Hybrid temperature effect on a quartz crystal microbalance resonator in aqueous solutions*

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The quartz crystal microbalance (QCM) is an important tool that can sense nanogram changes in mass. The hybrid temperature effect on a QCM resonator in aqueous solutions leads to unconvincing detection results. Control of the temperature effect is one of the keys when using the QCM for high precision measurements. Based on the Sauerbrey's and Kanazawa's theories, we proposed a method for enhancing the accuracy of the QCM measurement, which takes into account not only the thermal variations of viscosity and density but also the thermal behavior of the QCM resonator. We presented an improved Sauerbrey equation that can be used to effectively compensate the drift of the QCM resonator. These results will play a significant role when applying the QCM at the room temperature.

Keywords: quartz crystal microbalance, hybrid temperature effect, aqueous solution

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1. Introduction

A quartz crystal microbalance (QCM) measures the mass variation per unit area by measuring the change in frequency of a quartz crystal resonator. The changes in mass deposited on the crystal surface are directly reflected in changes of the resonator frequency. In 1959, Sauerbrey first developed a method for measuring the resonator frequency of the QCM resonator in air using an oscillator circuit.[1] In 1985 Sauerbrey's method was further developed by Kanazawa for measuring viscosity and density of a liquid.[2] Both methods continue to be used in nearly all applications as the primary tool in QCM resonator experiments for conversion of frequency to mass.[3]

A QCM sensor typically consists of an oscillator circuit containing a thin AT-cut quartz disk with circular electrodes on both sides of the quartz. Due to the piezoelectric properties of the quartz material, an alternating voltage between these electrodes leads to mechanical oscillations of the crystal. The resonant frequency of the quartz is directly affected by the thermal changes.[4,5] When a QCM resonator contacts liquids, both the accumulation of rigid mass and changes in the property of the liquid, e.g., density and viscosity, contribute to the frequency shift. The density and viscosity of solutions have a strong dependence on temperature.[6] Thus, the methods of Sauerbrey and Kanazawa are inappropriate for liquid measurement when considering the temperature effect. Control of the temperature effect is key when applying the QCM for high accuracy measurements. As we know, four methods are typically used to control the temperature effect on the measurements. First, keep the quartz crystal[7–9] sensors at a constant temperature by placing them in a temperature-controlled container. Second, use an additional reference crystal,[10–12] that is protected from mass changes yet experiences temperature changes identical to the measurement crystal. This is called the dual crystal method. Third, the baseline subtraction method uses QCM resonator data before and after the measurement,[13] which assumes that the drift of the QCM resonator baseline caused by the temperature effect can be modeled.[14] Fourth, compensate for the drift using analog or digital voltage.[15] However, when considering these 4 methods, the temperature of the liquid cannot be controlled for several possible applications (e.g., sea-water immersed sensor). The "dual crystal method" is difficult to employ due to the many temperature effects caused by transient temperatures that the reference crystal may not detect.[16] Furthermore, to compensate for drift using analog or digital voltage would be inaccurate due to the amplitude-frequency effect,[17] which denotes the variation in frequency with respect to the crystal[18–20] current after the temperature effect has been eliminated. In addition, the effect of change in a solution's density and viscosity induced by temperature is not taken into account.

In this paper, we discussed a problem concerning the hybrid temperature effect on a QCM resonator in aqueous solutions. Based on the Sauerbrey's and Kanazawa's theories, we then proposed a method for enhancing the accuracy of the QCM measurement, which takes into account not only the thermal variations of viscosity and density but also the thermal behavior of the QCM resonator.

2. Method and experiments

2.1. Method

2.1.1. Sauerbrey equation

According to Sauerbrey's theory, the rigid attachment of a film of mass Δm to the crystal surface causes a decrease in

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the resonant frequency Δf, and the relationship between Δf and Δm is linear in the limit of small Δm without regard to temperature changes. It can be expressed by Eq. (1)

$$\Delta f = -2.26 \times 10^{-6} \cdot \frac{f_0^2}{A} \Delta m, \tag{1}$$

where f_0 is the resonant frequency (in unit Hz), Δf is the frequency change (in unit Hz), Δm is the mass change (in unit g), and A is the piezoelectrically active crystal area (in unit cm^2),

2.1.2. Temperature characteristic equation of QCM

To analytically describe the frequency-temperature behavior, the measured change of frequency versus temperature can be developed in a power series, determined by first, second, and third-order temperature coefficients.[21–23] It can be expressed by Eq. (2)

$$\frac{\Delta f(T)}{f_0} = A_1 \cdot (T - T_0) + A_2 \cdot (T - T_0)^2 + A_3 \cdot (T - T_0)^3, \tag{2}$$

where, A_1, A_2, and A_3 are the temperature coefficients measured at the reference temperature T_0; T is the environmental temperature; and f_0 is the resonator frequency at T_0. The temperature effect is caused by the property of the crystal itself.

