

Rapid and sensitive detection of formaldehyde based on AC electrokinetic effects

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Formaldehyde is a toxic water-soluble organic matter. Its detection methods and detection limit have been attracting more attentions. An AC electrokinetic (ACEK)-enhanced capacitive sensing method for rapid detection of trace formaldehyde in liquid is presented using commercial microelectrode chips. By this method, a detection limit is achieved as low as 0.01 ppm ($\mu\text{g/ml}$) within a 60 s response time. It is proved that the trace amount of formaldehyde in common solvents can also be effectively detected. As a complement, the capacitance change rate dependence on testing voltage and frequency is investigated. Compared to other existing detection methods, this ACEK-based capacitive method is a low cost, high sensitive and fast detection means, which shows a great application prospect in industry and research such as food safety and specimen cleaning.

1. Introduction: Formaldehyde is an important industrial raw material and chemical reagent. It is colourless, soluble in water or organic solvents and has a pungent odour. It is widely used in medicine, wood processing industry, textile industry and so on. Owing to its toxic properties, soaking food using formaldehyde or over-standard concentration in air are prohibited by laws in most countries. Consequently, detection of formaldehyde sensitively is an attention-worth project in these fields.

Spectrophotometric method is a commonly used formaldehyde testing method with simple and relatively low-cost equipments [1]. The detection limit is about 0.02 mg/l. However, this method requires a specific chemical reaction, while the relative stability is poor, and the operation process is complex. Electrochemical method is also widely used in formaldehyde detection with a detection limit of 0.055 mg/l [2]. It requires electronic sensing equipments, and is easy to be disturbed. In fact, the data error of quantitative detection is large. Chromatography is another way of formaldehyde detection with a high sensitivity and quantitative accuracy [3]. The detection limit is as low as 0.0015 mg/l, while a wrinkle is that chromatography requires sophisticated equipments, long derivatisation time and complex process. These above methods are popular ways to detect formaldehyde in this decade, but they may be not suitable for low-cost, rapid and sensitive detection, especially for on-site detection. Therefore, developing a simple, rapid and sensitive formaldehyde detection method is useful and necessary.

This Letter presents an AC electrokinetic (ACEK)-enhanced capacitive sensing method for low cost, rapid and accurate formaldehyde detection. Commercially available microelectrode chips were modified and adopted as one time sensors, and the cost is <\$0.50 per test. ACEK capacitive sensing is an increasingly used technology recently [4–6], which applies AC signal on microelectrodes to generate local fluid motion facilitating particle movement and enrichment. ACEK-enhanced capacitive tests have been used for detection of protein [7], bisphenol A [8], progesterone [9] and other substances [10, 11]. It has advantages to realise cheaper and smaller sensors. According to our results, this method should have wide application prospect in spot test of formaldehyde.

2. Mechanisms and methods

2.1. Detection of molecule adsorption: Detection of molecules can be achieved by extracting changes from interfacial

capacitance, and there were traditional microelectromechanical system sensors which performed this process [12, 13]. DC voltages are always loaded to these high-cost sensors while the particle enrichment is not significant enough. We applied AC signal on two sets of interdigitated electrodes resulting in ACEK effect. The level of molecular adsorption on the electrodes can be judged by the change of interfacial capacitance at the electrode surface.

When ionised formaldehyde molecules are deposited on the electrode surface, there will be net charges on the surface. To keep conservation of charges, a thin layer of counter ions will accumulate in the fluid near the electrode surface, which is known as electric double layer (EDL) [14]. EDL can be regarded as a dielectric layer of the interfacial capacitor. If formaldehyde molecules are adsorbed onto the electrode surface, these molecules play a role of dielectric layer. As a result, the thickness and surface of dielectric layer for interfacial capacitor increase. Then, the interfacial capacitance will be changed.

As shown in Fig. 1, the dielectric layer of interfacial capacitor is made up by the EDL λ_d before formaldehyde adsorption. The initial capacitance can be expressed as

$$C_0 = \varepsilon_s A_{\text{int}} / \lambda_d \quad (1)$$

where ε_s is the permittivity of the sample fluid, A_{int} is the surface area of the interfacial capacitor and λ_d is the EDL thickness. After adsorption of formaldehyde molecules, the total capacitance is the initial capacitance and formaldehyde molecular layer capacitance in series. According to series capacitance formula, the capacitance changes to

$$1/C = \lambda_d / \varepsilon_s \cdot A_{\text{int}} + d_{\text{CHO}} / \varepsilon_{\text{CHO}} \cdot A_{\text{CHO}} \quad (2)$$

where A_{CHO} is the effective surface area of formaldehyde molecular layer and d_{CHO} is the thickness of formaldehyde molecular layer.

