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New HPLC stationary phases prepared by Ugi multicomponent synthesis

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The column, the 'heart' of the HPLC system, has changed greatly with the development of liquid chromatography. The stationary phases packed in the columns have been improved to provide greater separation speed and efficiency, as well as increased stability and reproducibility. Many new sorbents have been introduced for the extension of HPLC/UHPLC to a wider range of sample types or for better separation of compounds, which have in the past proved problematic. However, there exists still vital interest in new stationary phases possessing different separation selectivity or providing an improved separation with respect to commercially available columns. In addition, new techniques of stationary phase synthesis are continuously searched and proposed for their potential to provide novel stationary phases more easily or with some other benefit.

In our work, eight different stationary phases based on two aminopropyl silicas of different brands suitable for multimodal chromatography applications have been prepared by a four-component Ugi reaction. Our intention was to synthesize stationary phases significantly differing in their properties, thereby demonstrating flexibility of the Ugi synthetic protocol. Diverse functional groups including a nonpolar long aliphatic chain, a phenyl moiety, a cholic acid scaffold, phenylboronic and monosaccharide units, charged betaine and arginine moieties were immobilized on a silica surface. The novel sorbents were extensively characterized by a wide variety of analytical techniques, including elemental analysis, Raman spectroscopy and chromatography. Considering the anchored chemical structures covalently bonded to the silica surface, reversed-phase, hydrophilic and ion-exchange separation modes were expected. The chromatographic evaluation was performed to map the potential of the individual columns specifically in the mentioned chromatographic modes.

Conclusion: The Ugi synthetic protocol has proven to be a simple, feasible and versatile tool for the synthesis of sorbents with variable properties. The newly prepared stationary phases differed considerably in hydrophobicity and ion exchange ability. A significant influence of the supporting aminopropyl silica on the final chromatographic behavior was observed.

Biography

David Sykora PhD was born at Mestec Kralove, Czech Republic. He graduated from the Charles University in Prague, Department of Organic Chemistry. After graduation he worked as an application specialist at Tessek Ltd. where he was responsible for the design and evaluation of new sorbents for HPLC and the development of new chromatographic applications. He received his PhD from the Institute of Chemical Technology (now the University of Chemistry and Technology, UCT Prague). As a postdoctoral fellow, he stayed in the laboratory of Professor J. M. J. Frechet at the University of California, Berkeley, where he worked in the area of chromatographic characterization of synthetic polymers and development of innovative stationary phases, particularly monoliths, for HPLC, CEC and GC. Currently he is an Associate Professor at UCT Prague. His interests cover the development of chromatographic methods, mainly suitable for medicinal research and analysis and the development of new separation media for HPLC, CE and CEC. His scientific interests also include hyphenated techniques; particularly LC-MS. He published more than 100 papers in impacted journals.

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