2.1.3. Viscosity-density effect on the solution

According to Kanazawa, if a Newtonian liquid is in contact with one of the QCM resonator surfaces, the resonator frequency change is proportional to the square root of the product of viscosity and density.[2] This can be expressed by Eq. (3)

$$\Delta f_{\eta\rho} = -f_0^{3/2} \left(\frac{\eta_\mathrm{l} \rho_\mathrm{l}}{\pi \mu_\mathrm{q} \rho_\mathrm{q}} \right)^{0.5}, \tag{3}$$

where, η_l and ρ_l are the solution viscosity and solution density, respectively. Note that the solution density and solution viscosity vary with the temperature, ρ_q is the density of the quartz ($\rho_\mathrm{q} = 2.643\ \mathrm{g/cm^3}$), and μ_q is the shear modulus of the AT-cut crystal quartz ($\mu_\mathrm{q} = 2.947 \times 10^{11}\ \mathrm{g \cdot cm^{-1} \cdot s^{-2}}$).

2.2. Experiment

2.2.1. Assumptions

Assumption 1 Although frequency change can be affected by surface roughness, this research does not consider its effect. Because the same crystal electrodes were used for all tests, there was no change in surface roughness during the experiments.

Assumption 2 To prevent instantaneous frequency changes and drift due to transient temperature effects, the resonant frequency of the crystal was measured as the temperature performs a slow sweep over the desired temperature range.

Assumption 3 The Hybrid temperature effect (Δf_T) on the QCM resonator in aqueous solutions is divided into two parts, namely, the direct temperature effect (Δf_Td) and the indirect temperature effect (Δf_Tnd). Thus, $\Delta f_\mathrm{T} = \Delta f_\mathrm{Td} + \Delta f_\mathrm{Tnd}$.

Assumption 4 The direct temperature effect on the QCM resonator for all aqueous solutions has the same characteristics as in pure water.

2.2.2. Materials and equipment

In the experiments, three identical 8-MHz AT-cut QCMs (AC8AP14, JJK Electronic Co., Ltd.,) coated with gold were used, as shown in Fig. 1. The diameter of the crystal was 14 mm, the areas of the electrodes were about 28.26 mm^2, and the resolution ratio of the QCM was 5.5 ng/Hz. The QCM resonator was mounted in an electrolytic cell, as shown in Fig. 2.

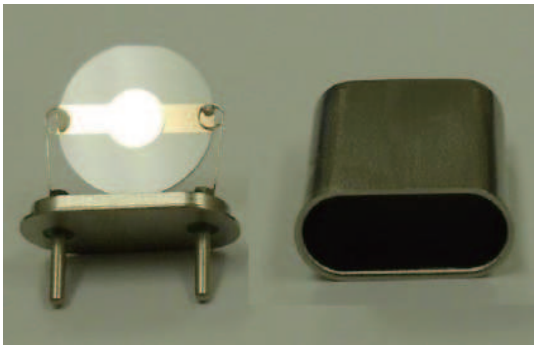


Fig. 1. (color online) Photo of the QCM resonator.

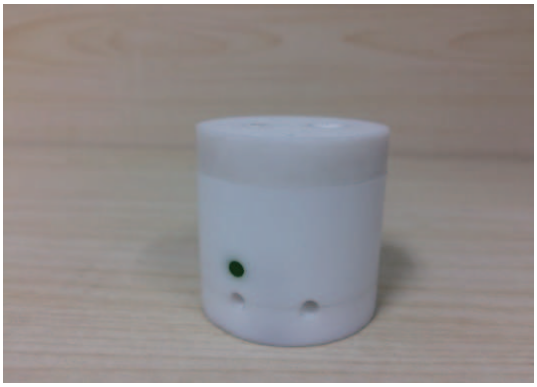


Fig. 2. (color online) Photo of the electrolytic cell.

