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Real-time Simultaneous Separation and Detection of Chemicals using Integrated Micro Column and Surface Plasmon Resonance Imaging Micro-GC

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Abstract

An integrated and miniaturized Micro-Gas Chromatography with real-time imaging capability for simultaneous chemical separation and detection was developed. Surface Plasmon Resonance imaging (SPRi) was used as a sensitive and real-time imaging based detector for various gaseous chemical mixtures and good gas chromatographs were obtained. The system integrated a home-made miniaturized molecular sieve packed spiral micro-channel column with the SPRi imaging chip and real-time chemical separation and detection were demonstrated using alkanes. The chemical separation processes were simulated using COMSOL and matched well with experimental results. The system enabled the study of chemical separation processes in real-time by miniaturizing and integrating the Micro-GC separation and detection units. This approach can be expanded to multidimensional GC development.

Index Terms

Gas Chromatography (GC); micro-GC; chemical separation; GC column; GC detector; surface plasmon resonance imaging (SPRi); minituration and integration

I. Introduction

Gas Chromatography (GC) is one of the most important and widely used tools in analytical chemistry. Tremendous efforts and progresses have been made in improving the separation column, the detector, and in the development of portable Micro-GC [1].

Despite the fact that benchtop GC instruments are reliable and accurate, analytical samples are often at risk of sample contaminations, degradation, decomposition, and loss due to storage and transportation from the collection to the analysis site.

Micro-GC is portable and energy efficient, which allows on-site, real-time biological, forensic, and environmental analyses [2]–[4].

Several groups have been working on developing Micro-GC systems focusing on the development of miniaturized column, sensitive and reliable detector, as well as system integration of various components [5]–[10]. For example, Qin et al. and Cossolin et al. demonstrated Micro-GC systems using cleanroom fabricated capillary columns [5]–[6], Akbar, et al developed a Micro-GC using micro-fabricated semi-packed capillary column and a Micro-Photon Ionization Detector (Micro-PID) as detector [7], Contreras et al. used commercial capillary column and toroidal ion trap mass spectrometer as detector [8], Wright et al. used nanoparticle-coated chemiresistor array as Micro-GC detector [9], Chen et al. explored the possibility of using micro multi-column chip and stop-flow system to build multidimensional GC [10]. It can be seen that Micro-GC typically uses either commercial capillary columns (which are expensive) or cleanroom fabricated micro columns (involves complicated column design and fabrication processes) as separation unit. Furthermore, due to the small diameter (μm) and long column length (m), the column back-pressure is too high for power constrained pump to provide the needed flow rate. More importantly, to our knowledge, none of the Micro-GC system developed to-date let us observe chemical separation in real-time. The separation process is like a black box, with the detector measuring only what comes out of the column but not the separation process inside the column.

In order to overcome the disadvantages mentioned above, a molecular sieve packed spiral column was developed and integrated directly onto the detector imaging platform, with which real-time chemical separation was observed. The column occupies a 7mm by 7mm area that maintains low back pressure due to its millimeters channel size and 3cm channel length, making it possible to use a low power pump to deliver gas samples.

In order to observe the chemical separation process while simultaneously detect chemical signal peaks in a real-time fashion, Surface Plasmon Resonance Imaging (SPRi) was used as the detection and imaging platform. Surface Plasmon Resonance, a refractive index based sensing mechanism, has been studied and widely applied in low concentration bio-sensing and gas sensing applications [11–13]. Surface plasmon microscopy has been used to directly observe the sensing and detecting processes [14–16]. Several gas and bio-sensing applications have been explored by using portable and miniaturized SPR instruments [17–18]. Moreover, compact design and multi-analytes analysis capability of portable SPR devices have been demonstrated and proven successful. SPRi based commercialization efforts have been made as well [19–21].

In this work, we integrated miniaturized molecular sieve packed column directly on top of the real-time SPRi chip. The first advantage of this approach is that the separation and detection units are integrated, hence, the system is miniaturized and more importantly, it can

directly image the real-time chemical separation process through simultaneously detecting various chemical signal peaks. The signal peaks are caused by the refractive index change above the imaging chip when channel. This novel approach provides new way of studying the basic chemical separation processes in a real-time imaging based approach, which when combines with Micro-GC separation and detection unit further shrinks the form factor and potentially provides new opportunities for multi-dimensional GC development.

II. Experimental Setup

Firstly, the feasibility of using gas phase SPRi platform as imaging based GC detector was demonstrated. A detection cell was fabricated using Acrylic plastic. A thin channel was etched from the surface of the cell, while the gas inlet and outlet are drilled at the two sides of the channel. The cell was fixed on top of the SPRi gold chip, which in turn is sitting on top of the optical prism, by four screws. Teflon tubing was used to connect a 20m long commercial capillary column to the inlet of the detection cell. A three-way switching solenoid valve was used to switch between lab air and a known component sample airbag. A pump was used to actively sample the gas. The start of injection and injection duration can be controlled by controlling the switching of the valve. A 650nm laser source was used as the light source and a CCD camera was used to capture the SPR images. The whole setup is shown as schematics in Fig. 1.

