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Applications of Synthetic Filaments in Separation Science

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1. Introduction

As a scientist studying applications of synthetic filaments in separation science, our research has led us to develop—and explore applications of—two specific technologies: (1) a filament-packed separation column, consisting of a cylindrical column loaded with a large number of synthetic filaments (with the filaments oriented parallel to the column axis), and (2) a capillary for preprocessing samples[1-3]. The internal structure of the filament-packed separation column differs from that of the separation columns commonly used for liquid chromatography (LC) and gas chromatography (GC), and in previous work we have presented case studies of separation problems in which the unique capabilities of synthetic filaments—their ability to resist dissolving in solvents, their good chemical resistance, and their high heat tolerance—were put to good use. The sample preparation capillary offers the convenient advantage of online coupling with micro-column LC systems and has been successfully used to miniaturize the entire LC process, from sample preprocessing to separation. In this article, I discuss applications of synthetic filaments (Figure 1) to the development of separation and extraction media, and then present a partial survey of innovative research made possible by this novel technique.

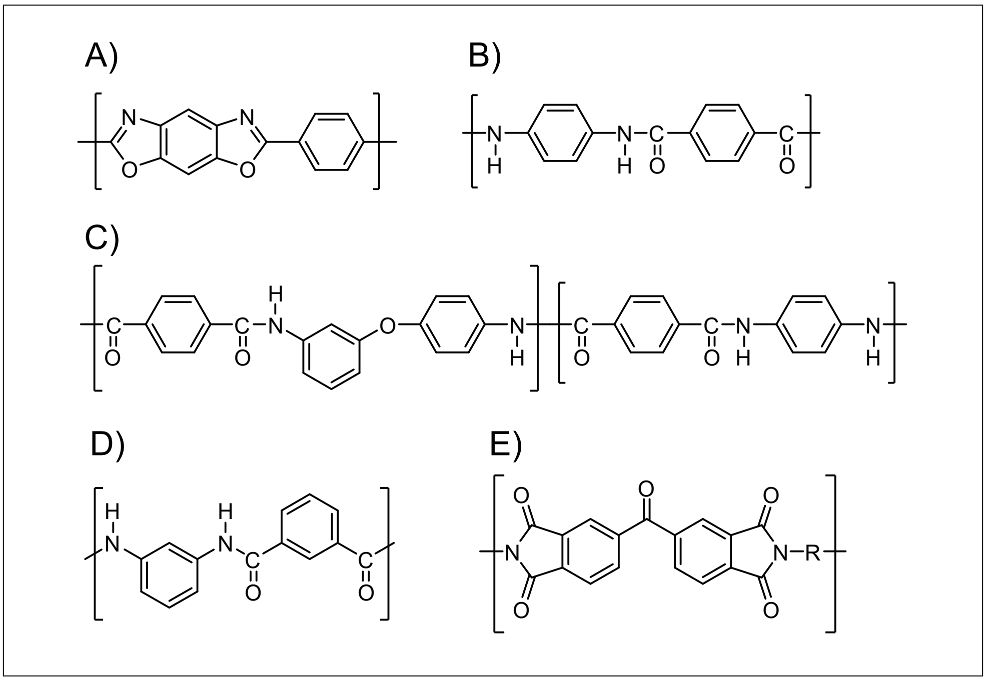

Fig. 1 Examples of synthetic filaments used as separation and extraction media: (A) Zylon, (B) Kevlar, (C) Technola, (D) Nomex, (E) Polyimide (P84)

