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The Integrated Mini GC-PID System for Monitoring Air Pollution

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<http://dx.doi.org/10.5772/59725>

1. Introduction

Volatile organic compounds (VOCs) are the premise of pollutants for forming PM_{2.5} or heavy haze and choking smog, so monitoring VOCs is a very important measure for preventing environmental pollution. In addition, rapid detection of VOCs is also very necessary in some on-site detection, including indoor air quality, industrial emissions control, air pollution emergency in urban area, rapid detection of explosives in public place, and breath analysis. Therefore, fast, on-site, high-precision, and portable systems are the pressing need for providing real-time monitoring for quantification and identification analysis of environmental samples.

Conventional gas chromatography (GC), which is a powerful and commonly used analytical technique, has become the premier technique for separation and analysis of harmful compounds due to its high resolving power. However, in some cases, gas samples need to be collected in the field and analyzed in the laboratory using conventional GC, which makes this process inefficient and inconvenient in field use.

As MEMS technology matures, micro gas chromatographic systems (μ GC) [1-3] have a prospect future with some advantages of small volume, rapid analysis, and less power consumption compared with conventional GC systems, which are very urgent to be used for analyzing the complicated gas mixtures. Therefore, these attributes make these systems based on microelectromechanical systems (MEMS) technology attractive for a number of applications involving on-site monitoring of environmental samples, and this research direction is under intensive research.

In this chapter, we reported the design, fabrication, and characterization of micro pre-concentrator, micro GC column, and mini photoionization detector (mini PID). Then a mini

GC system based on mini PID was integrated with the micro GC column and the micro pre-concentrator.

2. Schematic representation of the mini GC system

A mini (portable) GC system usually includes an injection unit, a micro pre-concentrator, a micro GC column, and a mini PID. Figure 1 shows schematic of the mini GC system for environmental monitoring and analysis.

Figure 1. A schematic representation of the mini GC system architecture

The injection unit supplies sample to the pre-concentrator or the GC separation column. The pre-concentrator is a sampling and concentrating device that can significantly improve detection limit of mini GC system with 1~3 orders. The GC separation column separates the complicated mixtures on the basis of their affinity with the stationary phase distributed all along the column length. The detector acquires the signals provided by the separated components at the GC column outlet.

The mini GC system is characterized by a series of innovations. 1) The micro dryer and purifier can remove vapor and particulates in environmental sample. 2) The integrated pre-concentrator can make the system easy to deploy trace gas and suitable for in-field use. 3) A micro GC column instead of a conventional column was used to separate mixtures, which can greatly reduce volume of the system.

3. Micro dryer and purifier

Environmental samples do not only have a very low concentration but also contain vapor and various particles, which will make GC column and detector inactive or even fail. Therefore,

the proposed mini GC system was integrated with a micro dryer and purifier which can clear up the vapor and micro pollutants in the samples.

In order to rapidly and effectively eliminate the vapor from the environmental samples, two kinds of micro-pillars were fabricated in micro flow channel of the dryer. The shape of the former pillar is shown in Figure 2(b) (refer to the I pillar), which can break and rapidly atomize the water droplets in the samples. The second pillar (refer to the II pillar in Figure 2(b)) has a relatively large surface area, which can completely come into contact with the micro droplets and then quickly vaporize them.

Fabrication of the chip used silicon wafer and Pyrex 7740 glass wafer as the substrates. The patterns of the dryer and purifier were formed by lithography and fabricated through a series of lithography, etching, and bonding process, where the channels and pillars were fabricated using a deep reactive-ion etching technology. For details on the fabrication process of the micro dryer and purifier, refer to the work in [4]. The length of the chip is 10 mm and the depth and width of the dryer channel were 400 μm and 500 μm , respectively. In order to clear up the vapor, molecular sieve 5A (60–80 mesh) was used as adsorption material and filled in the channel of the micro purifier. In order for the dryer to have a fast self-heating capacity, a micro heater was integrated on the backside of the dryer. The heater with resistance of 8 Ω was fabricated as a 20 nm/250 nm Cr/Pt stack deposited by a magnetron sputtering technology and patterned by a lift-off technology, which can increase the temperature at a speed of 10°C per second and the highest temperature can be raised up to 200°C in less than 30 s. Figure 2 shows the proposed micro dryer and purifier.

