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Review

Miniaturized planar chromatography using office peripherals — Office chromatography

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ABSTRACT

Office chromatography (OC) harnesses the novel combination of miniaturized planar separation science and modern print & media technologies. Interdisciplinary knowledge is the essence: Printing of solutions on powerful miniaturized planar separation materials in combination with image capturing and evaluation tools enables an innovative analytical online system. Site-specific printing as lines or areas on defined sections of the layer comprises important steps like application of samples, feeding of the mobile phase as well as supply of the derivatization reagent. Also printing of bioassays can be combined for effect-directed detections and the homogeneous printing of the ultrathin layer itself, enabling tailor-made gradient-layer or multi-layer plates. OC exploits image-giving miniaturized chromatograms being captured and processed with a flatbed scanner or mini-camera. Thus, miniaturized separation materials are the core of OC. Monolithic, electrospun, nanostructured glancing angle deposition and carbon nanotube-templated microfabricated layers or even pillar arrays or polymer brush coated sub-μm silica particles were demonstrated, showing promising results. Layer thicknesses from 50 μm down to few micrometers were explored. A high-throughput capacity is given through the parallel development of as many as possible tiny-printed samples on the separation material. The migration time was reduced to a few minutes and the calculated analysis time per sample lasted few seconds. Considering a substantially reduced solvent consumption at short run times for parallel analysis of numerous samples at the same time, OC is an appropriate analytical technique for green chemistry. OC facilitates the whole planar separation process to be performed with no other equipment but a combined device of printer and flatbed scanner or mini-camera. At the same time, OC can be expected to become a widespread and economical technique with the user-friendliness of high-end office tools, appealing to users.

Contents

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1. Trend of miniaturization in planar chromatography

Since 1938, planar chromatography has been known in separation science. Its progress was described in detail in a chronology on instrumental developments [1]. It comprises various techniques, for which the size of the separation material was ongoingly reduced over the last seven decades (Fig. 1) [2]. Thin-layer chromatography (TLC) is a simple, qualitative technique with samples applied manually on TLC plates of typical particle sizes of 10 to 15 μm. High-performance thin-layer chromatography (HPTLC) is an indispensable tool in a variety of fields and is used for both qualitative and quantitative analysis. HPTLC uses modern instrumentation, sorbents of a lower particle size as well as a narrower and more homogeneous particle size distribution than TLC [3]. HPTLC is a cost-effective, matrix robust and quantitative method [4]. The results obtained by either TLC or HPTLC are so different in their quality (Fig. 1) that a clear differentiation between TLC and HPTLC is relevant. The stationary phase was further miniaturized to a considerable degree in ultrathin-layer chromatography (UTLC): The size was reduced by a factor of about 10 and 20, if compared to HPTLC and TLC, respectively. Also the layer thickness decreased from 200 to 250 μm in TLC/HPTLC to ≤50 μm in UTLC [5–7]. Hence, UTLC is visually defined by a significantly reduced layer thickness on a miniaturized plate format. Initially, the term UTLC was used for the monolithic layers [5], but nowadays it comprises a variety of different layer microfabrication techniques. Paper chromatography, also being used in a miniaturized form [8], was not included in this comparison due to its low separation power. However, functionalized papers tailor-made for chromatography are of future interest.

So far, the shortcoming of this miniaturization trend in planar separation science was the lack of special instrumentation for UTLC. Miniaturized plate formats are very difficult to handle with the current TLC/HPTLC equipment commercially available. This deficiency combined with the reduced surface activity of miniaturized layers has hampered the progress of UTLC in the last decade. A reduced surface activity meant that active sites were less available for the separation process and the elution strength of the mobile phase had to be reduced [9]. Thus, HPTLC methods could not be transferred to UTLC methods without adjustment. Moreover, the application of nanoliter volumes of a higher concentrated solution, if compared to HPTLC, was required to generate narrow start zones [9]. This was crucial for a good separation, as an increased spreading of the start zone during the application of polar to middle polar sample solutions was a side-effect of the reduced surface activity. Hence, a boost to UTLC is strongly related to the availability of special instrumentation for miniaturized planar separations and to accompanying tutorials for use of the miniaturized layers.

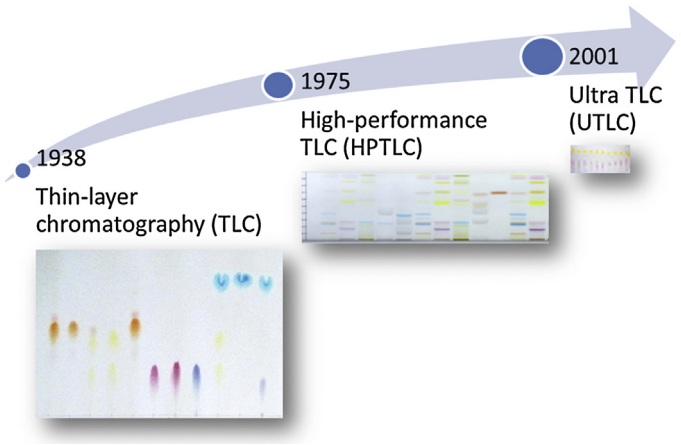

Fig. 1. Clear trend to miniaturization over the last seven decades: Comparison of the sizes of planar chromatographic layers, shown are separations of water-soluble food dyes on silica gel layers.

2. Past automatization approaches to obtain a fully online system

In 1970, the Baker Chemical Company built an automated TLC system that was patented [10]. Applications running on that machine were never shown. In 1989, Prošek et al. reported a fully online system [11]. In 1993, Delvordre and Postaire described a totally automatic TLC machine [12,13]. All these approaches described a large, inflexible mechanical instrument without any proof of principle. In 1997, Nyiredy applied for a Swiss patent coupling a liquid jet with TLC, which was obtained in 2001 [14]. Neither a proof of principle nor any results were reported. The problem associated with these previous, theoretical approaches was the dimension of the layer size and the layer thickness because regular TLC/HPTLC layers were utilized. These large plate formats could not be handled in a rational fully-automated mode with regard to all TLC/HPTLC steps involved, and thus, a proof-of-concept was difficult to perform. Additionally, these large machines were inflexible with regard to the plate processing. In that time, it was concluded that fully online automation in HPTLC is not that necessary as for high-performance liquid chromatography (HPLC) [15]. This was true thanks to the intrinsic capability of HPTLC to perform many separations in parallel. However, in the past time with its boost of the online techniques, step-automated HPTLC (employing 3–5 different devices, depending on the task) lost market. Advancement of HPTLC was sparsely due to the loss of the critical mass (substitution of TLC methods by HPLC methods and not by HPTLC methods), associated with a decrease in teaching and training. Despite the ongoing online trend (now for 4 decades), recent advances in HPTLC/mass spectrometry (MS) [16] and effect-directed detection (EDA) [17–19] attracted new interest in step-automated, hyphenated HPTLC methods.

3. Development of office chromatography

3.1. The idea of a fully online system for planar separation science

On the one hand, working with miniaturized plate formats provided an understanding of the problems associated with their use. Spray applications used in HPTLC were not suited for use in UTLC