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Fabrication of high sensitivity 2-PEA sensor based on Aldehyde-functionalized mesoporous carbon

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ABSTRACT

2-Phenylethylamine (2-PEA) is one of the main ingredients for stimulants. Therefore, it is necessary to limit its use and illegal trade by conveniently detecting 2-PEA vapour. Here, a QCM based 2-PEA gas sensor was constructed by using aldehyde functionalized mesoporous carbon (FDU-15-CHO) as sensing materials designed according to Schiff base adsorption role. The 2D hexagonal mesoporous structures of the sensing material have been confirmed by small-angle X-ray diffraction (SXRD), transmission electron microscopy (TEM), and N_2 adsorption-desorption isotherms. The covalent grafting of aldehyde group onto the FDU-15 was confirmed by Fourier transform infrared spectroscopy (FT-IR). FDU-15-CHO based Quartz Crystal Microbalance (QCM) sensor shows better sensitivity to 2-PEA than its parent FDU-15. Besides, the detection limit of FDU-15-CHO based sensor can reach down to 1 ppm, and its selectivity and reproducibility are satisfactory. The high concentrations of active sites in the mesopores of FDU-15 are believed to facilitate 2-PEA adsorption, and thus the presence of the -CHO group leading to sensitive and selective response, which is verified by Gaussian simulation

The illicit trade of drugs is a worldwide societal problem, associated with illegal business and organized crime. Due to the high price of strongly addictive drugs, drug abuse is responsible for crimes like shoplifting, burglaries and robberies [1]. The world's largest illicit drug product in volume terms is cannabis. Second is cocaine, followed by heroin [2]. The detection of organic amines which are the main ingredient of drugs has gained more and more interests, as many of volatile amines have been heavily used in diverse fields ranging from chemical to pharmaceutical and even to food industries [3,4]. Therefore, a variety of analytical methods have been established to detect organic amines, including thin-layer chromatography (TLC), gas chromatography/mass spectrometry (GC–MS), high-performance liquid chromatography (HPLC) and immunoassay method [5–8]. However, most of these methods aim to detect liquid phase species. 2-phenylethylamine (2-PEA) is a common organic amine, and belongs to the group of so-called trace amines, a family of endogenous amines [9,10]. Lawless persons often make use of 2-PEA as one of the raw materials to prepare drugs, such as hallucinogen [11]. Thus, there remains a great demand on a sensitive, selective and rapid on-site 2-PEA vapour detection.

Nowadays, many kinds of gas sensors have been developed for gas detecting [12–14]. amongst them, the mass-sensitive devices including bulk acoustic wave (BAW) and surface acoustic wave (SAW) become more and more popular due to the advantages of their high stability and good sensitivity. As a typical BAW device, quartz crystal microbalances (QCMs) can determine a small electrode-mass change down to the nano-gram level [15–17]. Sauerbrey has firstly derived the quantitative relationship between change of frequency of piezoelectric crystal Δf (Hz) and the mass change caused by mass loading on the piezoelectric crystal surface.

$$\Delta f = \left(-2.3 \times 10^6 \times f_0/A\right)\Delta m \quad (1)$$

where f_0 (10^6 Hz) is the basic frequency of the unloaded piezoelectric crystal, Δm (g) is mass change on the surface of the crystal, and A (cm^2) is the surface area of the electrode [18]. Therefore, researchers usually coat various sensitive materials on the electrode of QCM, as a transducer, to selectively detect different gases [19].

In addition, carbon nanomaterials, such as carbon nanotubes and graphenes, have been demonstrated as promising candidates for ammonia sensing due to their good chemical and thermal stability and charge transfer characteristic, and the sensing mecha-

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nism is considered to the charge exchange between carbon nanostructures surface and the absorbed ammonia molecules [20,21]. As an important kind of nanostructured carbon materials, ordered mesoporous carbon (OMCs) have gained increasing attention in the past ten years due to their outstanding physicochemical properties and their wide applications [22–25]. Because of their high surface area and large pore volume, OMCs can provide huge interface for the creation of active sites for interaction with ammonia vapour [26]. As a representative common OMC, FDU-15 has been used for molecular adsorption, electrocatalytic oxidation, *etc.* [27,28]. However, no work found so far to graft aldehyde group onto OMCs with open mesopores and rich active sites for 2-PEAdetection.