The main crystal electrode acted as a working electrode in solution, while the opposite side of the crystal was exposed to air. The electrodes were connected to an oscillator circuit using small, flat-nosed alligator clips. Tests were conducted using a frequency measuring device (EQCM-400C, Shanghai, Instruments, Inc.) which outputs ten frequencies per second, and a Lab-VIEW 2014 virtual instrument developed in the author's laboratory. In this research, the frequency-change measurements were made using Lab-View software. The viscosity of the liquids (NaCl solutions and pure water) at different temperatures was measured using a Digital Rotary Viscometer (NDJ-8S, Shanghai Pingxuan Instrument Co., Ltd.), while the density of the liquids at different temperatures was measured using a specific gravity meter (DA-130N, Kyoto Electronics

Manufacturing Co., Ltd.). The environmental temperature was directly measured during the operation of the QCM using a digital thermometer (TM-902C, LUTRON ELECTRONIC ENTERPRISE Co., Ltd.) with a sensitivity of 0.1 °C. Lastly, the temperature was controlled using an electrothermal constant temperature incubator (BEIJING KEWEI YONGXING INSTRUMENT Co., Ltd.) with a temperature sensitivity of 0.1 °C. For all experiments, three cleansers, 95% alcohol solution, 99% acetone solution, and pure water, were used sequentially to clean the QCM resonator with the ultrasonic cleaner (JP-008, Skymen cleaning equipment Co., Ltd.) for 5 min at room temperature. Then, the QCM resonator was dried using nitrogen.

2.2.3. Test samples

We conducted three groups of experiments: i) experiments of the temperature effect on the NaCl solution; ii) experiments of the direct temperature effect on the QCM; iii) experiments of the indirect temperature effect on the QCM.

In the first group of experiments, we investigate the temperature effect on the NaCl solution, the densities and viscosities of five NaCl solutions were measured in the range of 20 °C to 35 °C.

For the second group of experiments, we measured frequencies of the QCM in air and pure water. We chose 37 temperature points over the range of 20.3 °C to 55.7 °C (see Table 1), and 27 temperature points, over the range of 21.3 °C to 51.2 °C (see Table 2), respectively. Here, the viscosities and densities of the pure water at different temperatures were independently measured.

Table 1. Temperature points in analysis of temperature effect of QCM resonator in air.

Temperature/°C							
20.3	25.0	28.7	33.6	38.6	42.6	48.6	54.1
21.5	25.6	30.5	34.9	39.1	43.8	49.6	55.7
22.5	26.1	31.8	35.8	39.3	44.9	50.1	
23.8	27.6	32.1	36.7	40.7	46.5	51.6	
24.6	28.5	33.0	37.3	41.8	47.8	53.2	

Table 2. Temperature points in analysis of temperature effect of QCM resonator in pure water.

Temperature/°C					
21.3	26.3	31.6	36.2	42.8	49.7
22.2	27.5	32.5	37.8	44.2	51.2
23.9	28.1	33.2	39.2	45.8	
24.8	29.8	34.5	40.6	47.3	
25.0	30.7	35.6	41.5	48.6	

In the third group of experiments, we used five NaCl solutions with different concentrations (see Table 3) as test samples, and the QCM resonator frequency measurements were conducted at 25 °C and 30 °C. NaCl was selected due to its minimal ability to adsorb at the surface of the QCM crystal electrode.

Table 3. NaCl solution concentrations in analysis of temperature–viscosity–density–frequency effect of QCM resonator.

Concentration/mol·L^{-1}				
0.01	0.1	0.5	1.0	2.5

2.2.4. Frequency data

One frequency datum was obtained for each experiment by averaging the output frequencies in a stable baseline. We averaged about 1000 or 2000 frequencies, with the EQCM-400C for a more accurate frequency change measurements. A median of five frequency data from five repetitive experiments was reported as a measuring result. In particular, all data points were taken at 25 °C.

3. Results and discussion

3.1. Temperature effect in NaCl solutions

Figures 3 and 4 show the linear relationships between temperature and density or viscosity in NaCl solutions (20 °C $\leqslant T \leqslant$ 35 °C). The density (ρ_l) dependence on temperature is generally represented by Eq. (4)

$$\rho_l = -aT + b, \tag{4}$$

where a is the temperature coefficient of NaCl solution density and b is a constant. The values of a and b for fitting the curves in Fig. 3 are summarized in Table 4.