Then the relative change of capacitance is given by

$$\Delta C / C_0 = (C - C_0) / C_0 = B / [B + d_{\text{CHO}} / (\varepsilon_{\text{CHO}} \cdot A_{\text{CHO}})] - 1 \quad (3)$$

where $B = \lambda_d / \varepsilon_s \cdot A_{\text{int}}$, and $d_{\text{CHO}} / A_{\text{CHO}}$ determines the rate of change in capacitance.

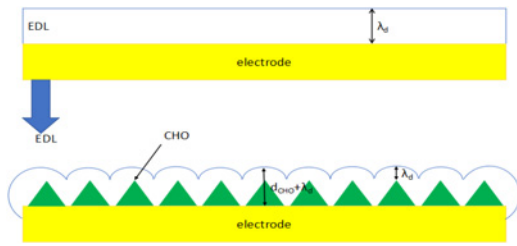


Fig. 1 Changes at the electrode surface due to molecule adsorption

Assuming spherical-shaped formaldehyde molecules, the relationship of surface area to thickness can be considered as a quadratic relation. So $d_{\text{CHO}}/A_{\text{CHO}} \propto 1/d_{\text{CHO}}$. Therefore, the normalised capacitance change rate can be described as

$$\Delta C/C_0 = B/[B + 1/(E \cdot d_{\text{CHO}})] - 1 \quad (4)$$

where $E = \varepsilon_{\text{CHO}}$ is a constant.

Consequently, the surface adsorption of formaldehyde, i.e. the increment of d_{CHO} , can be detected through $\Delta C/C_0$.

In this work, normalised capacitance change rate (i.e. $\Delta C/C_0$ over a fixed test period) is employed to indicate the surface adsorption events. Since $\Delta C/C_0$ can be directly correlated with the amount of formaldehyde molecule on the electrode surface, this is also a quantitative detection method. Also, using $\Delta C/C_0$ helps to achieve comparability of different sensor detections. In reality, when measuring the impedance of certain sample, inconsistency from sensor to sensor always exists. As a result, the baseline and initial capacitances have a range instead of a well-defined value. We can reduce the uncertainty of test results using normalised capacitance change rate, because these are relative changes. According to (4), the normalised capacitance change rate should increase when there is molecular adsorbent.

2.2. Alternating electric field effect: When electrodes are immersed in electrolytes, the electrolyte/electrode system can be represented as a network of surface components and bulk components, both with capacitive and resistive elements [15, 16]. For an AC frequency range below several hundred kilohertz, the electric network can be further simplified as a series connection of interfacial capacitance $C_{\text{int},0}$ and bulk resistance R_f [6], as shown in Fig. 2.

Interfacial capacitance $C_{\text{int},0}$ consists of EDL capacitance at the electrolyte/electrode interface [16] and dielectric deposition caused by adsorption of formaldehyde molecules on the electrode. R_f is the bulk fluid resistance due to current conduction between electrodes. Capacitive sensing utilises the change of the coverage/thickness and dielectric properties of the electrode surface caused by deposition of formaldehyde molecules, which will lead to a change of $C_{\text{int},0}$.

In this experiment, formaldehyde molecules depositing on the electrode surface mainly rely on ACEK effects. As shown in Fig. 3, target analytes move randomly in the fluid by Brownian motion without any AC signal. When AC signal is loaded, target