Binary chemical mixtures were used as sample gas. As the analyte passes through the channel, the image intensity increases due to the change of refractive index above the gold chip. The intensity change over time was extracted from the images and gas chromatographs with good signal-to-noise ratio and resolution were obtained.

Secondly, a PDMS coated home-made capillary column was fabricated. The column is cheaper, shorter and, more compact compared to the commercial column. Commercial column was then switched to the PDMS phase column and a mixture of gaseous alkanes was used. Separation of chemicals was observed using the SPRi detection cell.

Finally, to further shrink the column size and integrate it onto the SPRi gold chip, a molecular sieve packed spiral column with higher sample capacity and lower back pressure was fabricated and integrated onto the SPRi imaging platform. Several alkanes were used and real-time separation process of chemicals was obtained and validated by COMSOL simulation.

A. Commercial Capillary Column

A 20m long commercial column from Quadrex (Cyanopropylphenylmethyl polysiloxane coated) with column inner diameter of 0.25mm and film thickness of 1.0 μ m was used to separate gas mixtures and evaluate the SPRi detector performance. A diaphragm pump from Parker was used to deliver the gas samples into the SPRi detecting cell.

B. PDMS Coated Capillary Column

After the successful demonstration that SPRi can be used as GC detector, a more compact, less expensive column with much lower backpressure was developed. In order to do this, a

serpentine patterned column was fabricated on the surface of an acrylic sheet using a laser cutter. The column has a total length of 3m, channel width of .5mm, and channel depth of 1mm. The fabricated surface was then spin coated with a layer of PDMS solution. The piece was then cured at 65° in an oven overnight. After curing, there remains a gap in the channel due to the shrinkage of the PDMS, forming a micro-channel with a depth of .5mm for gas flow. Another piece of Acrylic was used to seal the column with screws. The thin layer of cured PDMS on the piece acts as a gasket, which helped seal the channel. Tests were performed to guarantee air tightness. After the fabrication of the PDMS capillary column, the commercial column was replaced by the home-made column. Mixture of alkanes was used to test the separation ability of the column. Gas Chromatograph was obtained using the SPRi detector and is shown in the result section.

C. Molecular Sieve (MS) Packed Minuturized Spiral Column

The PDMS column, though much smaller and cheaper than the commercial column, is still too big to be completely integrated onto the limited SPRi imaging area. Hence, a molecular sieve packed column was developed. In order to maximize the utility of area and increase the sample capacity, a laser cutter fabricated spiral column was developed, with a total column length of 3cm, column depth of 1mm, and width of 1mm. The column area forms a circle with a diameter of 7mm, which could be completely fitted inside the SPRi imaging area of 7mm by 7mm. The molecular sieve was purchased from MilliporeSigma. The mesh size is 4Å and the chemical formula is $\text{SiO}_2\text{Na}_2\text{OAl}_2\text{O}_3\text{MgO}$. Molecular sieve beads were grinded into smaller particles with diameters of $\sim 50\mu\text{m}$ which were then used to fill the inside of the channel. A PTFE sheet was used as a gasket to seal the interface between the Acrylic piece and the SPRi gold chip. Fig. 2 (a) shows the picture of the MS packed miniaturized spiral column.

D. Integrated MS Column and SPRi platform

The fabricated MS column was then tightened directly on top of the SPRi gold chip which was on top of the prism holder. Four screw were then used to guarantee the air-tightness of the system. Several alkane samples were used to test the chemical separation. The entire column area was imaged using the camera and the images were further analyzed. The assembly is shown in Fig. 2 (b).

It is important to mention that the whole integrated setup is miniaturized, lab fabricated, easy to use, and inexpensive.

III. Results and Discussion

We have demonstrated SPRi as a Micro-GC detector, fabricated and tested PDMS coated capillary and MS packed columns, integrated the MS packed column onto SPRi gold chip, and tested the separations of various chemicals. The results were corroborated with COMSOL simulation results.

A. Gas Chromatograph obtained using SPRi method

A 2m long commercial capillary column was used for chemical separation and SPRi detector was used to obtain the gas chromatograph. Fig. 3 (a) shows the image of the SPRi detector cell channel area and the area inside the yellow box was used in the analysis. Fig. 3 (b) is the intensity profile of this area using a mixed ethanol and hexane airbag with different injection times, showing two clear separated signal peaks representing hexane and ethanol for both tests with different injection times. The peak signals are higher with the increase in injection time, due to larger injection volume, without having obvious band broadening. Fig. 3 (c) shows the chromatograph using benzene, toluene, ethylbenzene, and xylene (BTEX) mixture. Four distinct signal peaks can be observed representing the four chemicals. Identification and separation of alkanes and BTEX are important due to their wide applications in chemical plants and atmospheric environmental monitoring [22]–[23].

