

Design, Modeling, Microfabrication and Characterization of the Micro Gas Chromatography Columns

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1. Introduction

Gas chromatography (GC) systems can separate different components of gaseous mixtures, and are important analytical tools for a variety of disciplines, including environmental analysis, methane gas probes, and homeland security and pollution monitoring. However, most modern GC devices suffered the problems of low detection speed, sensitivity, and poor stability. In addition, these conventional GC devices were bulky and fragile, which ruled out the possibility of the in-field use. In some cases, gas samples were collected in the field and analyzed in the laboratory using a conventional GC, which was inconvenient and inefficient.

Combined with micro-detector (such as micro thermal conductivity detector, micro photoionization detector, etc.), Micro GC columns can be developed into a miniaturized chromatographic system because of its very small size. This integrated GC system, with a small size, light weight, rapid analysis, high sensitivity, easy to use, etc., can be widely applied to environmental pollution, home safety, pesticide residues, food safety, pre-diagnosis of cancer and other areas for achieving on-site and on-line rapid testing.

In this chapter, several micro gas chromatography columns were designed for building micro μ GC systems, and this chapter will contain the following sections: Structural design, modeling analysis, microfabrication and characterization of the micro gas chromatography columns.

2. Structural consideration

Structure of the μ GC columns can be designed in accordance with the requirements of the designer. Several various structural designs are appeared in research papers, but their differences in-depth analysis of these designs are almost absence. In fact, structure is an important factor for affecting the separation performance of the μ GC columns, because the shape of the column, especially the mutations sections (such as the corner of the channel), will change the airflow velocity, pressure distribution, the thickness of the

stationary phase film and other important factors, moreover, these factors are key factors for affecting the separation performance of μ GC columns.

In this work, two frequently-used configurations (the spiral channel and the serpentine channel, as shown in Fig.1) of columns were designed for GC analysis, the shape of cross-section is rectangular, and their sizes are consistent. Then, the effect of airflow rate and pressure distribution in the channel was simulated using ANSYS analysis. Especially, the effect of these factors in the corners was the focus.

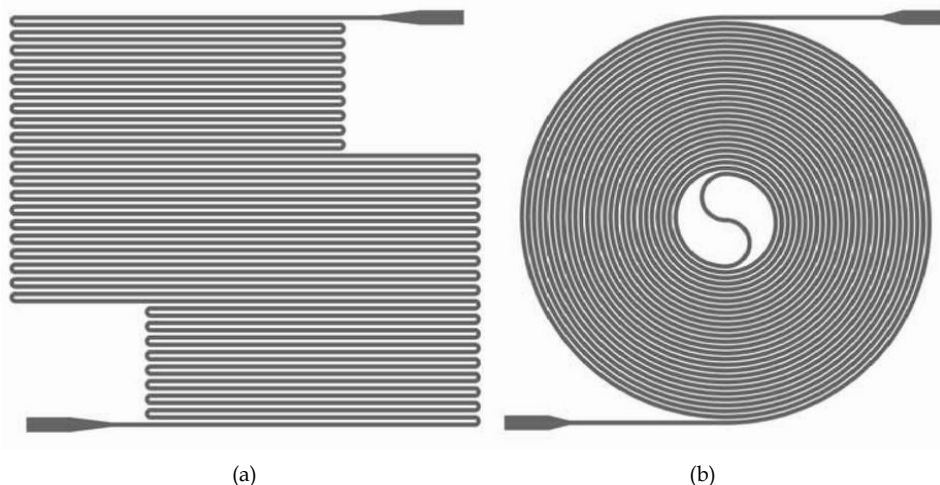


Fig. 1. The structure design, (a) the serpentine channel, (b) the spiral channel, for μ GC column.

3. Theoretical consideration and simulation

3.1 Theoretical consideration

If the volume and the concentration of a sample are small enough (about μmol) and in the linear range of adsorption isotherm, the elution curve equation can be determined by equation (1) based on the plate theory.

$$C = \frac{\sqrt{n}}{\sqrt{2\pi}} \cdot \frac{m}{V_r} \cdot \exp\left[-\frac{n}{2}\left(1 - \frac{V}{V_r}\right)^2\right] \quad (1)$$

Where C is the sample concentration at any point of the elution curve, m is the weight of the solute, V is the retention volume at any point of the elution curve, V_r is the retention volume of the solute, n is the number of theoretical plates. When $V = V_r$, C reaches its max value:

$$C_{\max} = \frac{\sqrt{n} \cdot m}{\sqrt{2\pi} \cdot V_r} \quad (2)$$

Derived from the elution curve equation, the number of theoretical plates n can be defined by the following equation:

$$n = 5.54 \left(\frac{t_r}{w_{1/2}} \right)^2 \tag{3}$$

Where t_r is the retention time, and $w_{1/2}$ is the width of the peak at half height.

And the theoretical plate height (H) can be determined by:

$$H = \frac{L}{n} \tag{4}$$

Where L is the length of the column. According to the equation (3) and (4), as the chromatography peak's bottom width (w) decreases, the number of theoretical plates (n) increases and the theoretical plate height (H) decreases accordingly, which results in higher column efficiency. Hence, n and H are the index of column efficiency.

Due to some effect factors for H can't be shown in equation (4), the theoretical plate height (H) can also be given by the following formula:

$$H = \frac{2D_g}{u} f_1 f_2 + \frac{(1+9k+25.5k^2)}{105(k+1)^2} \frac{w^2 \bar{u}}{D_g} \frac{f_1}{f_2} + \frac{2}{3} \frac{k}{(k+1)^2} \frac{(w+h)^2 d_f^2}{D_s h^2} \frac{1}{u} \tag{5}$$

where D_g and D_s are the binary diffusion coefficients in the mobile and stationary phases, respectively, d_f is the thickness of the stationary phase, w and h are the channel width and height, respectively, k is the retention factor, and f_1 and f_2 are the Giddings–Golay and Martin–James gas compression coefficients, respectively. The average linear airflow velocity is given by

$$\bar{u} = \frac{w^2 p_0 (P^2 - 1)}{24 \eta L} f_2 \tag{6}$$

Where P_0 is the outlet pressure, P is the ratio of the inlet to outlet pressure, L is the column length, and η is the carrier gas viscosity.

