

Surface areas in the range of 1–5 m<sup>2</sup>/g show that the diatomaceous earths are materials with a very low surface activity. Diatomaceous earths are used, for example, for the separation of anthraquinone derivatives (70), herbicides (71), phenolic compounds (72), tetracyclines (73), and vitamins (74) in a partition chromatographic mode.

Diatomaceous earths in thin-layer chromatography are not used only in their pure form; mixtures with surface-active silicas are also available. These mixed layers have a smaller adsorption capacity than pure surface-active silicas. The speed of chromatographic development with these mixed layers is very high. The separation of sugars (75) demonstrates that these layers can also be used successfully in partition chromatography.

## 2. Silica 50,000

An ideal carrier material for partition chromatography should have the following properties:

1. The sorbent must be only the support for the liquid stationary phase. There should be no retention of the samples by interaction with the carrier material.
2. The chemical composition and the physical parameters describing the structure have to be defined clearly and manufactured in a reproducible way.

Diatomaceous earth found in natural deposits fulfills these requirements only to some extent (see Sec. III.C.1). In particular, with regard to reproducibility and optimization of the structure parameters, it is obviously desirable to produce a synthetic material that is comparable with diatomaceous earths. Therefore, the development of a silicon dioxide named silica 50,000 was undertaken. This material consists of 100% SiO<sub>2</sub> with a mean pore size of 5000 nm, a pore volume of around 0.6 mL/g, and a specific surface area of approximately 0.5 m<sup>2</sup>/g. Silica 50,000 is available commercially as a precoated layer. The mean particle size and the particle size distribution correspond to HPTLC quality. Typical applications of this wide-pore material in partition chromatography are separations of amino acids (76), carbohydrates (77–79), and digitalis glycosides (80).

Diatomaceous earths and silica 50,000 are used not only in thin-layer chromatography as carriers for the partition chromatographic process, but also as inert sorbents for the so-called concentrating zone in front of the separation layer (see Sec. IV).

## D. Celluloses

Celluloses are used in paper and in thin-layer chromatography as organic stationary phases. In contrast to paper chromatography, where cellulose is applied as a self-supporting layer, in thin-layer chromatography the cellulose particles are classified and spread as layers on glass, aluminum, or plastic supports. As a result, cellulose layers can be produced in different qualities up to precoated layers for HPTLC. In general, celluloses used for chromatography are composed of long chains of  $\beta$ -glucopyranose units, which are connected to one another at the 1,4 positions.

In thin-layer chromatography two types of celluloses are distinguished (81):

1. Native cellulose has a degree of polymerization of 400–500 glucose units and a fibrous structure. The length of the fibers is in the range of 2–20  $\mu$ m, and the specific surface area measures around 2 m<sup>2</sup>/g.
2. Microcrystalline cellulose consists of an average of 40–200 glucose units.

The lower degree of polymerization of microcrystalline cellulose compared with that of native cellulose results from the process of synthesis: The amorphous parts of highly pure native cellulose are dissolved by acid hydrolysis. After this cleaning process, the residual cellulose forms rod-shaped crystalline aggregates. The specific surface area is comparable to that of native cellulose.

Like silica gel, microcrystalline cellulose is available not only as bulk TLC material for self-coating plates but also as industrially produced precoated layers for conventional thin-layer chromatography, high-performance thin-layer chromatography, and preparative layer chromatography. With regard to the different morphologies of the particles, particle size distributions and mean particle sizes are in ranges comparable to those of silica. Because both types of cellulose used in