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Novel Adsorbents for HILIC Obtained via the Ugi Multicomponent Reaction Varying Isocyanide and Carbonyl Compound

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Selectivity and efficiency in HILIC generally depend on the structure of the stationary phase, therefore, the actual direction in the method development is the synthesis of novel adsorbents and the study of their properties. Modification of silica substrate is not only to increase its hydrophilicity, but also opens up a versatile method to synthesize adsorbents with enhanced selectivity for diverse analytical tasks.

The aim of this work was to use flexibility of combinatorial Ugi reaction with using different commercially available compounds to manipulate the selectivity and increase efficiency of the resulting phases. A series of novel stationary phases was obtained by conducting the Ugi reaction directly on 3-aminopropyl silica substrate surface varying the structure of the carbonyl compound (acetone, 2-acetylfuran, 2-acetylpyrrole, and acetaldehyde) and isocyanide (*tert*-butyl isocyanide, ethyl isocyanacetate, 2-morpholinoethyl isocyanide, p-toluenesulfonylmethyl isocyanide, and diethyl isocyanomethylphosphonate). To increase the yield of the heterogenous reaction a synthesis time, a catalyst, and a solvent nature was varied.

Varying components in the Ugi reaction had a significant influence on the selectivity of the adsorbents toward sugars, amino acids, nucleobases and nucleosides, organic acids, and water-soluble vitamins. A 15–50% increase in the obtained phases efficiency (up to 60000 N/m) for different polar analytes as compared to the substrate confirmed the prospects for the formation of the proposed functional layers.

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Keywords: Hydrophilic interaction liquid chromatography, stationary phase, Ugi reaction, separation of polar compounds