B. Chemical separations using home-made column

We further replaced the commercial column with the homemade PDMS coated capillary column and tested its chemical separation performance. First, ethanol-acetone and ethanol-hexane mixtures were used to test the separation performance of the home-made column. Next, pure acetone, ethanol, and hexane were used to identify the peaks in the gas chromatograph by matching the elution times. The two gas chromatographs are shown in Fig. 4 (a) and (b).

In order to further shrink the size of the home-made column, molecular sieve (MS) packed column was used due to its higher sample capacity and lower back pressure. The MS packed column has a channel length of 1m, channel width of 0.5mm, and depth of 1mm. The column was then tested using a mixture of hexane, pentane, heptane, and octane. As is shown in Fig. 4 (c), hexane and pentane peaks co-elute due to the fact that their elution times are close to each other but the separation of hexane-pentane, heptane, and octane peaks can be clearly seen with their elution times match well with the corresponding pure chemical elution times. The results suggest that it is possible to develop integrated miniaturized MS packed column while maintaining separation capability. This was realized by grinding the MS beads into finer powder to increase the surface-to-volume ratio and to allow tight packing of the MS powder inside the microchannel.

C. Study of chemical separation using integrated MS packed column on SPRi platform

The miniaturized MS packed column was integrated on top of a SPRi imaging gold chip and tested using hexane, heptane, and octane gases. Fig. 5 shows the image of the entire spiral column and three areas of interest, which are labeled by red boxes and identified with numbers. Fig. 6 shows the intensity change over time for hexane (a), heptane (b), and octane (c) of the three areas of interest (AOI), which correspond to the inlet, near the outlet, and the outlet of the spiral column. It can be observed from the chromatographs that the positions of the chemical peaks for the same chemical progressed in time from the AOI near the inlet to the ones further down the column. The retention time increases as the alkane carbon number increases. Similarly, the chemical elution time, which is when the signal peak arrives at the outlet, increases as the carbon number of the chemical increases. This is expected due to the differences of the chemicals' equilibrium constants. The experimental elution times of the

three chemicals are shown in Table I and can be used to identify the alkane species. It needs to be noted that the sudden intensity fluctuation from 5s to 15s is due to the switching of the 3-way valve which caused a sudden pressure change. Nevertheless, the fluctuation does not interfere with the chromatograph analysis.

D. Simulation study of separation process and comparison with experimental data

COMSOL Multiphysics simulation study was conducted on the separation processes of alkanes in the molecular sieve column. Fig. 7 shows the geometry of the cylindrical column used in the simulation with a diameter of 1mm and length of 3cm. The inlet and outlet are labelled in the picture. The molecular sieve used in the simulation was $\text{AlO}_2\text{O}_3\text{-4SiO}_2\text{-2MgO}$ with a density of 600kg/m^3 and porosity of 0.6. Linear velocity of flow field is 0.05m/s and chemical concentration is 2000mol/m^3 . Transport of diluted species in porous media physics was used to conduct the simulation; assuming constant flow rate, Millington and Quirk model for diffusion process, and Langmuir model for adsorption process.

The equations for analyte transport through a chromatographic column with constant porosity are as follows:

$$P_{1,i} \frac{\partial c_i}{\partial t} + P_{2,i} + \nabla \cdot \mathbf{\Gamma}_i + \mathbf{u} \cdot \nabla c_i = R_i + S_i \quad (1)$$

$$P_{1,i} = (\varepsilon_p + \rho k_{p,i}) \quad (2)$$

$$P_{2,i} = (c_i - \rho_p c_{p,i}) \frac{\partial \varepsilon_p}{\partial t} \quad (3)$$

$$\rho_p = \frac{\rho}{(1 - \varepsilon_p)} \quad (4)$$

$$N_i = \mathbf{\Gamma}_i + \mathbf{u} c_i = -D_{e,i} \nabla c_i + \mathbf{u} c_i \quad (5)$$

in which c_i is the chemical concentration, R_i is the reaction rate term (which is neglected here), S_i is the fluid source term, ε_p and ρ are the molecular sieve porosity and density respectively, ρ_p is the density of the non-porous molecular sieve, $k_{p,i}$ is the Langmuir constant of species i in porous medium, $c_{p,i}$ is the concentration of species i in porous medium, $D_{e,i}$ is the fluid diffusion coefficient of species i in certain medium, $\mathbf{u}$ is the

velocity field vector, Γ_i is the differential analyte concentration change with time, $P_{1,i}$ is the coefficient for concentration change with time, and $P_{2,i}$ equals to zero since the MS porosity does not change with time.