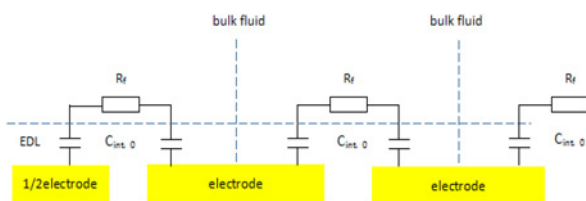


Fig. 2 Simplified equivalent circuit of electrolyte/electrode system

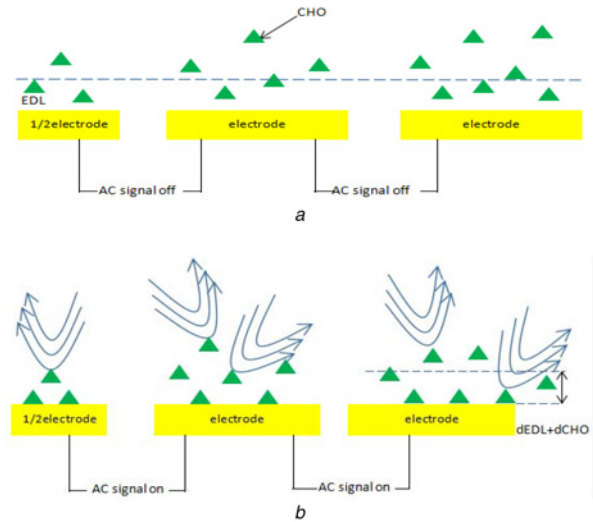


Fig. 3 ACEK effect on target analytes

a AC signal off

b AC signal on

analytes are convected toward the electrode surface by ACEK effects and deposit there.

In this research, ACEK effect is a general term which may include several effects as discussed below. When inhomogeneous AC electric field is applied from microelectrodes to an aqueous solution, AC electro-osmotic (ACEO) pump causes fluid to produce a directional flow through the non-uniform alternating electric field to induce the charge in the asymmetric EDL. Dielectric electrophoresis (DEP) makes movement to neutral particles in suspensions by polarisation effect, so that different samples are separated. Furthermore, it is found that during alternating electric field being applied to control the fluid and particles, high electric field strength is produced in solution near the surface of the electrode. Although the alternating voltage may be low, the electric field strength can be high due to short distance of several microns between adjacent electrodes. At the same time, there are electric field gradients from electrode surface to the solution. The inhomogeneous distribution of electric field leads to changes of solution conductivity and dielectric constant, and then AC electrothermal (ACET) flow effect appears as a result. In brief, ACEO pump, DEP and ACET flow constitute the ACEK effects.

On the basis of Maxwell's classical electromagnetic field theory, Pohl established calculation model of DEP tapping in 1978 for operation and separation of particles to be detected. The calculation model of the electrophoretic force F_{DEP} can be expressed as

$$F_{\text{DEP}} = 2\pi\varepsilon_m r^3 \text{Re}[K(\omega)] \nabla |E_{\text{rms}}|^2 \quad (5)$$

where ε_m is the relative permittivity of suspension medium, r is the particle radius, $\text{Re}[K(\omega)]$ is the real part of Clausius–Mossotti factor and $K(\omega)$ is Clausius–Mossotti factor given by

$$K(\omega) = (\varepsilon_p^* - \varepsilon_m^*)/(\varepsilon_p^* + 2\varepsilon_m^*) \quad (6)$$

where ε_m^* is the composite permittivity of the suspension medium, ε_p^* is the composite permittivity of the particle, ω is the electric field angular frequency and σ is the conductivity. $\text{Re}[K(\omega)]$ is limited between -0.5 and 1 . $|E_{\text{rms}}|^2$ is the gradient of the square of electric field strength in root mean square value. On the basis of (5), for nanoscale molecules, DEP is effective with high $|E_{\text{rms}}|^2$. To detect low abundance analyte in sample fluid, a longer range mechanism such as fluid flow field is needed to transport the molecules to the electrodes.

Concomitant to DEP effect, AC electric field can generate micro-flows through one or both of two ACEK flow mechanisms. ACEO typically dominates when the conductivity of the liquid is low, and ACEO flow velocity has been observed to decrease significantly with increasing fluid's electrical conductivity and eventually drop to zero at above 0.085 S m^{-1} [17]. When we used formaldehyde solution diluted in deionised (DI) water with the conductivity of formaldehyde being $4 \times 10^{-4} \text{ S m}^{-1}$ and DI water being $4 \times 10^{-6} \text{ S m}^{-1}$, ACEO was a contributing factor. When tap water or normal saline both with much higher conductivities were adopted as the solvents, ACEO almost had no effect on flowing.