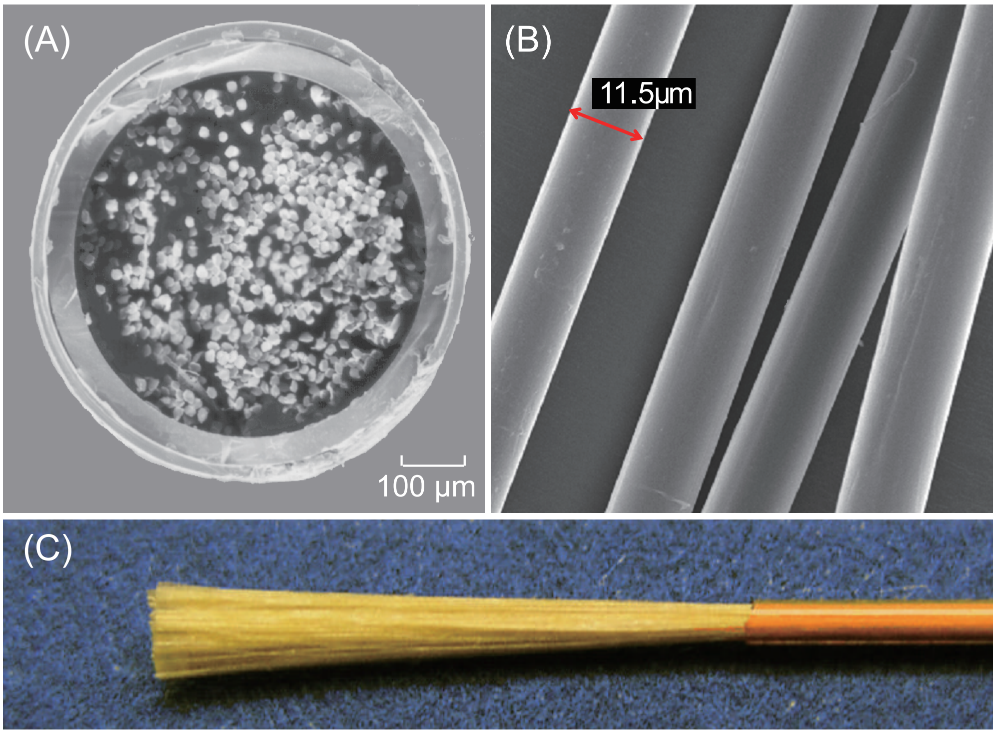

Fig. 2 Filament-packed capillary column for gas chromatography[4,5]. (A): Cross-sectional SEM image. (B): Higher-magnification SEM image of filaments. (C): Capillary column: A fused-silica capillary with a length of 1 m and an inner diameter of 0.53 mm packed with approximately 330 Zylon filaments.

2. Filament-Packed Capillary Column

The columns used for LC are typically packed with particulate packing materials. For GC, the most common choice is an open-tubular capillary column consisting of a fused-silica capillary with a stationary-phase liquid film formed on its inner walls; however, the alternative choice known as a packed column—a glass tube or similar tubing packed with particles of packing materials such as diatomaceous earth whose surfaces are coated with a stationary phase—has also been used for some purposes. In contrast, filament-packed capillary columns are packed with bundles of 10-12 µm-diameter filaments, oriented with the length of the filaments parallel to the column axis; the key characteristic of these columns is that their internal structure (Figure 2) differs from that of the columns conventionally used for LC and GC[4].

These novel columns, particularly when used as packed capillary columns for GC, offer high separation performance even for short column lengths of 1 m or below; in addition to the enormous increase in sample-loading volume, they can be used for temperature-programmed separation, just like conventional capillary columns (Figure 3). In other words, filament-packed capillary columns simultaneously offer the advantages of conventional packed columns regarding sample-loading volumes and the advantages of conventional open-tubular columns regarding temperature-programmed separation. The total surface area of the packed filaments is much greater than that of an open-tubular capillary column, but that entire surface area may be treated with liquid-phase coatings just like open-tubular capillary columns[5,6]. Moreover, derivatization reactions of functional groups existing on fiber surfaces[7,8] may be used to control the separation selectivity.