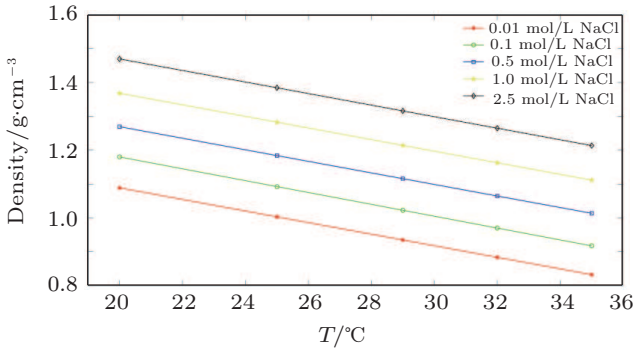


Fig. 3. (color online) Density versus temperature in NaCl solution.

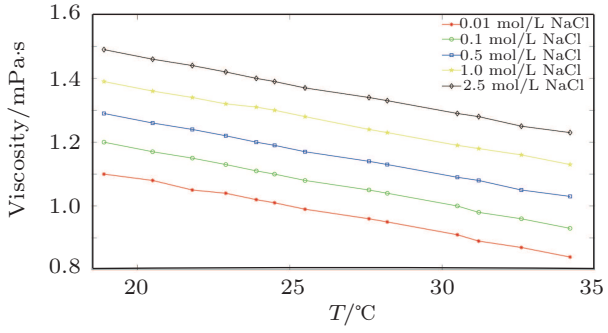


Fig. 4. (color online) Viscosity versus temperature in NaCl solution.

The viscosity (η_l) dependence on the temperature is generally represented by Eq. (5)

$$\eta_l = -cT + d, \tag{5}$$

where c is the temperature coefficient of the NaCl solution viscosity and d is a constant. The values of c and d for fitting the curves in Fig. 4 are summarized in Table 5.

Table 4. Density–temperature coefficients of NaCl solutions with different concentrations.

Concentration/mol·L^{-1}	a/g·cm^{-3}·°C^{-1}	b/g·cm^{-3}
0.01	1.707	1.43
0.1	1.750	1.53
0.5	1.704	1.61
1.0	1.710	1.71
2.5	1.704	1.81

Table 5. Viscosity–temperature coefficient of NaCl solutions with different concentrations.

Concentration/mol·L^{-1}	c/m·Pa·s·°C^{-1}	d/m·Pa·s
0.01	0.0003521	1.013
0.1	0.0003581	1.021
0.5	0.0003531	1.049
1.0	0.0003503	1.079
2.5	0.0003576	1.108

3.2. Direct temperature effect on QCM

Figure 5 shows the relationship between frequency changes of the QCM and temperature changes in air. The equation for fitting the relationship is expressed as follows:

$$\begin{aligned}\Delta f_{\mathrm{Td}} = {} & 0.02048\,(T-T_0)^3 - 1.369\,(T-T_0)^2 \\ & + 31.12\,(T-T_0) - 15.07\end{aligned} \tag{6}$$

for 20 °C $\leqslant T \leqslant$ 55 °C, where T is the test temperature and T_0 is the reference temperature ($T_0 = 25$ °C).

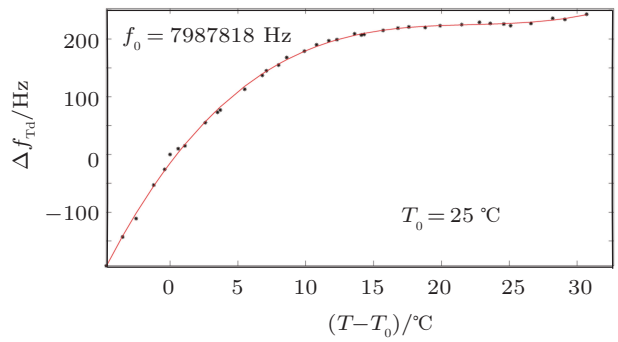


Fig. 5. (color online) Direct temperature effect of the QCM resonator in air.

According to Eq. (6), a 350-Hz frequency change appears when the temperature change of the air is 35 °C (20 °C $\leqslant T \leqslant$ 55 °C). In other words, the average frequency change is 10 Hz/°C. The resonator frequency must compensate for any critical application of the QCM measurement.

Figure 6 shows the relationship between the resonator frequency changes of the QCM and temperature changes in pure water. According to Assumption 3, the direct temperature effect (Δf_{Td}) can be obtained when the indirect temperature effect (Δf_{Tnd}) is removed from the hybrid temperature effect (Δf_{T}).