Herein, we report a facile synthesis of aldehyde group functionalized FDU-15 with two-dimensional (2D) hexagonal mesostructure. It was used as the sensing material for QCM transducer to detect 2-PEA vapour based on its aldehyde group molecule probes. We studied the effects of the pore size, pore volume, surface area, as well as the interaction between aldehyde probeand 2-PEA on the sensitivity, response time, and selectivity of the sensor. The results show that the gas sensor shows excellent sensing characteristics, including suitable sensitivity and stability for PEA vapour in a wide range of 10–50 ppm in air at room temperature (298 K). In addition, the sensor has desirable reproducibility, good selectivity, fast response and recovery quality. Besides, we used Gaussian software to simulate the adsorption enthalpy (ΔH) between aldehyde group of the material and 2-PEA molecule during the sensing process.

Nitric acid and dichloromethane were purchased from Shanghai Chemical Industrial Co., Ltd. Thionyl chloride and 2-phenylethylamine werepurchased from Macklin reagent. Triblock copolymer poly(ethylene oxide)-*block*-poly(propylene oxide)-*block*-poly(ethylene oxide) (Pluronic F127, PEO-106PPO70-PEO106, Mw = 12,600) was obtained from BASF. All chemicals were used as received without any further purification. Deionized water was used in all experiments.

Functionalization of ordered mesoporous carbons (the synthesis process of ordered mesoporous carbon is detailed in Support information): Through different oxidation treatments in the liquid phase, we modified the texture and surface chemistry of the mesoporous carbon to create functional groups. To create surface oxygen groups, reflux of the mesoporous carbon was carried out in 5 mol/L HNO_3 at 313 K for 3 or 4 h. Finally, it continued with filtering, rinsing with distilled water, and drying of the carbon supports at 381 K for 12 h. We stirred the carboxylated OMCs (150 mg) at 70 °C for 24 h in a 30 mL solution of a 20:1 mixture of thionyl chloride ($SOCl_2$) and dimethylformamide (DMF). Through vacuum distillation, we removed $SOCl_2$ and DMF after acyl chlorination.

We added *p*-hydroxybenzaldehyde with a mole ratio of 1%, 3% and 5% to evaluate its reaction with acyl-chlorinated OMCs at 30 °C for 12 h to 2 d. Then, to remove the excess *p*-hydroxybenzaldehyde, the OMC samples were repeatedly washed with ethanol for five times. Finally, the black solid was dried overnight at room temperature in vacuum. The resulting chemical reactions are illustrated in Fig. 1.

The X-ray diffraction (XRD) data was collected by a Dmax 2500 V diffractometer, using Cu Kα radiation as X-ray source ($\lambda = 1.5418$ Å) with a scan speed of 0.5°/min. Transmission electron microscopy (TEM) images were recorded on a JEOL JEM-2010F electron microscope operated at an accelerating voltage of 120 kV. Before TEM observation, the samples were first sonicated in ethanol solution and then drop-cast onto a carbon-coated copper grid and dried under air. Fourier transform infrared spectroscopy (FT-IR) was analysed by AVATAR 370. Nitrogen sorption isotherms were measured at 77 K by using a Micromeritics ASAP 2020 system, about at least 0.1 g of sample was degassed at 373 K for 12 h.

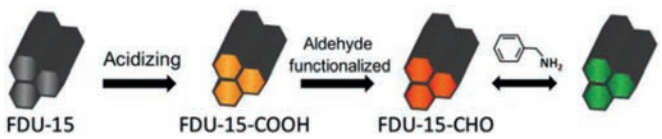

Fig. 1. Scheme of the experiment.