From the equation (5) and (6), the airflow rate and pressure are key factors for the separation performance of the μ GC columns.

Resolution is called overall separation efficiency, which is defined as the difference of retention time between two adjacent chromatography peaks divided by the half of the sum of these two peak's bottom width:

$$R = \frac{t_{r_2} - t_{r_1}}{\frac{1}{2}(w_1 + w_2)} = \frac{2(t_{r_2} - t_{r_1})}{w_1 + w_2} \tag{7}$$

The definition of resolution (R) in equ (7) does not reflect all the factors which influence resolution, because resolution is actually determined by column efficiency (n), selectivity factor (α) and capacity factor (k), hence the resolution also can be described by equation (8):

$$R = \frac{\sqrt{n}}{4} \left(\frac{\alpha - 1}{\alpha} \right) \left(\frac{k}{1 + k} \right) \quad (8)$$

After the stationary phase is chosen, the selectivity factor could be correspondingly fixed, which means that the resolution is only affected by n . For a column with a certain theoretical plate height, square of the resolution is proportional to the length of column:

$$\left(\frac{R_1}{R_2} \right)^2 = \frac{n_1}{n_2} = \frac{L_1}{L_2} \quad (9)$$

Therefore the major approach to improve resolution is to increase the column length.

3.2 Simulation

Base on chromatography theory, the airflow rate and pressure are key factors for the separation performance of the μ GC columns. An obviously different variation of the airflow velocity, pressure distribution and other factors will be appeared in these μ GC columns due to their different structural, and these variations will affect the separation performance of μ GC columns. So a detailed comparative analysis for the two frequently-used configurations of columns is made in this paper, which provides a theoretical basis for designing ideal μ GC columns.

In this chapter, the effects on the airflow rate and pressure were simulated using ANSYS analysis. Because the major effect of airflow rate is come from the mutation section (such as the corner of GC-channel) of these channels, the corner of the chromatographic channel are selected for the simulation. In the simulation, the first step is modelling. The parameters of the modelling are consistent with the size of the actual channel (The cross-section is rectangular, the width of the channel is 150 μ m, and the depth of the channel is 100 μ m).

After modelling, boundary conditions were set as follows: the airflow rate in the gas inlet was set to 18 cm/s, the pressure of the gas outlet was set to 0, the displacement of the other part was set to 0).

Fig.2 (a) and (b) show the solutions of the simulation, the airflow rate in the corner of serpentine channel is 15.73 cm/s, the difference of airflow rate between in the corners of serpentine channel and in the gas inlet reaches 3.13 cm/s. However, the airflow rate in the mutation section of the spiral channel is 16.93 cm/s, the difference of airflow rate between in the corners of spiral channel and in the gas inlet is only 1.33 cm/s. Moreover, the changes in distribution of airflow rate in the corners of the serpentine channel are obvious and relatively large. But the distribution of airflow rate in the corners of the spiral channel is relatively uniform. Fig.3 (a) and (b) show the solutions of the pressure distribution in the serpentine channel and spiral channel, the simulation results show that there exists a pressure gradient in these two kinds of channel, but the change rate of pressure in serpentine channel is obviously much larger than that of the spiral channel, the maximum

pressure value in spiral channel is only 32.80 Pa, however, the maximum pressure value in the serpentine channel is over 150.0 Pa, the value is close to 5 times compared to the former. Seen from the above analysis, the impact on airflow rate and pressure in serpentine channel is very significant, and these effects will lead to deterioration of separation performance. Because the difference of the airflow rate in the channel would change the thickness of the stationary phase film. Consequently, the variation of the thickness of the stationary phase film in the channel would lead bad tailing peaks.

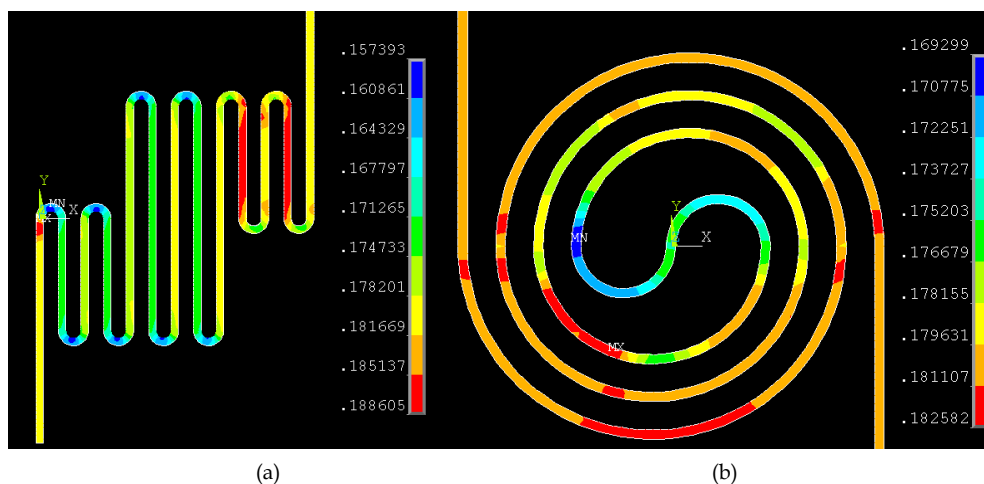


Fig. 2. The effect of the airflow rate in the corners of the channel (a)the serpentine channel,(b) the spiral channel, were simulated using ANSYS analysis.