The uneven distribution of field strength leads to temperature gradient. As a result, a so-called ET power is produced, which leads to ET flow effect, i.e. ACET. Previous studies have shown that ACET effects play an important role in improving detection sensitivity. The ACET force can be expressed as follows:

$$F_e = \frac{1}{2} \frac{\epsilon_m (\alpha - \beta)}{1 + (\omega\tau)^2} (\nabla T \cdot \mathbf{E}_{\text{rms}}) \cdot \mathbf{E}_{\text{rms}} - \frac{1}{4} \epsilon_m \partial |\mathbf{E}_{\text{rms}}|^2 \nabla T \quad (7)$$

where T is the temperature of solution, ϵ_m and σ_m represent the dielectric constant and conductivity of solution and \mathbf{E}_{rms} is the effective value of alternating electric field strength. Assuming that the solution temperature change is very small, α and β can be expressed as

$$\alpha = \frac{(\partial \epsilon_m / \partial T)}{\epsilon_m}, \quad \beta = \frac{(\partial \sigma_m / \partial T)}{\sigma_m} \quad (8)$$

3. Experimental details

3.1. Preparation of experimental samples: This experiment was composed of three parts, which were test of formaldehyde diluted in DI water, tap water and normal saline (0.9% sodium chloride aqueous solution). We diluted liquid formaldehyde in these three types of solvents to get three groups of formaldehyde aqueous solution, and every group contained six concentrations from 0.01 to 10 ppm.

3.2. Processing of microelectrode chips: In this work, commercially available electrode chips, i.e. surface acoustic wave (SAW) resonator chips (SF-R433H, manufactured by China Electronics Technology Group Corporation) were slightly modified to obtain the microelectrodes. The metal covers of the SAW chips were removed, so that the inside working electrode array could be exposed. The metal casing around the electrode chip is about $10 \mu\text{l}$, serving as a microchamber for sample solutions. The interdigitated electrodes are made of aluminium deposited on quartz substrate. There are two sets of interdigitated electrodes in one chip, each with 425 fingers. The electrode finger length is $1620 \mu\text{m}$ and the thickness is $2 \mu\text{m}$. The finger width and distance between adjacent fingers are both $20 \mu\text{m}$. Images of an original and modified SAW chip as well as the scanning electron micrographs of the microelectrode array are provided in Fig. 4.

Next, electrode chips need to be cleaned. We adopted standard cleaning steps according to large quantities of similar research. The SAW chips are thoroughly cleaned by acetone, isopropyl alcohol and DI water to remove possible contamination during chip packaging. Then, the chips were plasma treated for 30 s. After thorough cleaning of the electrodes, we put appropriate amount of sample into the chip sample solution chambers.

3.3. ACEK impedance measurement: The interdigitated electrodes were electrically connected to two contact pads on the chip bottom, which would be connected to an impedance analyser (Wayne Kerr, WK6500B) during detection. The measurement schematic is as shown in Fig. 5.

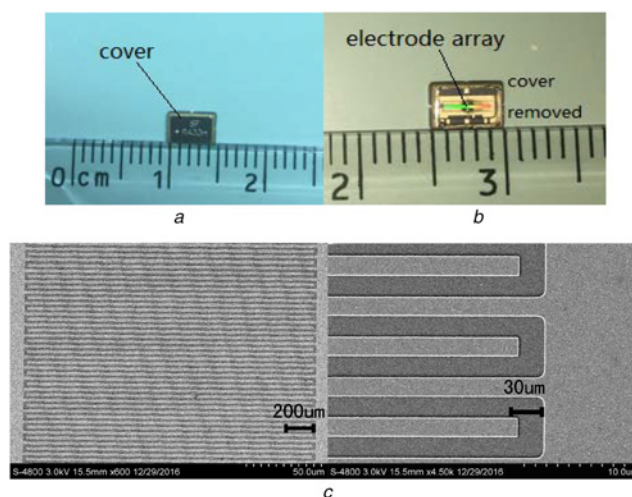


Fig. 4 Images of sensor chip with interdigitated electrodes
a Original SAW chips
b Modified SAW chips
c Scanning electron micrographs of interdigitated electrodes