The QCM technique was employed for detection of the sorption/desorption behaviour of the synthesized materials to analyte vapour. The chemical sensing experiments were performed by using a modified setup that we reported before [26]. In order that they could act as electrodes, the QCM resonators with silver electrodes were purchased from Chenjing Electronic Co., China. The resonance frequency was 10^7 Hz(AT-cut) and frequency decreased (ΔF) proportionately with an increase in mass (Δm) according to the Sauerbrey equation [27]. To get the QCM sensors based on sensing material, the material was first stabilized as water suspensions, and then dropped onto a QCM electrode, followed by heating the QCMs in a furnace at 50 °C in a vacuum drying oven. After evaporation of the solvent, the as-made QCMs were yielded. Prior to a measurement, the QCM sensors were exposed to nitrogen stream until a stable baseline was established. The liquid analyte was introduced by injection. The analyte vapour was generated from the liquid analyte by a heater. The exact volume of the injected analyte liquid was calculated in μL based on the following equation: $V_l = cVM10^{-6}/22.4dp$, where V_l represents the volume of liquid in μL, c is the liquid concentration in ppm, M represents the molecular weight, d is the liquid density, and p represents the degree of purity. The QCM sample crystal was exposed to the analyte vapour until a stable frequency shift was obtained. At the end of each cycle, continuous nitrogen stream would wash away the analyte and re-establish the baseline. The response value is the negative number of frequency shift value. A schematic diagram of the 2-PEA-sensing system is presented in Fig. S1 (Supporting information).

Gaussian 09 software was used to calculate the ΔH of the sensing process [28]. DFT method and the hybrid B3LYP functional and 6–311, G(d, p) basis set was used to optimize the geometries of the calculated molecules. The optimized structures were confirmed to be minima in the potential energy surface *via* vibration frequency analysis. The interaction energy between 2-PEA molecule and sensing molecule probe was calculated according to the following equation.

$$\Delta H(\text{kJ/mol}) = H_{\text{sensing robe}-2-\text{PEA}}(\text{kJ/mol}) - H_{\text{sensing probe}}(\text{kJ/mol}) - H_{2-\text{PEA}}(\text{kJ/mol}) \quad (2)$$

The characterization of aldehyde-functionalized FDU-15. Three well-resolved diffraction peaks are demonstrated by the small-angle XRD patterns of FDU-15, FDU-15-CHO-2 and FDU-15-COOH (Fig. 2A). These peaks could be indexed to (100), (110) and (200) reflections, which indicate the presence of the ordered mesostructure [29]. Since the functional groups of –CHO and –COOH are attached to the wall inside the pores; the order degree of pure materials is reduced. Additionally, these functional groups cause the peak intensities of unfunctionalized mesoporous materials to be higher than that of the functionalized ones. There are three diffraction peaks at (100), (110) and (200) reflections that are non-overlapping or well resolved. The indexing of these demonstrates consistency with our previous reports, which specified that in these materials, the presence of a long-range mesostructure is evident. This may result from the increased organic groups functionized in the carbon wall and the pores that decreased the long range ordering of the mesoporous carbon. Furthermore, with increasing number of aldehyde groups, the intensity of the 100 diffraction peaks gradually decreased (Fig. S2 in Supporting information) The TEM images shown in Fig. 2B also confirm that FDU-

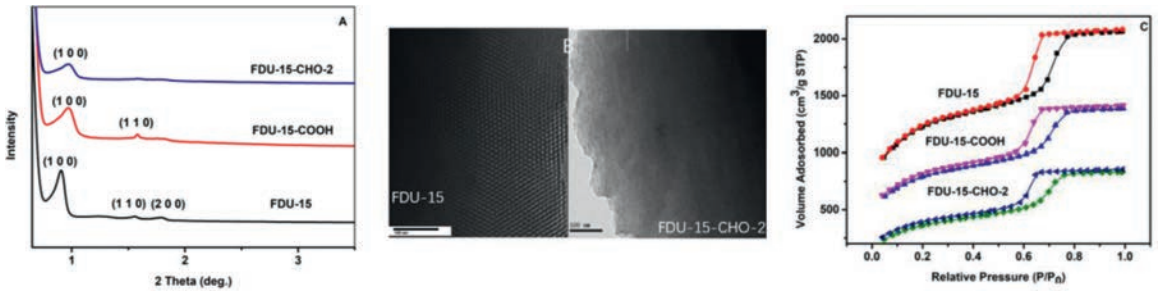

Fig. 2. (A) Small angle powder X-ray scattering patterns of FDU-15, FDU-15-COOH and FDU-15-CHO. (B) TEM images of FDU-15 and FDU-15-CHO. (C) N_2 adsorption-desorption isotherms of FDU-15, FDU-15-COOH and FDU-15-CHO.

