3.3, the mica-COOH/PAH multilayer film prepared at pH 7 had higher charge density than that prepared at pH 3, therefore, the adsorbed water molecules interact much most strongly (in terms of binding affinity and sticking coefficient) with the mica-COOH/PAH multilayered thin film prepared at pH 7 than that prepared at pH 3. When volume ratio of moist air was 36.1 ppm$_v$, the sensitivities of the mica-COOH/PAH multilayer films deposited from the mica-COOH solution with salt at pH 3 and pH 7 were 0.524 and 0.363, respectively. The sensitivity of mica-COOH/PAH multilayer films deposited with salt exceeded that of those without salt because polyelectrolyte/mica composite segments with salt counterions are more hydrophilic. Additionally, the mica-COOH/PAH multilayer films deposited from the mica-COOH solution at pH 3 with salt were the most sensitive because the surface roughness of the film was high, the number of active sites was high (as was the specific surface area), such that the most water vapor could be adsorbed.

Fig. 6 plots the calibration curves of the mica-COOH/PAH multilayer films in the deposition solutions at pH 3 and pH 7 with and without salt. The slope and linear correlation coefficient were calculated as shown in Table 3. For mica-COOH/PAH multilayer films, the linear sensing characteristics at the ranges of 36.1–475 ppm$_v$ and 475–1640 ppm$_v$ were shown different performance. The steep decreased in slope was observed at the volume ratio of moist air of 475–1640 ppm$_v$. The humidity sensing of ceramic and porous materials was known to mainly occur as a surface mechanism [27]. The mica-COOH on the surface of the mica-COOH/PAH multilayer films (as shown in Fig. 4), provided more active sites, presented a high local charge density and a strong electrostatic field which promoted water adsorption on mica-COOH. But, at higher volume ratio of moist air (475–1640 ppm$_v$), suggested the decreasing of active

Table 2
The sensitivity of the mica-COOH/PAH multilayer films assembled on QCM in the deposition solutions containing with and without salt at pH 3 and pH 7 for different volume ratios of moist air.

Volume ratio (ppm$_v$)	Δppm$_v$[a]	Sensitivity[b] (without salt)		Volume ratio (ppm$_v$)	Δppm$_v$[a]	Sensitivity[b] (with salt)	
		pH 3	pH 7			pH 3	pH 7
40.5	37.6	0.173	0.204	39.0	36.1	0.524	0.363
478.2	475.3	0.021	0.025	443.8	440.9	0.088	0.067
1081.9	1079.0	0.011	0.013	1081.8	1078.9	0.045	0.035
1582.8	1579.9	0.008	0.011	1642.5	1639.6	0.036	0.029

[a] The Δppm$_v$ was defined as that the each volume ratio subtract the initial volume ratio of moisture air (2.9 ppm$_v$).

[b] The sensitivity of the various sensing film was defined as $-\Delta$Hz/Δppm$_v$ [28].

Table 3
The linear sensing characteristics of mica-COOH/PAH multilayer films assembled on QCM in the deposition solutions containing with and without salt at pH 3 and pH 7 for different volume ratio of moist air.

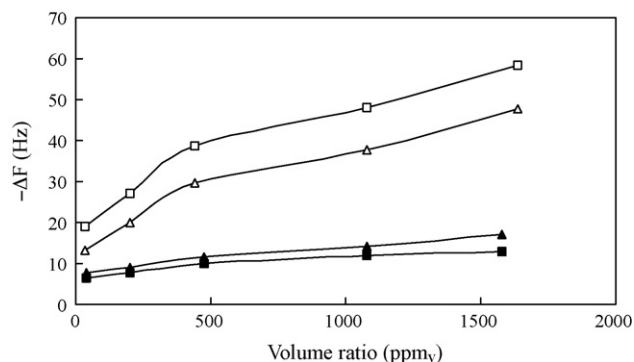|	Sensing characteristics							
	Without salt				With salt			
	36.1–475 ppm$_v$		475–1640 ppm$_v$		36.1–475 ppm$_v$		475–1640 ppm$_v$	
	Slope	Linearity[a]	Slope	Linearity[a]	Slope	Linearity[a]	Slope	Linearity[a]
pH 3	0.0082	0.9992	0.0026	0.9979	0.0489	0.9997	0.0163	0.9970
pH 7	0.0091	0.9994	0.0048	0.9884	0.0410	0.9995	0.0150	0.9905

[a] Correlation coefficient.

Fig. 6. Frequency changed ($-\Delta$Hz) as function of water vapor concentration (ppm$_v$) on mica-COOH/PAH multilayer thin films from deposited solutions without salt at (■) pH 3 and (▲) pH 7, and from deposited solutions with salt at (□) pH 7 and (△) pH 3.
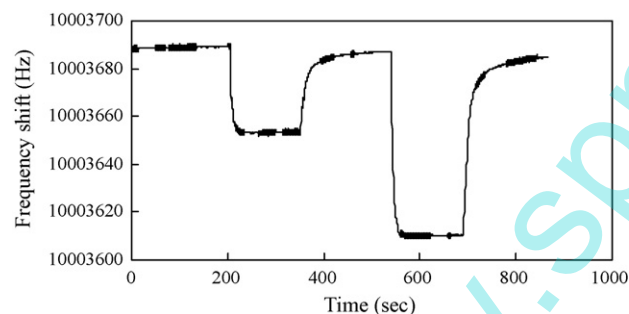

Fig. 7. Adsorption and desorption of water vapor (60.4 and 534.3 ppm$_v$) on mica-COOH/PAH multilayer thin films from deposited solutions at pH 3 with salt.

sites for reaction to occur and, hence, the steep decreased in slope was observed.

Fig. 7 plots adsorption/desorption of water vapor on the mica-COOH/PAH multilayer films that were deposited from a mica-COOH solution at pH 3 with salt which was assembled on QCM at two testing concentrations, 60.4 and 534.3 ppm$_v$. The response (RT$_{90}$)/recovery time of the mica-COOH/PAH multilayer were 13/35 s at 60.4 ppm$_v$. The adsorption and desorption of water on the mica-COOH/PAH multilayer films were reversible at two tested water vapor concentrations.

4. Conclusion

Mica-COOH/PAH multilayer thin films were assembled layer-by-layer in the deposited solutions with and without salt at various pH values and characterized using QCM and AFM. The thickness of the mica-COOH/PAH multilayer thin film increased with the pH of the mica-COOH solution because the charge density of the mica increased with pH, causing it to be adsorbed in larger amounts. The ionic strength of the deposition solutions significantly affected

mica-COOH/PAH multilayer thin film growth: multilayers were thicker, smoother and grew more regularly when the deposition solution contained salt. When salt was present in the mica-COOH solutions, multilayers deposited at pH 3 were rougher than those deposited at pH 7, because of the ionization of mica-COOH within the bulk multilayer films as the environmental pH varied.

The mica-COOH/PAH multilayer thin film was fabricated on a QCM electrode by layer-by-layer self-assembly. The sensor was extremely sensitive, especially to low humidity, with acceptable linearity, a fast response time and favorable reversibility. Therefore, the combination of a mica-COOH/PAH multilayer film with QCM is reliable for measuring humidity.

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.snb.2009.01.053.

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