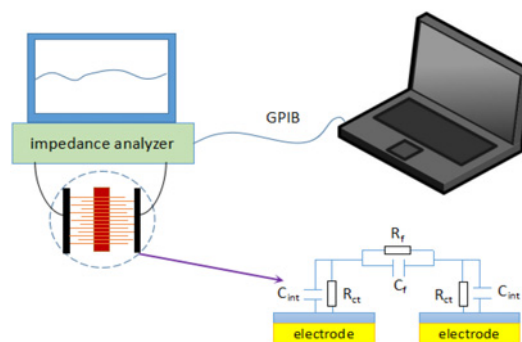


Fig. 5 Microelectrode sensor measuring system

The sensor capacitance was continuously measured for 60 s at 30 kHz with 50 mV AC signal, and the detection would be finished when the capacitance measurement was done. The change rate of capacitance was then calculated to indicate the adsorption level of formaldehyde molecules, which is shown by slope of normalised capacitance versus time (%/min). In addition, blank controls (DI water, tap water, normal saline) without formaldehyde were tested to identify any possible artefacts caused by the electric signal.

4. Results and discussion

4.1. Trace formaldehyde detection: Formaldehyde samples of various concentrations dissolved in DI water were tested at 30 kHz and 50 mV_{rms} AC signal for 60 s. Fig. 6 shows how normalised capacitance changes with time.

At the beginning, we tested four formaldehyde solution samples of different concentrations as 0.01, 0.1, 1 and 10 ppm. It was found that the slope increases from 0.01 to 1 ppm. However, it turns low as small as 0.01 at 10 ppm, which indicates that the adsorption has been saturated at 10 ppm. To accurately find out the concentration when adsorption starts to be saturated and get the linear detection limit of the sensor, we added tests and got two sets of data at the concentrations of 5 and 2.5 ppm. Finally, we found that when the concentration is about 2.5 ppm, the change rate of capacitance reaches the maximum. Moreover, then the slope became smaller when we continued to increase the concentration.

To verify the reliability of the experimental results, we repeated the tests 3–5 times using new chips for each concentration of

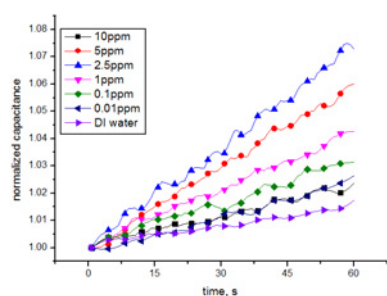


Fig. 6 Change rate of normalised capacitance with time

formaldehyde solution samples. As the blank control group, the capacitance change rate of DI water was also measured several times. We calculated the mean of values from each group. The average change rates of capacitance of different concentrations are shown in Fig. 7, where the error bars (black segments) represent the standard deviation of three test results.

It can be seen that the average test results are in good agreement with single test obtained before, and the blank control group without formaldehyde keeps lowest and stable capacitance change rate. These results indicate that our experimental method can reliably and effectively detect the formaldehyde molecules in DI water.

To confirm that formaldehyde can be detected in different solutions, we also performed tests of formaldehyde diluted in tap water and normal saline. Both types of solutions contained six groups of formaldehyde concentration such as former experiment. Also, the loaded voltage and AC signal frequency are the same. Fig. 8a shows the capacitance change rate in response to different concentrations of formaldehyde in tap water. Moreover, Fig. 8b