15-CHO with high degree of ordered porous texture are obtained the synthesis.

N_2 desorption isotherms (Fig. 2C) of the three samples FDU-15, FDU-15-COOH and FDU-15-CHO show typical type-IV curves, with a distinct condensation step and a hysteresis loop. The surface area of BET specific surface decreased from 2994 m^2/g (FDU-15) to 790 m^2/g (FDU-15-CHO-2). The –CHO embedding on the FDU-15 channel's internal surface confirms these results. To explore the textural properties of these FDU-15-CHO materials, we investigated the increase in –CHO functionalization on the inner wall of the pores and evaluated the decreased ordering for the mesoporous carbon. These results are presented in Fig. S3 (Supporting information). The FT-IR spectra for FDU-15, FDU-15-COOH and FDU-15-CHO display several characteristic bands. At 3433 cm^{-1}, the broad band is mainly assigned to the O–H stretching vibrations of adsorbed water (Fig. S4 in Supporting information). There is a possible chance that for the small amounts of quinine, C=O and C–H groups of carbon materials and the C=C groups on the surface aromatic structures, there is an existing ascription of the weak bands at 1643 cm^{-1}, 1490 cm^{-1} and 1412 cm^{-1}. There is also an attribution of the new intense band at 2750 cm^{-1} to the carbonyl of nonaromatic carboxyl groups after the modification. To imply that numerous oxides are generated on the surface, the bands at 1228 cm^{-1} and 1005 cm^{-1} can be ascribed to the C–O bonds of phenolic, carboxylic, etheric and ester groups. The Raman measurement results show the D-band at 1360 cm^{-1} is due to defects in the carbon materials and disorder. The G-band at 1580 cm^{-1} is produced by all the sp^2 atoms in the carbon material. The area ratio of the D-band and G-band for the two materials is almost unchanged, indicating that FDU-15 has good crystallinity. The arrangement of carbon atoms is hardly affected by functional grafting. The detailed data is in the support information Fig. S5 (Supporting information).