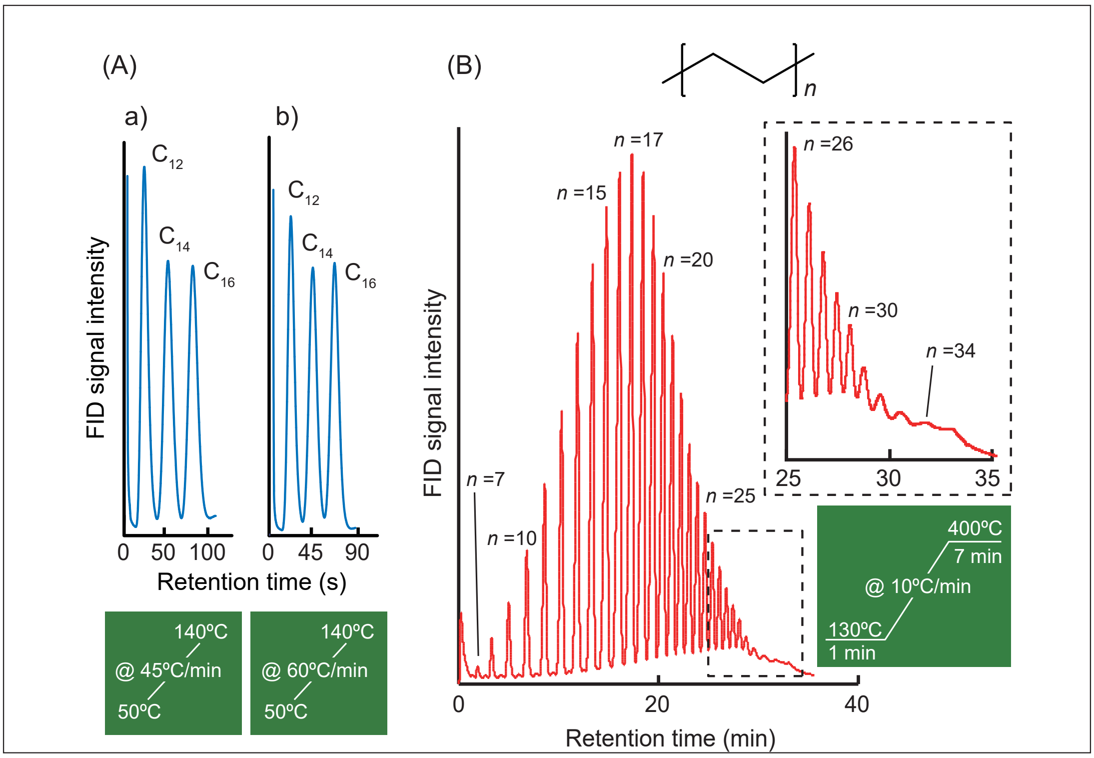

Fig. 3 High-speed temperature-programmed separation of alkanes using short filament-packed capillary columns[6].
(A) High-speed separation of an alkane mixture (C_{12}, C_{14}, C_{16}) using a short metal capillary column with an inner diameter of 0.3 mm and a length of 5 cm. Heating rates: a) 45°C/min, b) 60°C/min.
(B) Separation of Polywax 500 using a metal capillary column with an inner diameter of 0.3 mm and a length of 1 m packed with liquid-phase-coated filaments. Liquid-phase: PDMS.

3. Filament-Packed Sample Preparation Cartridge

In this section we introduce a micro-LC sample preparation device that exploits the outstanding insolubility and chemical resistance of synthetic filaments[9,10]. Like the filament-packed capillary column, the miniaturized sample preparation cartridge consists of a tube made of a substance such as PTFE or PEEK packed with a large number of filaments oriented with their lengths parallel to the tube axis. Only small pressures are required to induce fluid flow for liquids such as sample solutions or desorption solvents. Moreover, even at lengths of just 5 mm—the size of a grain of rice (Figure 4)—these cartridges have been shown to offer more than adequate extraction performance. We have also developed a system for online coupling of micro-scale sample preparation with micro-column LC. This system incorporates our ultraminiature sample preparation cartridges in the rotor parts of micro-column LC injectors[11].

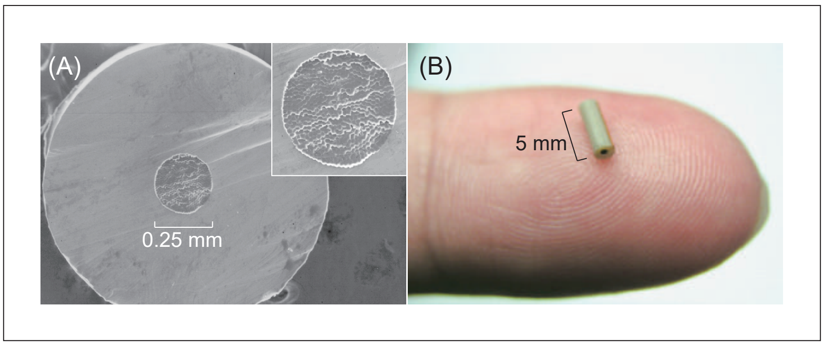

Fig. 4 Ultraminiature sample preparation cartridge for use in micro-LC[11].
(A) Cross-sectional SEM image of a PTFE tube with an inner diameter of 0.25 mm and a length of 5 mm packed with approximately 380 Zylon filaments.
(B) Photograph of a cartridge for injection valve installation consisting of a PEEK tube with an inner diameter of 0.50 mm and a length of 5 mm packed with approximately 1,500 Zylon filaments.

4. Prospects for Braided Filaments

By intertwining filaments in braided configurations, it is possible to form filament bundles with hollow interiors, which may be used to enclose stainless-steel wires or other thin metal strands (Figure 5). These bundles simultaneously offer a large total surface area and low resistance to the flow of sample solutions in the direction of the bundle axis. As in the case of filament-packed capillary columns, coatings or other surface treatments may be applied to the filaments to achieve sample-specific extraction selectivity for various purposes. These cartridges have many advantages for use as micro-scale sample preparation devices, conceived primarily for the purpose of extracting trace quantities of organic compounds from aqueous samples[12]. Moreover, resistive heating of the enclosed metallic wire allows efficient desorption processes after sample extraction (Figure 6).