Figure 2. The fabricated micro dryer and purifier (a) and micro-pillars in channel (b)

In order to evaluate the performance, one end of the device is connected to a sampling pump, and the other end is connected to a transparent pipe filled with anhydrous CuSO_4 . Firstly, an environmental sample containing a certain amount of water vapor was passed through the device which is in working state. After being processed by the device, the anhydrous CuSO_4 in the pipe did not change in color, which verifies that vapor was completely absorbed by the device.

4. Micro pre-concentrator

After removing vapor and particulates in environmental samples by the micro dryer and purifier, the sample can be introduced into a pre-concentrator for further processing.

The principle of the gas concentrator was detailed as follows: Analytes were transported into the pre-concentrator through a sampling pump, and the analytes were absorbed by the adsorption material. After the adsorption capacity was saturated, the pre-concentrator was heated to release the absorbed components. Finally, the released components with high concentration were transported into the detector.

The pre-concentrator can concentrate trace gas, that is, makes its concentration reach or exceed detection limit of the detector and makes the detector yield a good response for trace environmental sample. Therefore, pre-concentration of sample prior to analysis as it enables detection of trace gas and also improves detection sensitivity of mini-instruments, and sample pretreatments occupied in an important position for on-site rapid detection.

The conventional pre-concentrator configurations consist of a trap metal tube that is cooled by a flow of cold gas during sample collection and successively heated in order to release the sample through a rapid thermal desorption mechanism. However, the typical pre-concentrator needed a high power due to its larger thermal mass. Moreover, the conventional pre-concentrator is very difficult to be integrated into a portable GC system, resulting in an inconvenient in-field application.

Micro pre-concentrator with reduced thermal mass can raise temperature much faster at lower power compared with conventional desorption tubes, realizing a much higher concentration factor. Micro pre-concentrators with a small size and high concentration efficiency were easily integrated into micro/portable chromatographic systems, which make these systems very suitable for on-site or online analysis. Therefore, micro pre-concentrator received an unprecedented development with the development of MEMS technology.

Many efforts to improve concentration efficiency have been made in many works [5-13], and these efforts included the use of promising materials for high adsorption capacity, fabrication of high aspect ratio of the channels, improvement of sample capacity by fabricating multi-channels, and fabrication of micro channels with embedded micro-pillars which are able to fill more sorbent material and increase high aspect ratio of the channels. Although these efforts have yielded important progress, the released components during thermal desorption process should be controlled and the peak broadening also should be compressed.

4.1. Consideration of the micro pre-concentrator

As concentration of components in environmental gas is very low (ranged from several ppb to hundreds ppb) and the available analytical instruments are not sufficiently sensitive, the development of micro pre-concentrators for effectively concentrating components is very important and necessary.

According to theories of the pre-concentrator, there are several methods for improving concentration factor: (1) optimization of device structure for increasing the adsorption capacity; (2) the use of efficient adsorption material for improving specificity and adsorption capacity; (3) integration of cooler and heater on the pre-concentrator for improving adsorption capacity and desorption efficiency, respectively; and (4) integration of a micro valve behind the pre-concentrator for closing the released components during the desorption stage.

Therefore, in order to improve the concentration factor, in this work, we optimized the above considerations including measures as follows: the use of nanomaterials as adsorbent materials, integration of a heater and cooler on the pre-concentrator, and integration of a valve behind the pre-concentrator.

4.2. The adsorption material

The adsorption material is a key factor for affecting performance of a pre-concentrator device. Firstly, the material characteristics determine the types of concentrated gas. Secondly, material characteristics also determine the adsorption capacity of samples. Generally speaking, in order to improve the concentration factor, the first consideration is the selectivity and specificity of adsorption material, and then the adsorption capacity of the material is the next consideration.

With the development of technology, there are more adsorption materials that are available than before. In addition to the traditional adsorption material, such as Tenax-TA, Carboxen X, and activated carbon, recently some nanomaterials were developed and widely used as adsorption material, such as carbon nanotubes, molecularly imprinted polymers, nanoparticles, and so on.

Single-walled carbon nanotubes (SWCNTs) have a lot of advantages, such as large aspect ratio, high effectiveness of surface area, chemical and thermal stability, a large affinity to nonpolar compounds, and higher adsorption capacity than Carboxen X. Moreover, SWCNTs cannot be dissolved in water and organic solvents. Therefore, SWCNTs were the preferred material used as adsorbent material.

