# A new nano-on-micro stationary phase based on nanodiamond bonded on silica for hydrophilic interaction chromatography

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## Chemicals and Reagents

All chemicals and reagents were used without further purification. Spherical porous silica (diameter: 5 μm, pore size: 90 Å, surface area: 306 $m^2$ $g^{-1}$), from Fuji Silysia Chemical Ltd. (Aichi, Japan). Nanodiamond (particle size : <10nm, 97.00%) and Diethyl Cyanophosphonate (93%) was purchased from TCI Chemicals (Shanghai). 3-aminopropyltriethoxysilane (98%) got from Energy Chemical. Sulfonamide drugs were obtained from Aladdin (analytical standard: sulfadimoxine, sulfamerazine, sulfapyridine, sulfanilamide, sulfadimethoxine, sulfamethazine, sulfisoxazole) and Energy Chemical (analytical standard: sulfadiazine, sulfathiazole). The others chemicals and solvents are analytical standard from Energy Chemical.

## Instrument and method

Amino column (250 × 4.6 mm i.d.) was supplied by Lanzhou Institute of Chemical Physics (Lanzhou, China). The test of nine sulfonamides was carried out with a Shimadzu-GL LC-15C system including two high-pressure pumps, a SPD-15C UV/vis detector, a CTO-15C column oven and a 50 μL Shimadzu-GL microsyringe, the UV/vis detector was set at 254 nm wavelength. The test of four saccharides was carried out with Agilent 1260 Infinity Series modular system with quaternary pumps (Agilent Technologies, USA), a Alltech 3300 evaporative light scattering detector (Grace, USA) with GCK3302 air generator (BCHP Analytical Technology Institute, China). The ELSD was set as follows: gas flow, 2.0 L $min^{-1}$; evaporative temperature, 115 °C; photomultiplier, 1; gas pressure, 0.36 Mpa. The FTIR spectra was collected from IFS 120HR fourier transform infrared spectrometer (Bruker, Germany). Elemental analysis results were determined by Vario EL III elemental analyzer (Hanau, Germany). Transmission electron microscopies (TEM) imaging were obtained from Tecnai G2 TF20 transmission electron microscope (FEI, USA). Scanning electron microscopies (SEM) imaging received from JSM-6701F scanning electron microscope (JEOL, Japan).

## Characterization of Sil- OND

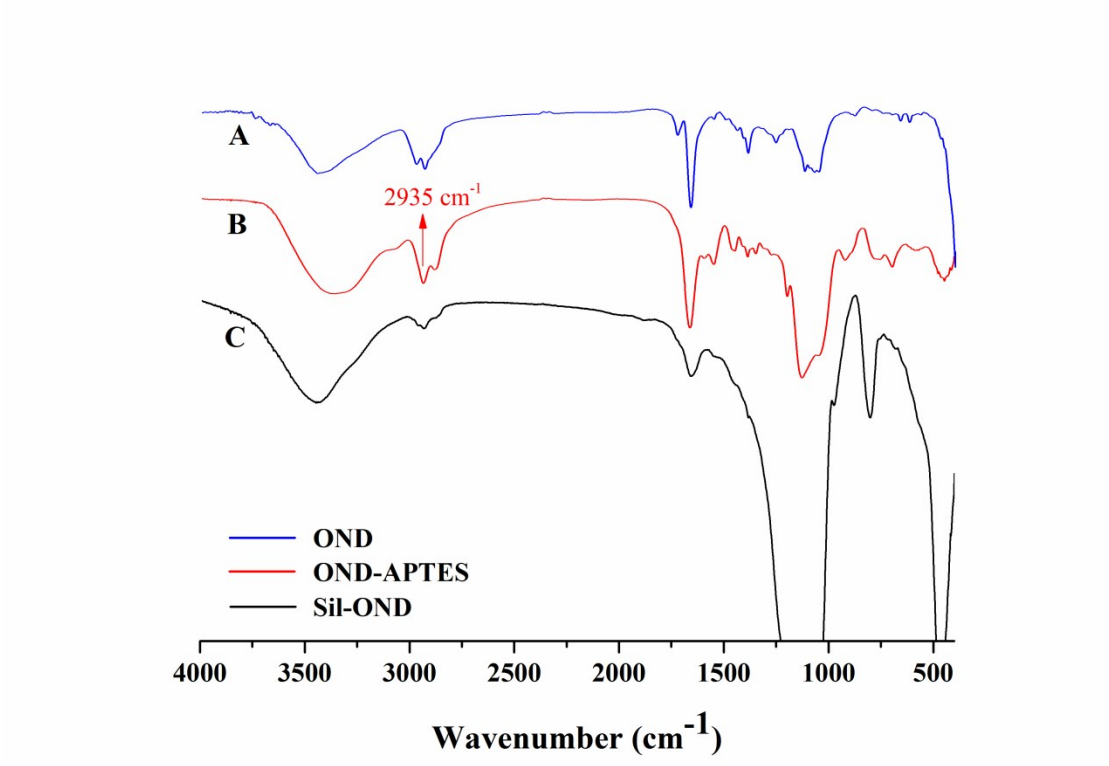


**Fig. S1** FTIR spectra of (A) OND, (B) OND-APTES and (C) Sil-OND.

**Table S1** The results of elemental analysis.

| Sample | C (%) | N (%) | H (%) |
|---|---|---|---|
| OND | 88.85 | 2.50 | 1.06 |
| OND-APTES | 56.43 | 5.72 | 5.22 |
| Sil-OND | 3.00 | 0.10 | 0.76 |