Braided filament bundles may also be used to enclose materials other than metal wires. For example, an enclosed bundle of carbon fibers could find applications as a novel extraction medium boasting hybrid extraction capabilities. Braided packed capillaries enclosing metal wires allow precision temperature adjustment via external electrical controls, possibly enabling applications to interfaces for GC-GC or other two-dimensional chromatography systems—specifically, as the devices known as modulators that control the motion of the analysis target analytes from the first dimension to the second dimension.

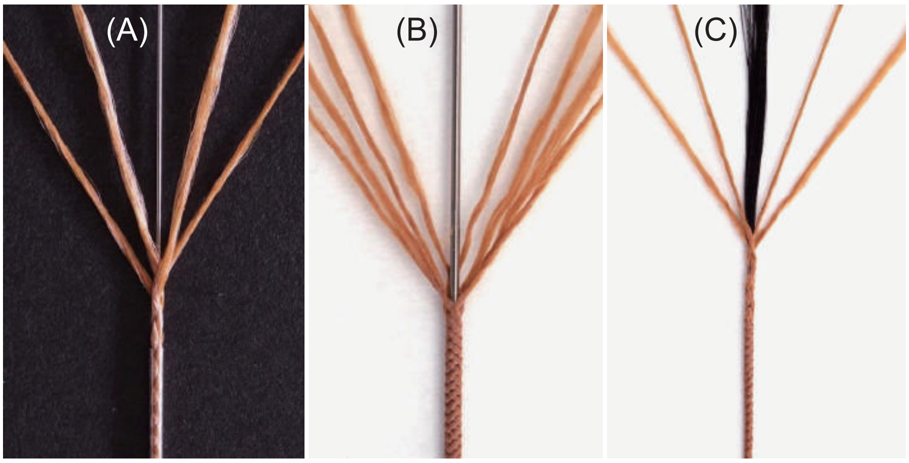

Fig. 5 Fabricated braids of filament bundles[12]. Each bundle consists of 166 Zylon filaments.
(A) A 4-bundle braid enclosing a stainless-steel wire with an outer diameter of 0.2 mm.
(B) An 8-bundle braid enclosing a stainless-steel wire with an outer diameter of 0.5 mm.
(C) A 4-bundle braid enclosing a bundle of 1,000 carbon fibers.

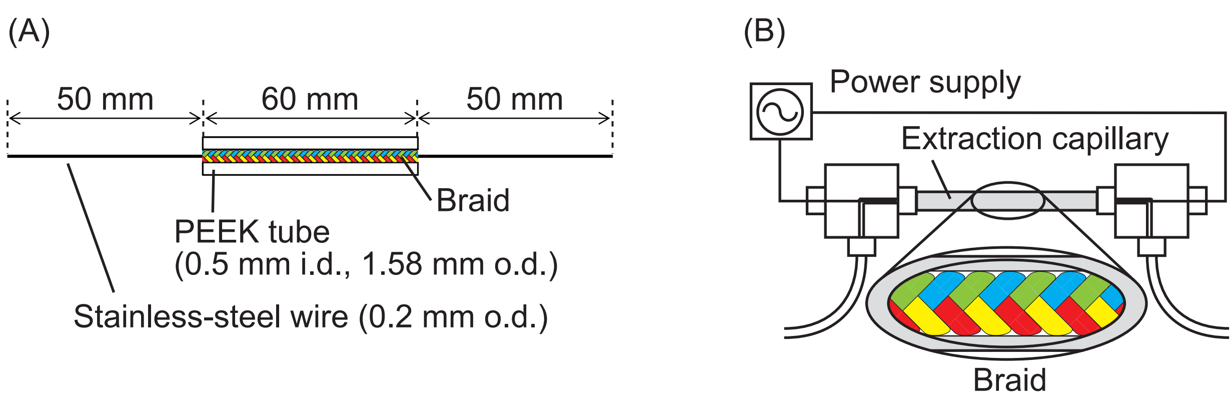

Fig. 6 Sample preparation device made from braid enclosing metal wire for electrical connectivity[12].
(A) Braided sample-extraction capillary.
(B) Schematic of sample preparation device enclosing extraction capillary. Application of a voltage on the order of a few volts allows ample capacity for resistive heating.

5. Conclusions

Synthetic filaments—particularly extremely high-strength filaments—are made possible by sophisticated modern chemical technology. Although they have found many applications as structural materials, as yet their uses as functional materials have remained limited. Similarly, braiding—despite being a traditional Japanese technology—has thus far been used for only a handful of industrial applications, including reinforcement for high-pressure pipes. In the future we may look forward to new tiers of applications for these basic technologies in the field of separation science.

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