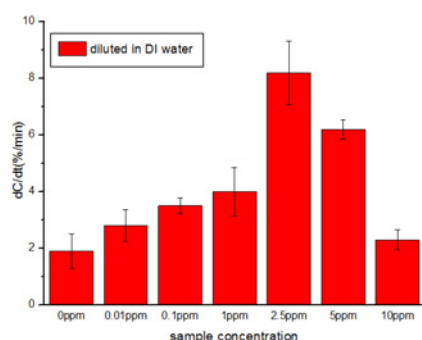


Fig. 7 Capacitance change rates versus different sample concentrations

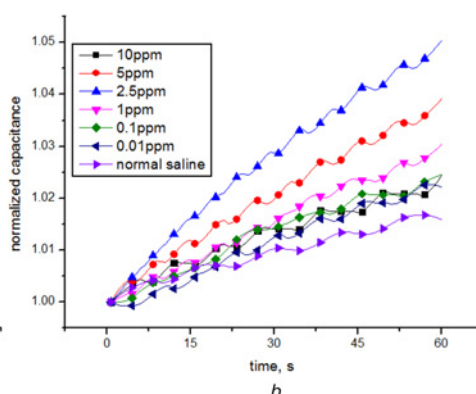
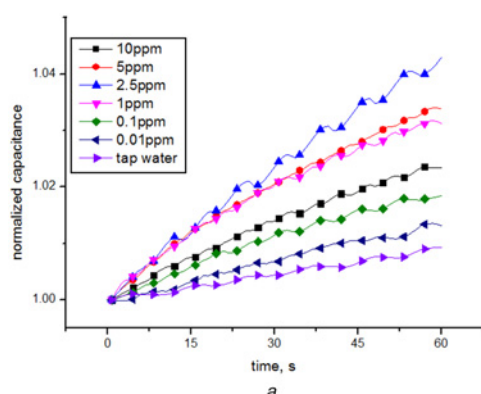


Fig. 8 Change rate of normalised capacitance with time
a Formaldehyde in tap water
b Formaldehyde in normal saline

gives the corresponding response in normal saline. Besides, curves of controls (blank without formaldehyde) are shown as the benchmarks in Fig. 8.

Similarly, to prove the reliability of our experiments, we made repeated measurements of each concentration and calculated the averages, which are shown in Fig. 9. Furthermore, the error bars from three tests are also given to provide consistency of different chips and tests in a certain group.

According to above results, we find the similar trend in detecting trace formaldehyde dissolved in different solutions. It indicates that for different common solvents, this capacitance sensing method based on ACEK can reliably and effectively detect trace amount of formaldehyde.

As for the microelectrodes we adopted, the change rate of capacitance is monotonically increased from 0.01 ppm until reaching a maximum of 2.5 ppm. Then with the concentration increase, change rate of capacitance will become smaller. So, if the concentration to be detected is below 2.5 ppm, formaldehyde can be detected rapidly and easily. However, if the concentration is above 2.5 ppm, it will be a challenge to judge whether the concentration is above 2.5 ppm or not only by measured data. In practical application, we can re-test the samples after appropriate dilution when we want to get the relative accurate concentration by inversion. If the change rate of capacitance becomes bigger than that of former test, the concentration of original solution should be between 2.5 and 10 ppm. Otherwise, the concentration of original solution should be lower than 2.5 ppm. In fact, high formaldehyde concentration detection is of no value for an innovation because there are already traditional methods to meet this requirement. Nevertheless, this presented capacitance method is a rapid and sensitive way to detect trace formaldehyde in solutions.

4.2. Effect from electric field and frequency: According to ACEK theory, the forces on particles increase with electric field strength. If the short 'sample to results' time is a result of EK enrichment, the capacitance change rate should exhibit a monotonically increasing dependence on applied voltage amplitude. Therefore, we selected a sample of formaldehyde in DI water at a concentration of 2.5 ppm and tested different amplitudes of 30 kHz AC signals for impedance assay. The experiments were repeated three times with voltage amplitudes from 25 to 200 mV_{rms}. The capacitance change rates were obtained and plotted in Fig. 10a as a function of voltage amplitude.

On the other hand, the ACEK impedance assay is expected to exhibit frequency dependence because DEP force has frequency dependent. In theory, the DEP property of target molecules will determine the frequency dependence of its adsorption to the electrode and further affect the magnitude of capacitance change.

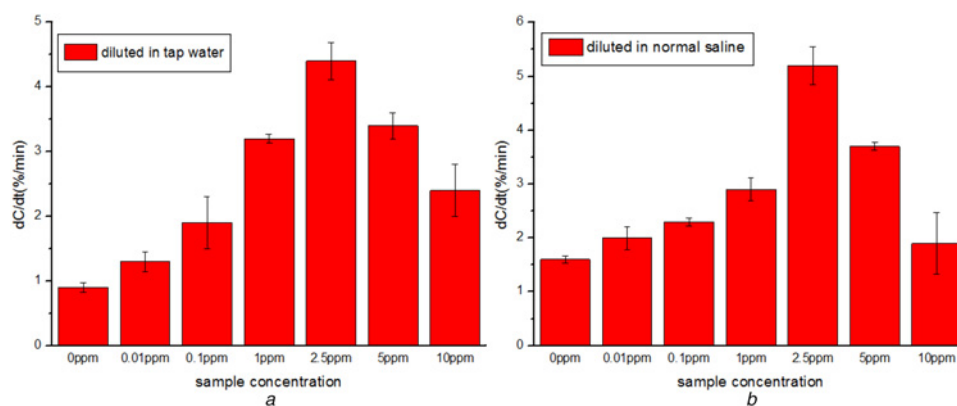


Fig. 9 Responding of capacitance change rate to formaldehyde concentrations
a Diluted in tap water
b Diluted in normal saline