4.3. Fabrication of micro pre-concentrator

In this work, in order to improve the concentration factor of the pre-concentrator, a micro pre-concentrator with four parallel channels filled with SWCNTs as adsorbent materials was developed for concentrating VOCs in environmental samples. The details of the fabrication process of the pre-concentrator are the following (refer to Figure 3). Firstly, configuration of the micro pre-concentrator was drawn by AutoCAD software. Subsequently, rectangular micro channels were etched on silicon and glass wafer by laser dicing saw according to the configuration, and the length, depth, and width of the channels were 20 mm, 400 μm , and 1000 μm , respectively. Then these channels on silicon were aligned and bonded to these channels on glass wafer.

In order to increase adsorption capacity as much as possible in adsorption process, a cooler was integrated upside of the pre-concentrator, and the temperature of the pre-concentrator

Figure 3. The details of the fabrication process

can be cooled down to 5 °C in 100 s. In the desorption stage, the faster the increase of the temperature, the higher concentration factor of the pre-concentrator. Therefore, a micro heater was integrated on the backside of the pre-concentrator, which can quickly release the analytes. The heater with resistance of 8 Ω was fabricated as a 20 nm/250 nm Cr/Pt stack deposited by a magnetron sputtering technology and patterned by a lift-off technology. The heater can rapidly heat the pre-concentrator at a speed of 10°C per second, and the highest temperature can be raised up to 200°C in less than 30 s, which the instantaneous concentration factor can be significantly improved. Furthermore, to compress peak broadening and further improve the concentration factor, a micro valve (purchased from Shenzhen Keyto Fluid Control Co., Ltd) was integrated behind the pre-concentrator (refer to Figure 4), which can close the released components during thermal desorption process, thus increasing the concentration factor as maximum as possible. In addition, four micro filters integrated in the end of the channels can prevent SWCNTs out of the pre-concentrator from the outlet. A photograph of the fabricated micro pre-concentrator is shown in Figure 5.

Figure 4. The diagram of the pre-concentrator

Figure 5. The channels on the glass wafer (a) and silicon wafer (b), the fabricated micro pre-concentrator (c)(d)

4.4. Filling of the adsorbent material

The details of filling process were reported as follows: (1) SWCNTs were added into TNWDIS solvent for forming suspending agent; (2) one end of the column was connected to a capillary which was submerged into a SWCNT solution, and the other end of the pre-concentrator was connected to a micro-pump. (3) The SWCNT solution was transported into the channels after the pump was turned on, and then nitrogen gas was continuously delivered through the channel to completely evaporate the TNWDIS solvent. (4) Then the pre-concentrator was put into a temperature-programmed oven under a nitrogen flow inside, and the temperature was increased gradually by 5 °C/min until 200 °C and kept for at least 4 h.

4.5. Characterization of the micro pre-concentrator

To improve the concentration factor, a cooler and a heater were, respectively, integrated on the upside and backside of the micro pre-concentrator. The cooler and heater cover the whole pre-concentrator chip region (i.e., the channel region) which has a temperature distribution over the chip surface as uniform as possible. The low temperature can increase adsorption capacity as much as possible in adsorption state, and the high temperature can rapidly and completely release the analytes from the adsorbent materials.

To evaluate the temperature characteristics of the micro pre-concentrator, a measurement cycle of 200 s was considered, and the result is reported in Figure 6. As can be disclosed, temperature of the pre-concentration chip can be dropped down to 5 °C in 100 s in the state of cooling and then can be raised up to 180 °C after 150 s and successively settles to a steady value of 200 °C in the heating state.

Figure 6. Temperature characteristics of the micro pre-concentrator

In order to evaluate the concentration factor of the fabricated micro pre-concentrator, tests of adsorption and desorption of analytes were carried out (refer to Figure 7). Firstly, the analytes were introduced into the micro pre-concentrator using a sampling pump, and the analytes were absorbed by the SWCNTs at the temperature of 5 °C (the cooler is at cooling state) until the adsorption saturation. Then, the micro pre-concentrator was heated, but the desorbed analytes from SWCNTs were closed in the channels by the control of the valve. When the analytes were completely desorbed, the analytes were consequently carried to a laboratory-made portable photoionization detector (PID) [14] by carrier gas after the valve was opened.