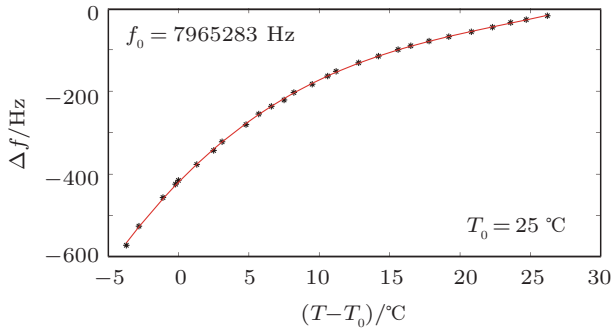


Fig. 6. (color online) The resonator frequency of QCM in pure water.

Figure 7 shows the curve of Δf_{Td} in pure water, and the equation fitting the curves can be expressed by the following equation

$$\begin{aligned}\Delta f_{\mathrm{Td}} &= \Delta f - \Delta f_{\eta\rho} \\ &= 0.01689\,(T-T_0)^3 - 1.188\,(T-T_0)^2 \\ &\quad + 27.95\,(T-T_0) - 4.63\end{aligned} \tag{7}$$

for 20 °C $\leqslant T \leqslant$ 55 °C.

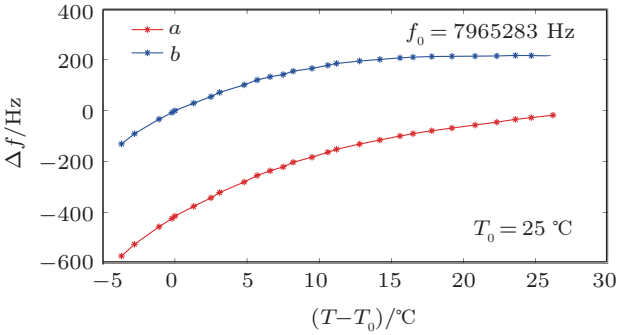


Fig. 7. (color online) The resonator frequency of QCM in pure water (curve a); The direct temperature effect (Δf_{Td}) of QCM in pure water (curve b).

By comparing Eqs. (6) and (7), we find an obvious difference, which indicates that the direct temperature effect is different for the QCM resonator in air and pure water. When the QCM resonator is used in an aqueous solution, the effect on the QCM resonator in pure water should be used.

3.3. Indirect temperature effect on QCM resonator

Figure 8 shows the indirect temperature effect on the QCM resonator in the NaCl solutions, where the resonator frequencies were measured by experiments and calculated by Kanazawa's theory in the NaCl solutions with different concentrations at 25 °C and 30 °C.

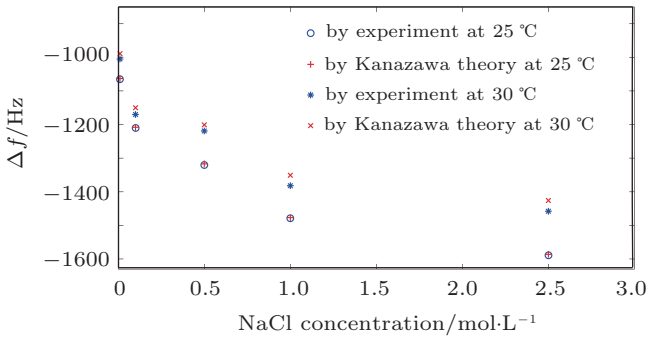


Fig. 8. (color online) Indirect effect on the QCM resonator in the NaCl solutions with different concentrations.

At 25 °C, a good agreement was found for Δf between the experimental results and Kanazawa's theory, which states that the resonator frequency change is proportional to the square root of the change in viscosity and density.[24] On the other hand, an obvious difference can be found at 30 °C, where the resonator frequency change does not agree with Kanazawa's theory, as shown in Fig. 9. The differences in the observed frequency changes are due to the hybrid temperature effect on the QCM resonator.