We further investigated the response of the powdered samples towards 2-PEA vapour after our evaluation of their chemical nature. Additionally, the QCM sensor was installed into a testing chamber, which was kept at a constant temperature of 298 K and relative humidity of 45%–55%. The frequency response of the QCM sensors at 50 ppm 2-PEA vapour, as shown in Fig. S6 (Supporting information). Also, the stability of the QCM sensors coated with the FDU-15-CHO was evident. Regarding the increase of aldehyde group, the sensor cannot be restored, because the surface area and pore volume of the material decrease with the increase of aldehyde group. In the presence of 50 ppm 2-PEA vapour, Fig. 3A shows how the FDU-15-CHO-2-based QCM sensor has a frequency response that is time-dependant. Within 100 s, the frequency shift was less than 650 Hz, which the highly sensitive behaviour of the QCM sensor coated with aldehyde-functionalized FDU-15 to 2-PEA. As a comparison, the other FDU-15-based sensors were also studied for 50 ppm 2-PEA. In this case, the sensor's frequency shift is much smaller than that of the FDU-15-CHO-2-based QCM sensor, which is attributed to the aldehyde groups of FDU-15. Additionally, the FDU-15-CHO to 2-PEA response time is approximately 10 s, while the recovery time is approximately 11 s. For 45 days, we exposed the sensor to air for the purpose of testing the sensor's long-term stability. After a series of continuous tests for 45 days, all of the responses showed a slight decrease, as demonstrated in Fig. 3B. This indicates that all of the sensors had notable stability. The typical sensor response to 2-PEA vapour at a concentration of 1–100 ppm is exhibited in Fig. 3C. Evidence that supports how the 2-PEA sensor's detection limit can reach 1 ppm is presented by the frequency shift higher than 10 Hz at 1 ppm. Then, we measured the frequency response values for the FDU-15-CHO-2-based QCM sensor under varying relative humidity conditions upon exposure to 50 ppm 2-PEA gas. The temperature is maintained at 298 K in all of the experiments. Fig. 3D presents the exposure of the QCM sensor to 50 ppm 2-PEA, and the maximum frequency response values at relativity humidity levels of 15%, 20%, 25%, 30%, 35%, 40%, 48%, 53%, 60%, 65%, 70%, 75%, 80%, 85%, 90%, 93%, 95% and 98% RH are 500 Hz, 510 Hz, 525 Hz, 530 Hz, 538 Hz, 545 Hz, 551 Hz, 558 Hz, 565 Hz, 577 Hz, 590 Hz, 608 Hz, 620 Hz, 638 Hz, 645 Hz, 668 Hz, 672 Hz and 680 Hz, respectively. Under dry air, the frequencies are 400 Hz, 414 Hz, 435 Hz, 438 Hz, 448 Hz, 455 Hz, 463 Hz, 471 Hz, 488 Hz, 510 Hz, 534 Hz, 543 Hz, 558 Hz, 567 Hz, 578 Hz, 586 Hz, 593 Hz and 630 Hz, respectively, which indicates that at the same

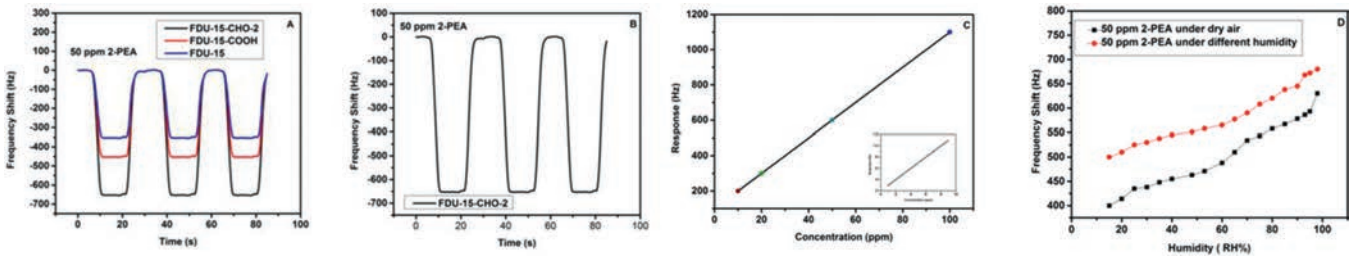

Fig. 3. (A) Short-term retability and reversible rapid sensing response curves of FDU-15, FDU-15-COOH and FDU-15-CHO based QCMs to 50 ppm 2-PEA. (B) The long-time frequency stability at 50 ppm 2-PEA for the sensor at 298 K. (C) The response of FDU-15-CHO towards 2-PEA with different concentrations. (D) Responses of the FDU-15-CHO based sensor under different humidity and dry air of 50 ppm 2-PEA.

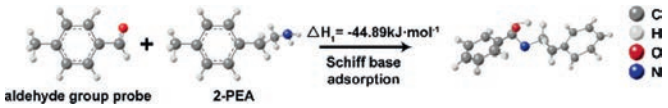

Fig. 4. Optimized geometries and interaction between aldehyde group probe and 2-PEA molecule.