In order to simulate the separation of alkanes in molecular sieves, their physical and chemical properties such as diffusion constants and Langmuir constants are needed. Typical values of diffusion constants and Langmuir constants are chosen from literatures. Adsorption maximum for hexane is obtained from reference while those for heptane and octane are obtained from Langmuir isotherm plots [24–28]. The parameters used in the simulation is shown in Table I.

Fig. 8 shows snapshot of chemical progression in the channel for hexane, heptane, and octane at 1s of the simulation. It can be seen that hexane travels the fastest, followed by heptane and octane, which are the same as what we have observed experimentally. Both the retention times and the elution times from the experiments and the simulation are summarized and shown in Table II.

The experimental elution times (which are obtained from the chromatographs at the column outlet) for hexane, heptane, and octane are 24.2s, 26s, and 33.8s respectively. Retention times (which are obtained by the difference of signal peak positions between the inlet and outlet signals) are 1.5s, 2.4s, and 8.4s respectively. The elution times for hexane, heptane, and octane from the simulation were 24.4s, 25.8s, and 32.6s, while the retention times were 1.4s, 2.8s, and 9.6s respectively. It can be seen that the experimental and simulated results correlate well. For instance, the difference in elution times between hexane and octane is around 8 seconds for both simulation and experiment. Simulated retention time for heptane is off from experimental result by 0.4s which translates to an error about 14%, and is the highest among all data.

IV. CONCLUSION

We have successfully developed and tested a real-time imaging based SPRi platform as a gas chromatography detector. We have tested the separation ability of inexpensive, miniaturized, easy to fabricate PDMS coated column using this SPRi detector and successfully separated ethanol and alkanes mixtures.

We further miniaturized the size of the column by using a molecular sieve packed spiral column, and integrated the entire column onto the SPR imaging area, hence, realized real-time, imaging-based, and simultaneous chemical separation and detection. This approach decreases the back pressure of column, reduces the energy consumption, and combines separation and detection units into one miniaturized unit. Alkane samples were used to test the chemical separation process and different elution times were obtained for hexane, heptane and octane, demonstrating the separation capability of the miniaturized system. The separation process was simulated using COMSOL and the simulation results match well with the experimental results.

In conclusion, we have demonstrated a new miniaturized and integrated Micro-GC system that can simultaneously separate and detect chemical in real-time using sensitive SPRi platform.

This system greatly facilitates the study of chemical separation processes, demonstrates a new approach of Micro-GC integration, and provides a new possibility for the development of multidimensional Micro-GC systems.

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Fig. 1. (a) Experimental setup schematics. (b) Detailed view of detector and fixation with SPRi gold chip and prism.

Fig. 2.

(a) Miniturized MS packed column. (b) Integrated column on top of SPRi platform.

Fig. 3.

(a) Image of the SPRi detector cell channel area. (b) Gas chromatograph of ethanol and hexane mixture using commercial column. (c) Gas chromatograph of BTEX mixture using commercial column.

Fig. 4. Gas chromatographs obtained using home-made columns. (a) Acetone-ethanol mixture using PDMS column. (b) Hexane-ethanol mixture using PDMS column. (c) Four alkane mixture using MS packed column.

Fig. 5. SPRi image of the entire integrated MS packed spiral column and three areas of interest (AOIs) corresponding to the inlet, close to the outlet and the outlet.

Fig. 6. Gas chromatographs obtained using miniturized and integrated MS packed column. (a) Hexane, (b) heptane, and (c) octane.

Fig. 7.
Geometry of the column used in the COMSOL multiphysics simulation.

(a)

(b)

(c)

Fig. 8. Snapshots of alkanes separation processes in MS column at 1s. (a) Hexane, (b) heptane, and (c) octanes.

Table I

Physical Properties of Alkanes in Molecular Sieve Medium Used in COMSOL Simulation.

Properties	Chemicals		
	Hexane	Heptane	Octane
Diffusion Coefficient (m ² /s)	4×10^{-13}	8×10^{-13}	1×10^{-12}
Langmuir Constant (m ³ /mol)	0.256	0.192	0.128
Adsorption Maximum (mol/kg)	10	17	61

Table II

Experimental and Simulation Results for Alkanes Separation in Molecular Sieve Packed Column

Time (s)	Chemicals		
	Hexane	Heptane	Octane
Experimental: Retention Time	1.5	2.4	8.4
Experimental: Elution Time	24.2	26	33.8
Simulation: Retention Time	1.4	2.8	9.6
Simulation: Elution Time	24.4	25.8	32.6