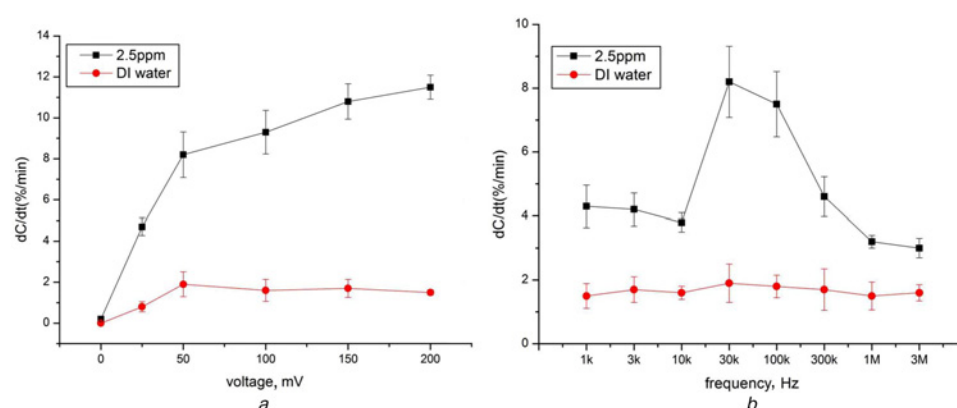


Fig. 10 Capacitance change rate dependence on voltage and frequency
a Capacitance change rates at different voltages
b Capacitance change rates at different frequencies

Capacitance change rates of formaldehyde in DI water at the concentration of 2.5 ppm were measured at various AC frequencies from 1 kHz to 3 MHz at 50 mV_{rms}. The measurements were repeated three times at each different frequencies, and the average capacitance change rates at different frequencies are given in Fig. 10b.

As the figure shows, the capacitance change rate increases rapidly when the applied voltage increases from 0 to 50 mV_{rms}. The increase of capacitance change rate slows down when the voltage rises above 50 mV_{rms}, which may be caused by possible adverse effect that electric field may have on the activity or adhesion of formaldehyde molecules on the electrodes. Nevertheless, the increase of capacitance change rate with voltage amplitude serves to corroborate the contribution of ACEK effect in this capacitive assay. The result in Fig. 10a indicates 50 mV_{rms} to be the optimal operating voltage of the ACEK impedance assay. Additionally, the control solution consistently exhibits low capacitance change in voltage range of 0–200 mV_{rms} at 30 kHz as shown in Fig. 10b, indicating that the capacitive response from formaldehyde solution sample is not caused by electrochemical reaction or other impedimetric artefacts in the measurements.

The capacitance change rate also shows obvious frequency dependence. The change rate reaches its maximum at about 30 kHz, and quickly drops when AC frequency deviates from this frequency. The bell-shaped frequency dependence clearly indicates DEP characteristics. When AC frequency is outside the optimal range, the capacitance change rates are still much higher than those of control sample. As a result, we chose the frequency of

30 kHz to perform all the tests, which could contribute to ACET microflows.

5. Conclusions: This work presents a trace of formaldehyde detection method based on ACEK effect. We obtained highly reproducible experimental results, and analysed the corresponding mechanisms during detection process. After enhancing ACEK effect to capacitance tests, it is possible to detect the rough concentration of trace formaldehyde within several tens of seconds. The detection range is from 0.01 to 2.5 ppm at 30 kHz and 50 mV_{rms} of AC signal. In conclusion, this ACEK capacitance detection method has advantages of short analysis time, low cost of reagents and equipment, simple operation and so on. It is more suitable for the detection of formaldehyde in low concentration compared with existing methods of formaldehyde detection. The sensing method reported here shows high potential to be an efficacious technology for soluble molecule detection.

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