Porous Separation Media Engineered through Glancing Angle Deposition

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Introduction
Analytical separations through porous media are common methods of characterizing biomolecules in mixtures. Ultrathin layer chromatography (UTLC) enables in-plane biomolecule separation. Solvent (mobile phase) wicks through a porous medium (stationary phase) and carries the analytes in a mixture through a different distance. UTLC exhibits reduced reagent needs and low limits of detection [1]. The method can also separate many samples in parallel.

Pore size and geometry dictate analytical separation behavior. While much success has been achieved with isotropic media, the ability to precisely tailor macroscopic width and shape can introduce new functionality.

Glancing Angle Deposition (GLAD)
Glancing angle deposition (GLAD) permits porous thin film microstructure fabrication [1-3]. Macropore size and geometry may be controlled over large areas with this single-step evaporation deposition process.

Growing nuclei cast microscopic shadows at oblique deposition angles (\(\phi\)). At highly-oblique angles (\(\phi > 80^\circ\)), the nuclei grow into well-separated columns (Fig. 1a).

Limitless porous microstructures are possible through computerized substrate motion control. For example, rapid rotation with constant \(\phi\) produces an isotropic vertical post film (Fig. 1b). Macroscopic shadows cast by a block mounted to the centre of a substrate chuck (Fig. 1d) introduce gradients in thickness and morphology to substrates that surround it (Fig. 1e) [3].

Early GLAD UTLC [1] warranted further study. UTLC conducted on anisotropic films was compared against control separations on isotropic films (Fig. 1c).

UTLC elution along the radial vector on films spotted with a cylindrical shadow block shows radial vector pointing outwards from the centre (Fig. 2).

UTLC across the radial vector with a SBD film is the degenerate case of a UTLC elution against a cylindrical shadow block.

Experimental Methods
Stationary Phase: SiO2 GLAD films (5 \(\mu\)m nominal thickness, \(v = 84^\circ\) deposited onto 1" square 8270 glass substrates
Mobile Phase: ethanol : water (35 : 60 : 3.5)

UTLC Development: 5 min in a horizontal development chamber machined from Teflon®. Mobile phase supplied to one end of the face-up spotted GLAD film by wicks.

Conclusions
Channel structures in anisotropic GLAD media provided preferential paths of mobile phase wicking and analyte migration. Best separations occurred in the “along-channel” direction. Development diagonal to the channel structures decoupled analyte migration from the elution direction. Separation quality in the anisotropic media may be associated with channel structure width. Faster migrations in serial bideposition films suggest that such media have wider channels than those of chevron films. Both anisotropic films enabled faster migration than isotropic vertical post films. Thicker films allowed faster migration. The spatially-graded post film was thicker further from the shadow block. UTLC in the “across-channel” direction achieved better separation than that in thinner regions.

Future investigation of GLAD separation media with and without an applied electric field is anticipated to yield new UTLC electrochromatography, and electrophoresis analyses for biologically-relevant molecules.

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