In order to evaluate capabilities of the micro pre-concentrator, the sample composed of benzene with concentration of 500 ppb was used to be concentrated. The setup of the experiment is shown in Figure 7, where the outlet of the pre-concentrator was connected with a micro valve. Firstly, an amount of sample was transported into the pre-concentrator by opening the sampling pump. In this adsorption state, the cooler was working and the pre-concentrator was in cooling state; the sample was adsorbed in the SWCNTs (acted as adsorbent material). After the sample was completely adsorbed, the micro valve was closed and the micro heater was in working state; the analytes were quickly released from the SWCNTs by rapidly increasing the temperature of the pre-concentrator. After the analytes were completely released, the micro valve was opened, and the released analytes were consequently transported into the PID by carrier gas. Compared with the response without the proposed pre-concentrator (refer to Figure 8), the response with the pre-concentrator was significantly improved over 20 times more. Moreover, the chromatography peak broadening was greatly compressed, and peak tailing was satisfactorily solved. Therefore, the micro-fabricated pre-concentrator can be easy to deploy and suitable for a number of applications involving on-site monitoring of environmental samples.

Figure 7. The setup for evaluating performance of the micro pre-concentrator

Figure 8. The performance of the pre-concentrator

5. Micro GC column

With more and more demands for online monitoring of environmental sample, high sensitivity of portable GC instruments were very urgent to be used. However, the development of the portable GC systems was limited due to the large volume, high power consumption of traditional GC column. As MEMS technology matures, the micro GC columns [15-19] have a prospect future with some advantages of small size, rapid analysis, batch production, and less power consumption. The portable system integrated with micro GC columns can provide real-time monitoring for quantification and identification analysis of environmental sample. Therefore, these attributes make micro GC columns attractive for a number of applications.

In this work, a high-separation-efficiency micro-fabricated GC column embedded with micro-pillars was developed. These embedded pillars can significantly improve the overall surface area of the columns and reduce the effective width of the column, which can enhance the sample capacity and obtain an excellent separation performance.

5.1. Column fabrication

In this work, an (100) n-type silicon wafer and Pyrex 7740 glass wafer were used as the substrates. The proposed column was fabricated through a series of lithography, etching, and bonding process, where the channels and pillars were fabricated using a deep reactive-ion etching technology. For details on the fabrication process of the micro column, refer to this work [20]. Figure 9 shows a photograph of the fabricated GC column and SEM of the channel and pillars. The diameter of pillars is 50 μm ; the depth and the width of the micro channels were 350 μm and 300 μm , respectively; and the length of the micro column is 2 m. Therefore, the overall surface area of the columns, which was able to support the stationary phase, is over three times larger than that of open rectangular columns with the same dimensions, leading to higher column efficiency.

In order to separate the sample, OV-101 was acted as the stationary phase, and the stationary phase was coated via a static coating procedure. The coating process was shown as follows:

1. Preparation of the coating solution. Stationary phase OV-101 with a mass of 10 mg was dissolved in n-pentane and dichloromethane solvent (the volume ratio of n-pentane and dichloromethane solvent is 1:1), and the coating solution concentration is 15 mg/ml.
2. Coating of the column. The coating solution was transported into the column by a micro-pump, and the column was kept in a water bath at 40 °C. After the column was filled with coating solution, one end of the column was sealed by wax, and the excess solvent is slowly evaporated by the pump from the other end of the column.
3. Aging of the column. The fabricated column was put into a temperature-programmed oven which was protected by a nitrogen flow, and the temperature of the column was successively increased to 80 °C and 120 °C for 2 h to harden the stationary phase film; then the temperature of column was heated up to 220 °C for 4 h to cure the stationary phase film, leaving behind a layer of thin and uniform stationary phase film with a thickness of about 0.20 μm on the column walls.