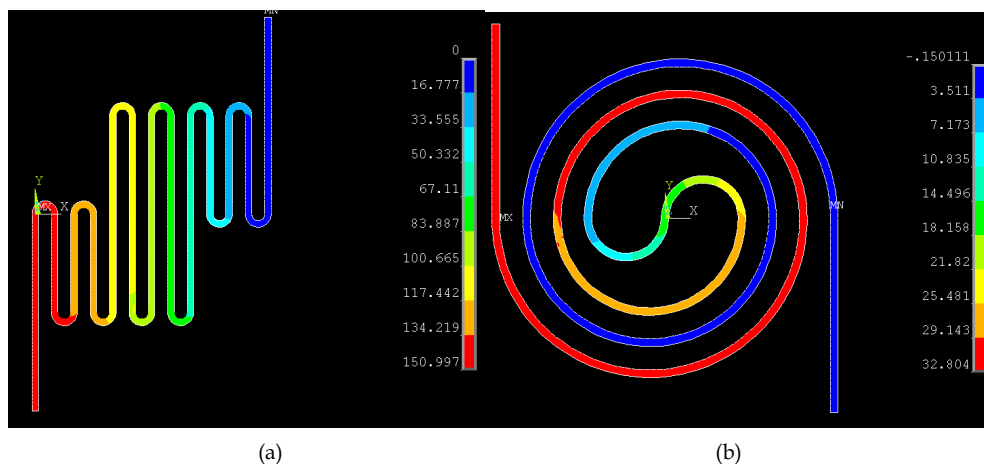


Fig. 3. The pressure distribution in the GC channel (a)the serpentine channel,(b) the spiral channel, were simulated using ANSYS analysis.

4. Microfabrication

4.1 Column fabrication

Fabrication of the μ GC column includes aluminium deposition, photolithography and deep reactive-ion etching (DRIE) (the fabrication process is illustrated in Fig.4). Firstly, a 2- μ m-thick electron-beam evaporation aluminium film was deposited on a p-type <100> silicon wafer which served as the etch mask in following steps. Secondly, a thickness of approximately 2 μ m AZ1500 photoresist was coated on the wafer and patterned as an etch mask for aluminium. Subsequently, aluminium without the protection of photoresist was etched away by an etchant (H_3PO_4) and the silicon surface was exposed. Then, a DRIE process, instead of the anisotropically KOH chemical etching process, was utilized to form the rectangular micro channels. Finally, all aluminium masks were removed and the silicon wafer and pyrex7740 glass were held together and heated to approximately 400°C. A 1000 V potential was then applied between the glass and the silicon, and the resulting electrostatic force pull the wafers into intimate contact.

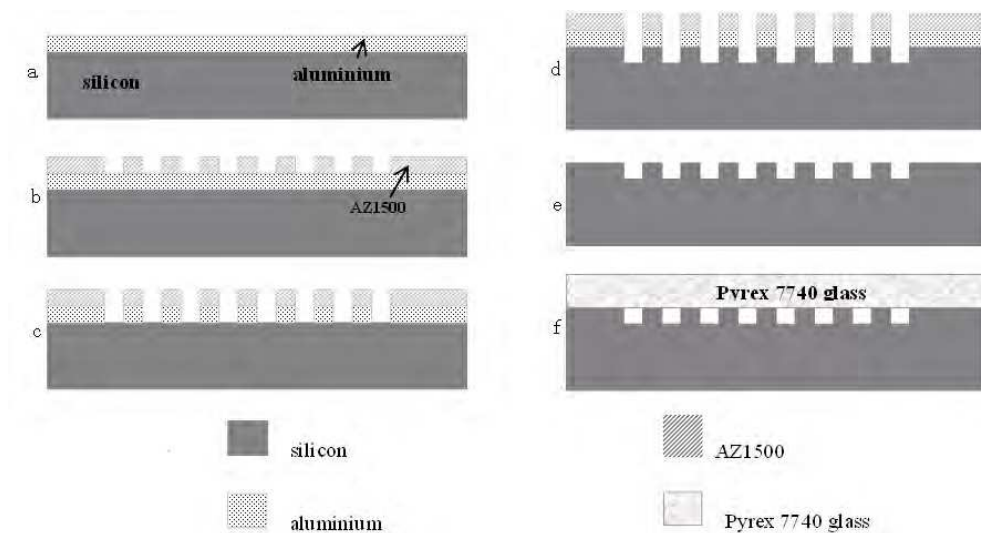


Fig. 4. The GC column process flow.

4.2 The stationary phase coating

The uniformity, stability and thickness of the stationary phase film are important factors that can affect the separation efficiency. In this section, the selection principle of stationary phase, coating methods and coating procedure are introduced.

There is no regularity to follow for choosing the stationary phase. Generally, "Like dissolves like" principle has been identified as the basic theory for selecting the stationary phase. In the application, the selection of stationary phase should be determined according to the actual situation.

- i. Separation of non-polar compounds: Generally, non-polar stationary phase is used. Each component flows over the GC column according to the order of boiling point. The component with a low boiling point is the first one out of the GC column, and followed by the component with a high boiling point.
- ii. Separation of polar compounds: Generally, polar stationary phase is used. The component with a small polar is the first one out of the GC column, and followed by the component with a relatively large polar.
- iii. Separating non-polar and polar compounds: Generally, polar stationary phase is used. The non-polar component is the first one out of the GC column, and followed by the polar component.
- iv. Separation of complex and difficult compounds: Two or more mixed stationary phase can be used.

Stationary phase coating methods are generally including static and dynamic coating. Static coating procedure is defined as: The stationary phase is filled with the GC column, one end is sealed, and the other end is connected a vacuum pump, the solvent is slowly evaporated under the pressure of vacuum pump, until all of the solvent is completely evaporated, and a stationary phase film with a thickness of 0.1-0.2 microns is left on the channel.