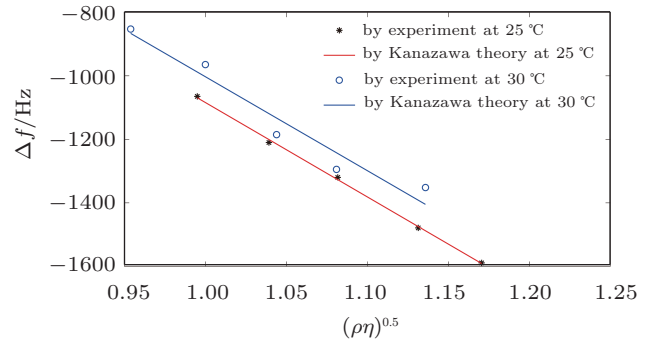


Fig. 9. (color online) Frequency change versus $\sqrt{\eta\rho}$ in NaCl solution at 25 °C and 30 °C.

Experiments demonstrated the phenomena of the QCM resonator frequency drift in the aqueous solutions when its temperature ranged from 20 °C to 35 °C. The drift resulting from the hybrid temperature effect which severely affected the performance of the QCM resonator. Thus, the Sauerbrey equation should be further improved for QCM drift compensation to enhance the measurement accuracy at room temperature.

4. Temperature compensation to QCM

4.1. Temperature compensation

According to Assumption 3, when the QCM measurement is is conducted in aqueous solutions, the frequency change Δf_{QCM} can be expressed as

$$\Delta f_{\mathrm{QCM}} = \Delta f_{\mathrm{m}} + \Delta f_{\mathrm{Td}} + \Delta f_{\mathrm{Tnd}}, \tag{8}$$

where Δf_{m} is the resonator frequency change due to the adsorption at the metal–liquid interface according to the Sauerbrey equation, Δf_{Td} is the resonator frequency change due to the direct temperature effect according to Eq. (7), and Δf_{Tnd} is the resonator frequency change due to the indirect temperature effect according to Eq. (3). The density (ρ_l) and viscosity (η_l) in Eq. (3) should be compensated using Eqs. (4) and (5).

Equation (9) shows the drift compensation equation for the critical calculation of the resonator frequency change of the QCM resonator in aqueous solutions

$$\begin{aligned}\Delta f_{\mathrm{QCM}} = {} & \Delta f_{\mathrm{m}} + 0.01689\,(T-T_0)^3 - 1.188\,(T-T_0)^2 \\ & + 27.95\,(T-T_0) - 4.63 \\ & - f_0^{3/2}\left(\frac{(b-aT)\,(c-dT)}{\pi\mu_{\mathrm{q}}\rho_{\mathrm{q}}}\right)^{0.5}\end{aligned} \tag{9}$$

for 20 °C $\leqslant T \leqslant$ 35 °C, where a, b, c, and d at a reference temperature T_0 must be obtained for a particular solution, μ_{q} is the shear modulus of the AT-cut crystal ($\mu_{\mathrm{q}} = 2.95 \times 10^{11}1$ g/cm $\cdot$ s^2), and ρ_{q} is the density of the crystal ($\rho_{\mathrm{q}} = 2.684$ g/cm^3).

Figure 10 shows compensatory frequencies (f_{tc}) of the QCM resonator in NaCl solutions with five different concentrations. Each compensatory frequency was calculated by using the following equation

$$\begin{aligned}f_{\mathrm{tc}} = {} & \Delta f_{\mathrm{m}} - \Delta f_{\mathrm{QCM}} \\ = {} & -0.01689\,(T-T_0)^3 + 1.188\,(T-T_0)^2 - 27.95\,(T-T_0) \\ & + 4.63 + f_0^{3/2}\left(\frac{(b-aT)\,(c-dT)}{\pi\mu_{\mathrm{q}}\rho_{\mathrm{q}}}\right)^{0.5}\end{aligned} \tag{10}$$

for 20 °C $\leqslant T \leqslant$ 35 °C.

The compensatory frequencies were obtained by considering the influences of the direct temperature effect and indirect temperature effect.

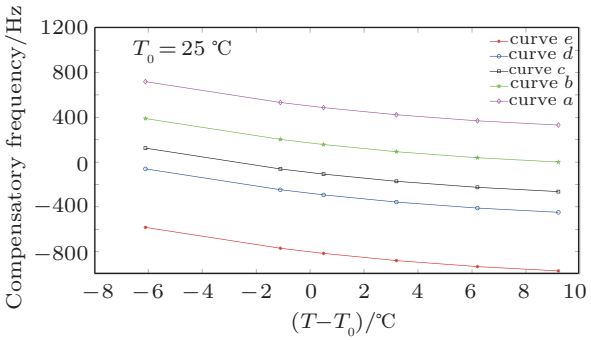


Fig. 10. (color online) Compensatory frequency of the QCM resonator in NaCl solutions with different concentrations. (a) NaCl concentration: 0.01 mol·L^{-1}; (b) NaCl concentration: 0.1 mol·L^{-1}; (c) NaCl concentration: 0.5 mol·L^{-1}; (d) NaCl concentration: 1.0 mol·L^{-1}; (e) NaCl concentration: 2.5 mol·L^{-1}.