Figure 9. (a) (b) The channels embedded with micro-pillars, (c) the heaters integrated on the GC column, and (d) photograph of the fabricated GC column

5.2. Column efficiency

There are many factors to evaluate the performance of GC column, such as the separation resolution, theoretical plate number, and separation speed, but the theoretical plate number can basically determine the separation performance of GC column. According to theory of chromatography, theoretical plate number and height equivalent to a theoretical plate (*HETP*) have a reciprocal relationship. Therefore, in order to evaluate separation efficiency of the proposed GC columns, we carried out a detailed theoretical analysis on *HETP*. According to previous reports, the *HETP* can be expressed in Equation 1 [21].

$$HETP = 2 \frac{D_g}{u} f_1 f_2 + \left(\frac{1 + 9k + 25.5k^2}{105(1+k)^2} \frac{w^2}{D_s} \frac{f_1}{f_2} + \frac{2kd_f(w+h)^2}{3D_s(1+k)^2 h^2} \right) u \quad (1)$$

where D_g and D_s are the binary diffusion coefficients in the mobile and stationary phases, respectively; d_f is the stationary phase thickness; w and h are the channel width and height,

respectively; and f_1 (varies between 1 and 1.125) and f_2 (varies between 0 and 1) are the Gidding-Golay and Martin-James gas compression coefficients, respectively.

The theoretical analysis of curve in Figure 10 shows height equivalent to a theoretical plate versus average carrier gas velocity from equation 5.1. The D_g and D_s were considered as 0.093 cm²/s and 6.4×10^{-6} cm²/s, respectively. k , h , and w were 3, 350 μ m and 150 μ m in the calculations, respectively. The minimum HETP value, H_{min} , found at the optimal average carrier gas velocity, u_{opt} , gives the maximum number of theoretical plates N . The column yielded a minimum HETP of 0.011 cm (9,100 plates/m) at a linear gas velocity of 18 cm/s.

Figure 10. Height equivalent to a theoretical plate versus average carrier gas velocity

6. High-sensitivity PID

In the developed portable GC system, the PID is the core elements, which can provide qualitative and quantitative analysis for air pollution. Therefore, it is very important for developing a high-sensitivity mini PID.

6.1. Performance optimization of PID

In general, there are several factors affecting its sensitivity, such as background noise, volume of the ionization chamber, and electron collection efficiency.

Firstly, it is common knowledge that background noise will affect the detection limit and detection sensitivity of the sensor; even large noise will cause failure of the detector. Therefore, reduction of background noises is the primary consideration in PID configuration.

In terms of ionization chamber, the large ionization chamber cell will increase the dead volume of the detector, which will greatly reduce the response sensitivity of the detector. In addition, larger ionization chamber will consume more carrier gas, which will greatly reduce the working time of portable GC system in field use.

Electron collection efficiency directly influences the response sensitivity of the detector, and the low collection rate will greatly reduce the sensitivity and detection limit of detector. Therefore, maximum collection rate is an important consideration for designing PID.

Figure 11. Schematic of the proposed PID

In order to develop a high-sensitivity PID, in this work, a mini PID with a lower background noise and a shorter response time was proposed. These important factors mentioned above were optimized for fabricating PID sensors. Figure 11 shows a side view of the PID proposed in this work. In this setup, a nozzle with a volume slightly less than the ionization chamber is parallel to the VUV lamp and positioned in the center of the ionization chamber, and thus the volume of the ionization chamber is substantially reduced to 10 μl , decreasing the response time and improving the response sensitivity.

In order to shield from the photoelectric effects produced by the VUV light radiation on the electrodes, an annular ion collection electrode that is perpendicular to the direction of energy radiated by the VUV lamp was positioned close to the inner wall of the ionization chamber, and an annular accelerating electrode was embedded in the groove of the nozzle. In addition, the ionization chamber was surrounded by an electromagnetic shield, which could prevent

the external electromagnetic noises including the electromagnetic radiation of the VUV lamp from penetrating the ionization chamber. Therefore, these designs enable the PID to demonstrate a very low background noise and a small baseline drift.

In order to reduce the recombination of ions and electrons before collection, the collection electrode was closely positioned in the lamp window, in which the ions and electrons can be immediately captured by the collection electrode after gas molecule ionization. Moreover, a high voltage (which ranged from 100 V to 300 V) was applied between the collection and accelerating electrodes to capture all the ionized species resulting from exposure to VUV radiation. In the end, the output signal of the PID was amplified by an electrometer and monitored by a personal computer.

6.2. Background noise level

In this paper, a few key steps (e.g., shielding the electrodes from the VUV light radiation, surrounding the ionization chamber with an electromagnetic shield) have been used to reduce the background noise level, producing a one- to two-order decrease in the noise magnitude.