Dynamic coating procedure is defined as: A stationary phase solvent is injected into the GC column, and the stationary phase solvent flows through the GC column under pressure, the thickness of Stationary phase can be controlled by changing the flow rate and the concentration of the stationary phase solvent. The stationary phase solvent is pushed out from the other side of the GC column, and the nitrogen gas was delivered through the column for several hours to completely evaporate the solvent, and a stationary phase film with a thickness of 0.1-0.2 microns is left on the channel.

In this work, separation of benzene and homologue of benzene was taken as for example. In order to completely separate the mixture, OV-1 or OV-101 was the optimal stationary phase. So OV-1 was selected to coat the micro GC column using a dynamic coating procedure. The process was shown as following: Firstly, about 5 μ l OV-1 was dissolved in 2.0 ml mixtures of n-pentane and dichloromethane with a volume ratio of 1:1. The mixture was agitated for 30 minutes to ensure full dissolution. Secondly, the inlet of the column was connected with a capillary, and the outlet was connected with a laboratory made micro-pump, which was used to inject the stationary phase solution into the GC column. After the GC column was full of the solution, the micro-pump was turned off for 30 minutes to make sure that it was long enough for the stationary phase to attach to the channel wall. Then, nitrogen gas was delivered through the column for a few hours to completely evaporate the n-Pentane and dichloromethane. Subsequently, the column was put into an oven under a nitrogen atmosphere in which the temperature of the oven was firstly increased gradually by 5 $^{\circ}$ C /min until 100 $^{\circ}$ C and then the temperature of the oven was kept at 100 $^{\circ}$ C for 4 hours. Fig. 5 shows an SEM view of the stationary phase film coated on the column wall, in which the film is uniform and adheres well to the channel. The composition analysis of the film (the selected area of the film on the channel wall can be seen from Fig. 6(a)), by X- Ray Photoelectron Spectroscopy, is described in the Fig. 6(b) which shows that the composition agrees with the composition of OV-1.

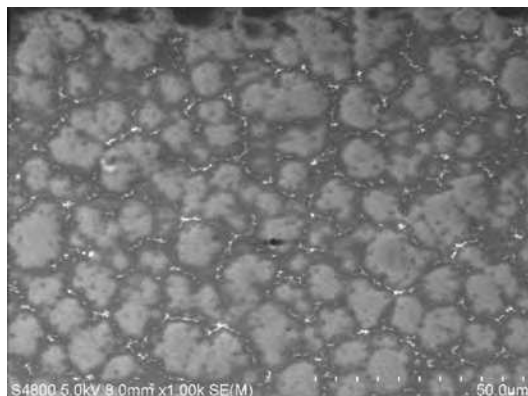


Fig. 5. The SEM view of the stationary phase film.

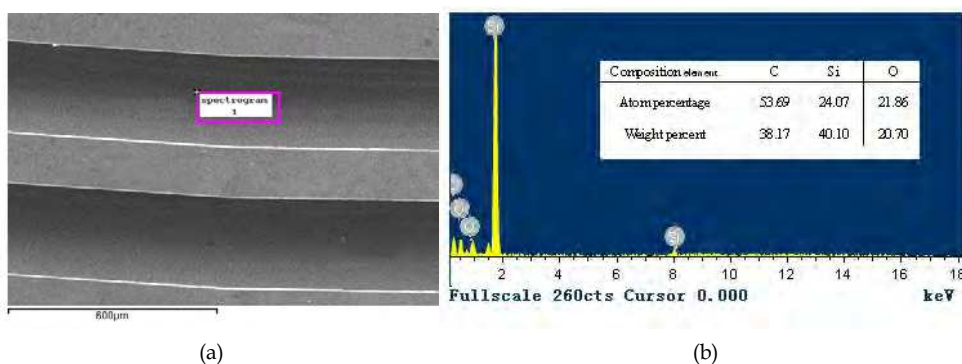


Fig. 6. The composition analysis of the film which operated by X- Ray Photoelectron Spectroscopy, (a) the selected area of the film on the channel wall, (b) the percent of the composition element in the film.

4.3 Interface technology

The inlet/outlet interface technology of MEMS-based GC columns is a key factor. If not properly tackled, some problems, such as instability of the interface, air leakage from the interface and the airflow jamming would happen, which usually led to the failure of the GC columns. In order to solve these problems, a fixed base (the base dimensions is 8.0 mm long, 4.0 mm wide and 5.0 mm high, See Fig.7) was used to connect the tubes with the inlet /outlet of the column. The major steps are briefly covered: Firstly, a film of heat-resistant adhesive was coated on the bottom surface of the base, and then the base was aligned with the inlet or the outlet carefully and bonded with the column; After 30 minutes, more heat-resistant adhesive was coated around the base with a steel tube (Tubing, steel, 1/32"×0.25mm ID) into the base, and then some heat-resistant adhesives were coated around the joint points. Subsequently, a burst pressure test with 0.4 MPa pressure was applied on the column and the interface remained undamaged. Fig. 8 shows a photograph of the completed GC columns including the connected base.

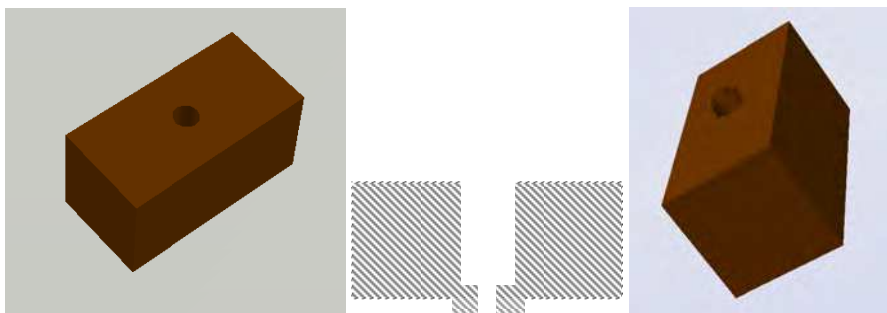


Fig. 7. The picture of the fixed base.