According to Eq. (10), an accurate frequency change can be obtained, which removes the influences of the hybrid temperature effect of the QCM resonator. The frequency change is shown in a drift compensation equation, namely, Eq. (11).

This may be used in the Sauerbrey equation to acquire a more accurate result.

$$\Delta f_{\mathrm{m}} = \Delta f_{\mathrm{QCM}} - 0.01689\,(T-T_0)^3 + 1.188\,(T-T_0)^2 - 27.95\,(T-T_0) + 4.63 + f_0^{3/2}\left(\frac{(b-aT)\,(c-dT)}{\pi\mu_{\mathrm{q}}\rho_{\mathrm{q}}}\right)^{0.5} \quad (11)$$

for 20 °C $\leqslant T \leqslant$ 35 °C.

4.2. Experimental verification

We validated Eq. (10) by calculating the compensatory frequencies of the QCM resonator in NaCl solutions with concentrations of 0.1 mol·L^{-1} and 1.0 mol·L^{-1} at temperatures of 22 °C and 30 °C, where the coefficients a, b, c, and d obtained at a reference temperature 25 °C for the NaCl solutions. Figure 11 shows the compensation frequencies of the QCM resonator.

Table 6 lists the compensation frequencies of the QCM resonator in NaCl solutions with different concentrations under different temperatures. The minimum and maximum absolute differences between the theoretical compensatory frequencies and the calculated compensatory frequencies were 2.22 Hz and 9.6 Hz, respectively. All absolute deviations were less than 5%. The results indicated that the calculated compensatory frequencies (f_{c}) matched the theoretical compensatory frequencies (f_{tc}) from Eq. (10). Thus, the presented drift compensation (Eq. (11)) is reasonable.

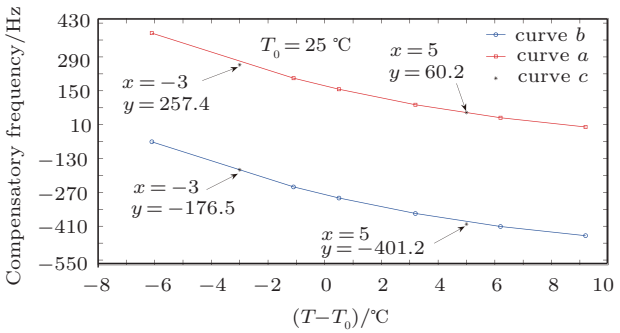


Fig. 11. (color online) The compensation frequencies of the QCM resonator in NaCl solutions with different concentrations. (a) 0.1 mol·L^{-1}; (b) 1.0 mol·L^{-1}; (c) Calculated compensation frequency values.

Table 6. Statistics of the compensation frequencies of the QCM resonator in NaCl solutions.

NaCl solutions /mol·L^{-1}	$(T-T_0)$/°C $T_0 = 25$ °C	Theoretical compensatory frequency f_{tc}/Hz	Calculated compensatory frequency f_{c}/Hz	Absolute difference $(f_{\mathrm{tc}} - f_{\mathrm{c}})$/Hz	Absolute deviation $[(f_{\mathrm{tc}} - f_{\mathrm{c}})/f_{\mathrm{tc}}] \times 100\%$
0.1	−3	265.1	257.4	7.7	2.9%
0.1	5	57.98	60.2	2.22	3.83%
1.0	−3	−184.4	−176.5	7.9	4.28%
1.0	5	−391.6	−401.2	9.6	2.45%

5. Conclusions

In this paper, we investigated the hybrid temperature effect on the QCM resonator in aqueous solutions to build a more accurate relationship among the resonator frequency of the QCM resonator, the adsorbed mass, and the environmental temperature. We presented an equation developed from the Sauerbrey equation based on the hybrid temperature effect. The proposed method effectively compensated for the drift of the QCM resonator. It avoided variations in the resonator response due to the environmental temperature variations occurred during the actual measurement. These results will play a significant role in future QCM application at the room temperature.

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