After a series of experiments, the results recorded a background current of 2×10^{-14} A; the output of signal acquisition system was 7.98 pA when the VUV lamp was off (see Figure 12 (a)). However, the output was increased to an upper limit of 8.00 pA when the VUV lamp was turned on (see Figure 12 (b)), which was still the lowest value reported in publications [22]. Moreover, the background noise of the PID was less than 1×10^{-14} A, lower than its commercial PID counterparts [23]. In addition, the baseline drift was extremely small, which was negligible in the test period.

Figure 12. The background noise and current of the PID when the VUV lamp was off (a) and on (b)

6.3. The response time of the PID

In this setup, the ionization chamber of the proposed mini PID with a cell volume of 10 μ l was much smaller than that of its commercial PID counterparts (from 40 μ l to 200 μ l [22,23]). The relatively large volumes of commercial PIDs lead to a large dead volume which can distort GC

elution profiles and compromise the device performance. With reduction of the ionization chamber, the response time can be dramatically reduced.

Figure 13. The response time of the proposed PID

In order to evaluate the response time of the mini PID, a capillary (20 cm×0.32 mm) instead of the GC column was used to directly connect the sample injector to the ionization chamber of the mini PID. The flow rate was set to 30 sccm so that the elapsed time in the capillary can be ignored. Figure 13 indicates the response time of the proposed mini PID. The response time was less than 30 ms from the sample entering into the capillary to 90 % response of the sample. To the best of the authors' knowledge, the response time reported in this study was the shortest among all the reported commercial counterparts.

7. Detection of harmful gases

Because of a large number of emissions of industrial waste gas and a substantial increase in automobile, air pollution is more and more serious. At present, PM 2.5, heavy haze, and choking smog have seriously affected people's health. In order to solve the problem of air pollution, real-time monitoring of air quality is very urgent and important. Volatile organic compounds (VOCs) are the premise of pollutants for forming the PM2.5 or heavy haze and choking smog, so monitoring VOCs is a very important measure. Therefore, we use the developed portable PID system for detecting these target pollutants released into the atmosphere, and these analytes mainly include benzene, toluene, styrene, phenol, etc.

Due to the lack of judgment on the qualitative components, the standard chromatogram of these main pollutants in the environmental air must be formulated. In this work, the standard sample with concentrations close to these components in environment was used to develop the standard curve, which can provide a scientific basis for environmental analysis (such as the city mobile monitoring air quality).

The experiments were performed under isothermal conditions at 40 °C with a carrier gas linear velocity of 18 cm/s. The pure He was used as carrier gas and the standard sample was composed of four components, namely, benzene, toluene, styrene, and phenol, with the concentrations of 5 ppm, 10 ppm, 8 ppm, and 10 ppm, respectively. The sample was diluted ten times and injected by a micro-pump and a six-port external sample injector, and carrier gas velocity was controlled by a gas flow controller. Figure 14 shows the chromatogram of the standard sample.

Figure 14. The chromatogram of standard sample

The result shows that the proposed system demonstrated a good separation and detection of these volatile organic compounds. The smallest response amplitude of the chromatographic peak (benzene) is also over 10 pA. Moreover, the minimum resolution of the closest two components is over 1.4. These experimental data are sufficient to show that the developed portable PID system can be widely applied to the trace detection of environment analysis.

8. Conclusion

The work here demonstrates that it is possible to fabricate a mini GC system integrated with a micro dryer and purifier, a micro pre-concentrator, a micro GC column, and a mini PID. The micro dryer and purifier can remove vapor and particulates from environmental samples, which makes the mini GC system suitable for field use. The micro pre-concentrator can concentrate the trace gas, which enables the mini GC system to detect environmental samples

and also improves detection sensitivity of mini-instruments. In addition, the micro GC column can reduce the volume of the system and overcome low-resolution and poor anti-interference ability of other instruments. Based on the above experimental results, the mini GC system can effectively separate and detect the air pollutants. Therefore, the developed portable PID system can be widely applied to the trace detection of environment analysis. However, the standard sample instead of the actual sample was used in performing the experiments. In the following work, we will carry out the city mobile monitoring air quality based on this experimental basis, and the results will be reported in the next works.

Acknowledgements

The authors greatly acknowledge the financial support from the National Science Foundation of China under grant numbers 61176112 and 60976088. The authors greatly acknowledge the financial support from the Beijing Science and Technology Plan project under grant number Z141100003414003.

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