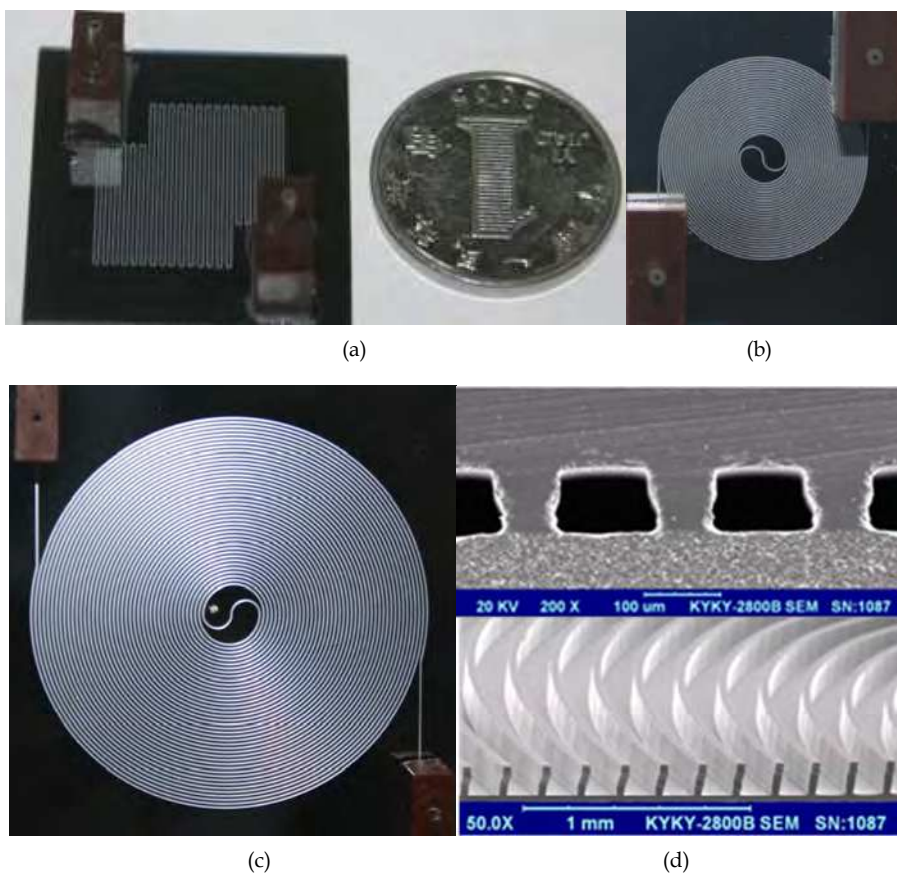


Fig. 8. The photograph of the completed GC columns, (a) 0.75m serpentine channel column, (b) 0.75m spiral channel column, and the chips dimension are all 2.2cm×2.2cm, (c) 3 m spiral channel column and the chip dimension is 3.5cm × 3.5, (d) SEM micrograph of the section and the channel.

5. Results and discussions

5.1 Experimental setup and test condition

GC performances were measured through the flame ionization detector (FID) method on an Agilent 7890A GC system. In the test, the carrier gas was helium and the original operating temperature of columns was 40°C, in order to reduce the total analysis time of the component mix, temperature programming, which the temperature is increased from 40°C to 80°C at a rate of 5°C / min, was used in the process of separating the sample. These samples were dissolved in 1.0 ml CS₂, and the volume of each component sample, which was injected at time zero, was 1 µl. The top-air method was used with a split ratio of 1:10.

5.2 Experimental results

In this section, the effect of the structure the flow rate and the length for separation performance were analyzed, and then the experiments for separating complex mixtures were studied.

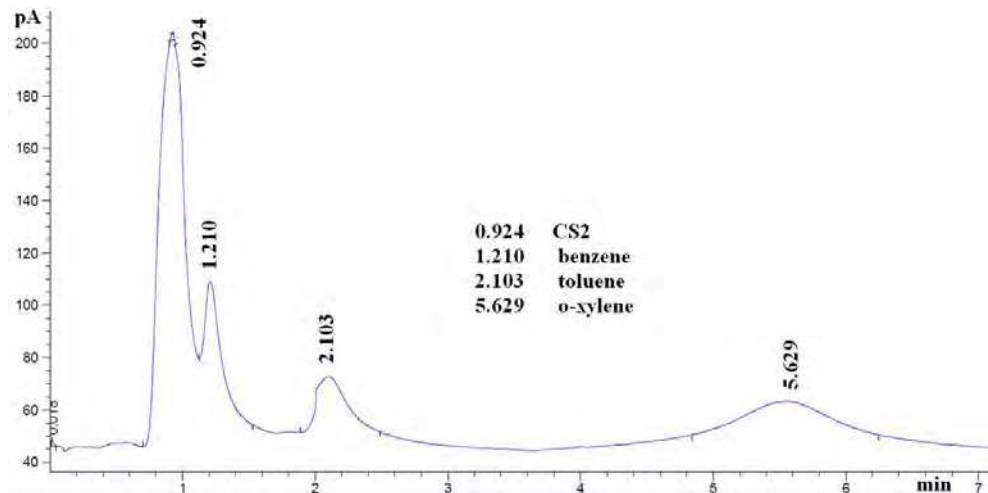
5.2.1 Effect of the structure for separation performance