concentration, the frequency response towards 2-PEA increases by a small amount with increasing relative humidity. The aldehyde group produces satisfactory humidity resistance results. Our material's gas sensing mechanism towards 2-PEA can be assigned to a multistage Schiff base mechanism. The comparison of the individual frequency responses in other interfering gases supported the investigation of selectivity. These gases are 2-PEA, acetone, H_2S, CO_2, NO_2, CO and CH_4. The concentration of all of the gases is 50 ppm. The individual responses of the FDU15-CHO based sensor towards these gases are shown in Fig. S9 (Supporting information). The response to 2-PEA is much higher than that for other gases, which is evidently attributed to the definite interactions occurring between the functional group and the $-NH_2$ group of 2-PEA vapour. The 50 ppm 2-PEA reaches a response at 680 Hz. The largest response to 50 ppm acetone vapour reaches approximately 182 Hz for all seven interfering gases. However, at the same concentration, it is significantly less than that of 2-PEA. This result demonstrates the reasonable selectivity of the FDU-15-CHO-based QCM sensor for 2-PEA. As discussed above, it can be concluded that a good candidate for sensing 2-PEA vapour is the well-defined aldehyde-functionalized FDU-15. We believe that the sensing performance depends on two factors: one is the provision of the mesoporous FDU-15 structure with numerous active sites, which is understood to assist in gas adsorption. The second is known as the presentation of aldehyde groups, which works in combination with the textural properties of the inorganic host matrix (FDU-15). In the context of 2-PEA, this allows improvements in selectivity. In mesoporous structures, the visible existence of –CHO donors lead to advancements in the sensor capabilities of 2-PEA. We consider that the main reason for 2-PEA sensing in our system is the Schiff base mechanism [16].

The response of FDU-15-CHO-2, FDU-15 and FDU-15-COOH toward chlorobenzene, toluene, *o*-xylene, and benzene should be attributed to π-π conjugation interaction between aromatic ring structure of analytes and OMC structure. The response disadvantage of FDU-15-CHO relative to FDU-15 toward chlorobenzene, toluene, *o*-xylene, and benzene should be attributed to the aldehyde group which can block these aromatic compounds. Besides, the response of FDU-15-CHO towards 50 ppm 2-PEA is larger than that of FDU-15. This group of experiments shows that the –CHO group of FDU-15-CHO has a better adsorption capacity for the amino group containing aromatic structure than other aromatic structures (Fig. S7 in Supporting information).

We apply the Gaussian 09 software to increase our understanding of the 2-PEA's sensing mechanism and simulate 2-PEA's molecular adsorption energy to the top of the aldehyde group probe of FDU-15 (-CHO). A Schiff base interaction amongst the aldehyde group (-CHO) of aldehyde functionalized FDU-15 and the amino group (-NH_2) of 2-PEA establish the adsorption process. The reversible Schiff base adsorption's enthalpy change (ΔH) is calculated to be −44.89 kJ/mol, as shown in Fig. 4. This change indicates that between the aldehyde functionalized FDU-15 and the 2-PEA molecule, the adsorption is moderately selective and reversible [30]. Therefore, to effectively detect 2-PEA, we use an ideally selective and reversible sensing material that is known as the aldehyde functionalized FDU-15.

After functional group grafting, the aldehyde-functionalized FDU-15 was successfully synthesized. The immediate response and notable selectivity towards the 2-PEA vapour are features of the FDU-15-CHO based QCM sensor, which occurred due to the amalgamation of the amino aldehyde Schiff base adsorption. The results indicated that the materials kept their mesoporous nature after grafting of the aldehyde groups. Gas-sensing tests were investigated by coating the aldehyde-functionalized FDU-15 onto quartz crystal microbalance transducers. The results reveal that the sensors can be as promising candidates as 2-PEA detectors with desirable sensing behaviors, including high sensitivity (the QCM sensors were exposed to nitrogen stream until a stable baseline (±2 Hz/min and the limit detection is 1 ppm), fast response (response time is and the recovery time is approximately less than 15 s) and good reversibility. For 45 days, we exposed the sensor to air for the purpose of testing the sensor's long-term stability. The QCM sensors coated with the aldehyde-functionalized FDU-15-CHO were very sTable 2-PEA. Also, the detection limit reaches 1 ppm. Gaussian 09 simulations, indicating that aldehyde-functionalized FDU-15 was suitable for 2-PEA detection owing to the moderate ΔH value of Schiff base adsorption.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2022.03.114.

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