These columns were operated at the velocity of 18 cm/s, the sample is a 3-component mixture including benzene, toluene, o-xylene, and the chromatogram graphs were shown in Fig.9 (a) and (b). In Fig. 9 (a), the serpentine channel GC columns separated the gaseous mixture with a resolution of 8.9 (calculated by equation (7)) between toluene and o-xylene and yielded about 1900 theoretical plates (calculated by equation (3)). However, the chromatographic peak emerged bad tailing peaks. The inconsistent of the film thickness were the primary cause for the tailing peaks. However, the spiral channel GC columns yielded about 3900 theoretical plates and separated the gaseous mixture with the resolution of 10.97 (as shown in Fig. 9 (b)). Moreover, the chromatographic peaks were greatly improved. So the structure of GC channel, especially the design of the corner, was a key factor for the separation performance. The spiral channel possessed streamlined shape. As a result, the airflow couldn't be baffled, and the separation experiment also verified that the spiral channel GC column showed excellent overall separation performance and separation efficiency for the gas mixture. Thus, the spiral channel column is superior to the serpentine channel column for GC analysis. Therefore, on this basis, we designed a 3 m spiral column for GC application, then, the effect of the flow rate and the length for separation performance was analyzed and its separation performance for complex mixture was studied.

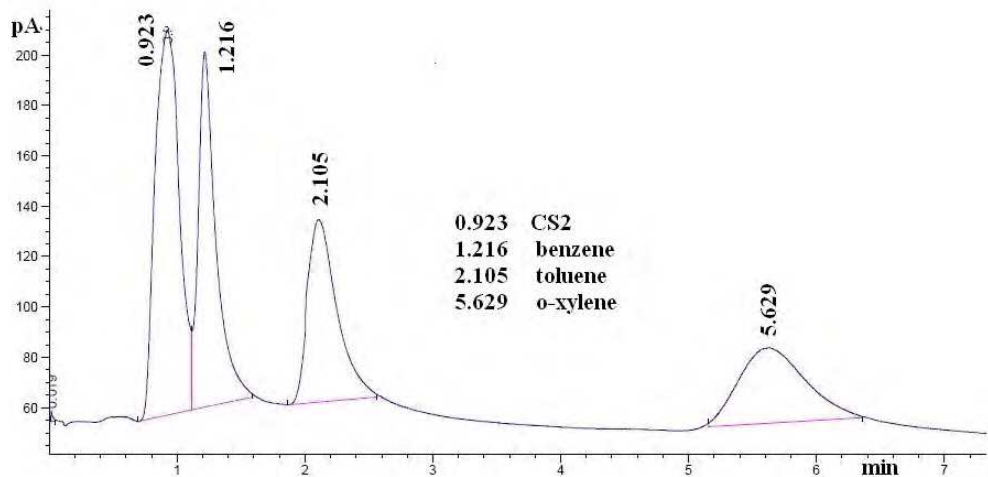
5.2.2 Effect of the flow rates for separation performance

Because the flow rate was a key factor for separation performance, different flow rates were used in the process of separating the sample (the sample is a 3-component mixture including benzene, toluene, o-xylene). Firstly, the 3 m spiral column was operated at the velocity of 42 cm/s, and the chromatogram graph was shown in Fig.10 (a). From experimental data, the number of plates can be calculated as given in equation (3). The 3 m spiral column yielded 3410 plates. The resolution, R, can be calculated by equation (7). The resolution between benzene and toluene was 4.05, and the resolution between toluene and o-xylene was 9.27. While, if the flow rate was appropriately reduced and the column was

operated at the velocity of 18 cm/s, the column yielded approximately 6160 plates (the experimental data was shown in Fig.10 (b)), moreover, the resolution between benzene and toluene was improved from 4.05 to 8.02, and the resolution between toluene and o-xylene was improved from 9.27 to 14.3. The separation efficiency of the gas mixture was strongly improved.

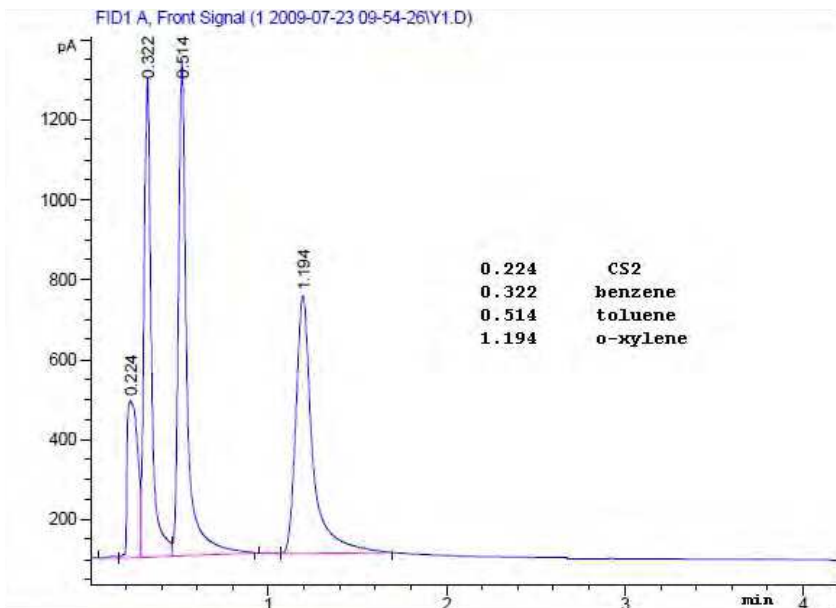


(a)

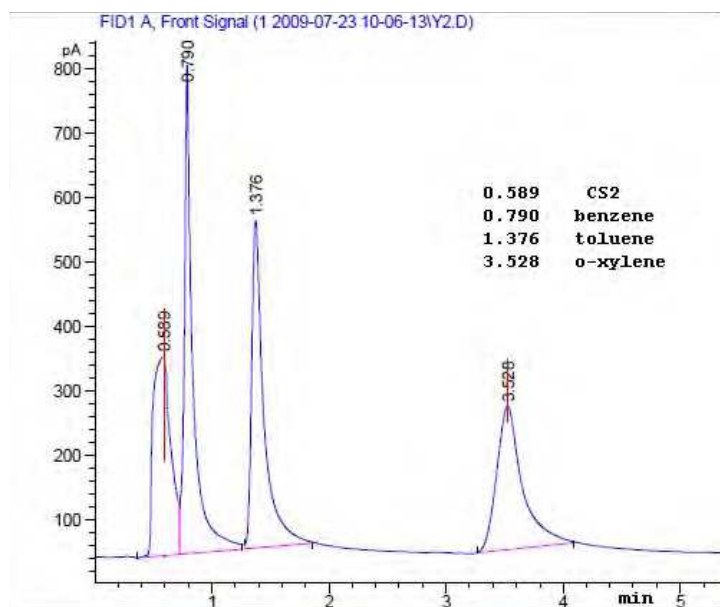


(b)

Fig. 9. Chromatogram of a gas mixture achieved using the (a) 0.75m serpentine channel column, (b) 0.75m spiral channel column.



(a)



(b)

Fig. 10. Chromatogram of a gas mixture containing benzene, toluene and o-xylene achieved using the 3m column coated with OV-1, (a) operated at the flow rate of 42 cm/s, (b) operated at the optimal flow rate of 18 cm/s.

5.2.3 The effect of the column length for separation performance

Based on the formula (9), the major approach to improve resolution is to increase the column length.

So in this paper, the separation performance of GC columns with different length (such as the length of the column is 0.5 m, 1 m and 3 m, but the section width and depth of the column are the same) were compared with, and the separation experiments were all operated at the velocity of 18 cm/s and used the same sample (the sample including 3-component mixture: benzene, toluene, o-xylene). The columns yielded approximately 2400, 3370 and 6160 theoretical plates (the actual test values), respectively.

The theoretical predictions values could be also roughly computed 2500, 5000 and 15000 plates (derived from the equation 1 and equation 4), respectively. The actual test values were about 41% of theoretical prediction values. When the column length increased to 3 meters (Assuming the maximum retention factors is less than 10, the diffusion coefficients in the gas and liquid phases are about 0.1~0.2 cm²/m, and the thickness of the stationary phase film is 0.2 μm), The comparison curve of the number of theoretical plates between the theoretical prediction values and the actual values was shown in Fig.11.

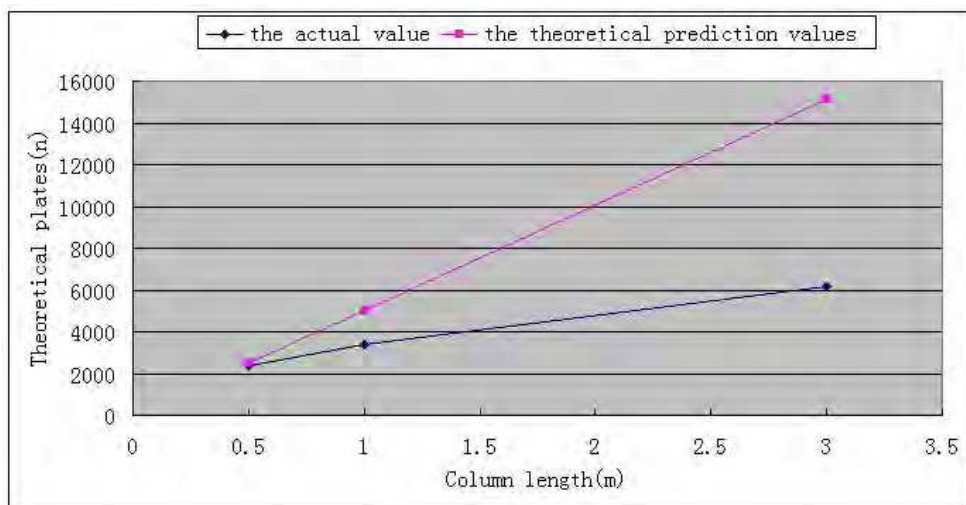


Fig. 11. The comparison curve of the number of theoretical plates between the theoretical prediction values and the actual values.

The discrepancy with the predicted number of the theoretical plates can be attributed to stationary phase film nonuniformity with the increase in column length. Consequently, the theoretical plates could be significantly increased by lengthening the GC column. Moreover, the separation performance of GC column can be greatly improved. The GC column can acquire a good overall separation performance when the length of the GC column is increased to 3 m.

5.2.4 Separation of the complex mixtures using 3 m spiral column

A separation experiment was performed to separate the sample mixture of benzene, toluene, ethylbenzene, p-xylene and o-xylene using the microfabrication GC column. The column was operated at the velocity of 18 cm/s, and the chromatogram graph was shown in Fig.12. From experimental data, the spiral 3 m column yielded 7100 theoretical plates, and the analysis time was less than 200 sec. The GC column acquired a good overall separation performance.

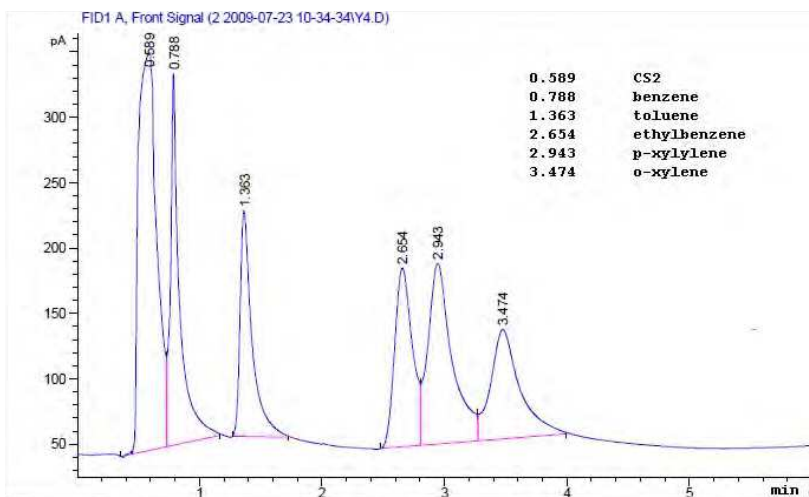


Fig. 12. Chromatogram of a gas mixture containing benzene, toluene, ethylbenzene, p-xylene and o-xylene, achieved using the 3m column coated with OV-1 which operated at the flow rate of 18 cm/s.

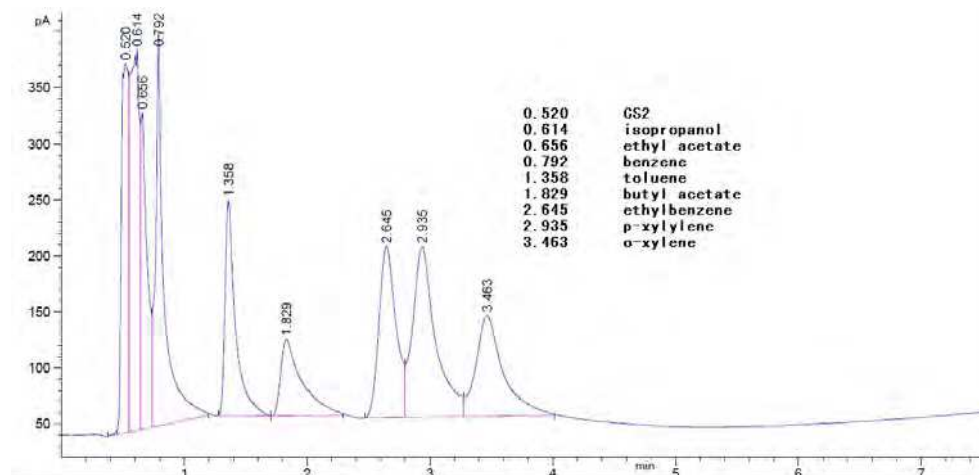


Fig. 13. Chromatogram of a gas mixture containing benzene, toluene, ethylbenzene, p-xylene, o-xylene, butyl acetate, isopropanol and ethyl acetate achieved using the 3m column coated with OV-1 which operated at the flow rate of 18 cm/s.

Then, three other components were mixture into the sample, of which two kinds of components were polar components (isopropanol and ethyl acetate), and another sample was the butyl acetate. The separation experiment was carried out under the same conditions, the chromatograms was shown in the Fig.13, these polar components and solvents came out almost simultaneously, which resulted in these two polar components couldn't be separated by non-polar stationary phase, but the acid butyl ester was perfectly separated. The result indicated the non-polar column with high selectivity and specificity.

6. Conclusion

In summary, this chapter has presented the simulation, fabrication and experimental results for the microfabricated GC columns. Prototypes of devices have been fabricated successfully and the stationary phase is tackled properly. From the result of the simulation, the changes in distribution of airflow rate in the corners of the serpentine channel are obvious and relatively large, and the change rate of pressure in serpentine channel is obviously larger than that of the spiral channel, the value is close to 5 times compared to the former. Seen from the experimental data, the impact on airflow rate and pressure in serpentine channel is very significant, and this effect leads to deterioration of separation performance. Moreover, the difference of the airflow rate in the channel changes the thickness of the stationary phase film when the stationary phase is deposited. Consequently, the variation of the thickness of the stationary phase film in the channel leads bad tailing peaks. So, the spiral channel column is superior to the serpentine channel column for GC analysis. A series of spiral GC columns are designed according to the above analysis. The 3 m yields approximately 7100 plates and perfectly separates the complex mixture in less than 200 sec. Combined with micro-detector and micro sampler, Micro GC columns can be developed a miniaturized chromatographic system and serve as a platform technology for gas mixture separations.

Currently, the developed micro-columns can not achieve its own heating, but the design and fabrication process of the integrated micro-heater GC columns have been completed, its performance will be reported in subsequent papers. Micro- thermal conductivity detector, micro-optical ionization detector and micro-meter will be developed in our further work. A new generation of micro-chromatographic system will be developed by integrating micro GC column with the micro detectors. The micro GC system will be applied to environmental pollution, home safety, pesticide residues, food safety, pre-diagnosis of cancer and other areas for achieving on-site rapid testing.

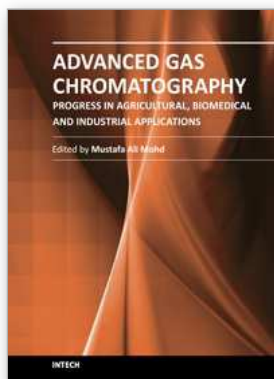
7. Acknowledgment

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Progress in agricultural, biomedical and industrial applications' is a compilation of recent advances and developments in gas chromatography and its applications. The chapters cover various aspects of applications ranging from basic biological, biomedical applications to industrial applications. Book chapters analyze new developments in chromatographic columns, microextraction techniques, derivatisation techniques and pyrolysis techniques. The book also includes several aspects of basic chromatography techniques and is suitable for both young and advanced chromatographers. It includes some new developments in chromatography such as multidimensional chromatography, inverse chromatography and some discussions on two-dimensional chromatography. The topics covered include analysis of volatiles, toxicants, indoor air, petroleum hydrocarbons, organometallic compounds and natural products. The chapters were written by experts from various fields and clearly assisted by simple diagrams and tables. This book is highly recommended for chemists as well as non-chemists working in gas chromatography.

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