## Influence of acetonitrile concentration

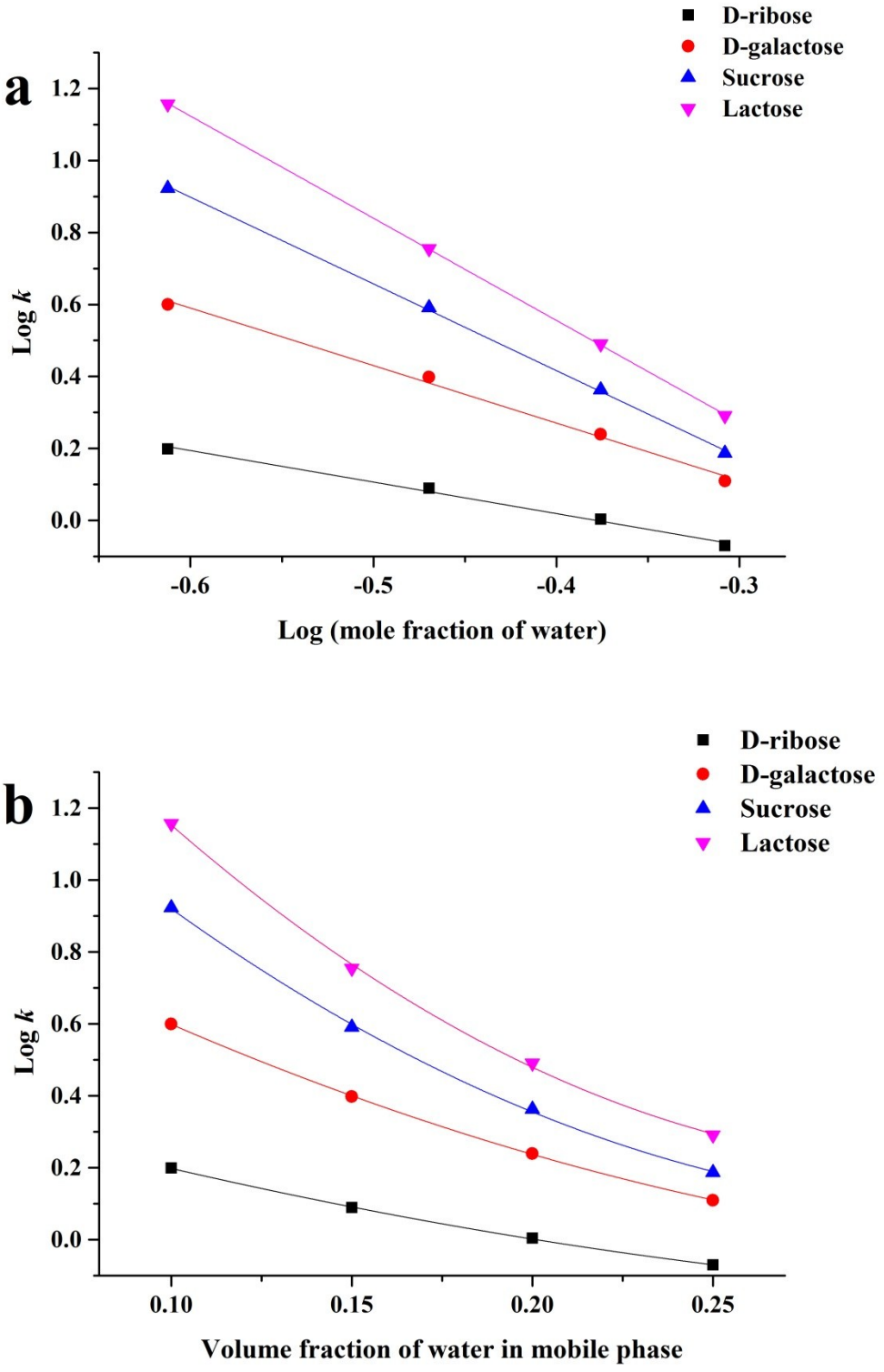


**Fig. S2** Dependence of log *k* of four saccharides on the logarithm of mole fraction of water (a) and the volume fraction of water in mobile phase (b). The aqueous part of mobile phase: 20 mmol ammonium acetate solution, pH = 6.6, flow rate = 1.0 ml $min^{-1}$, *T* = 16 °C.

## The reproducibility of Sil-OND column

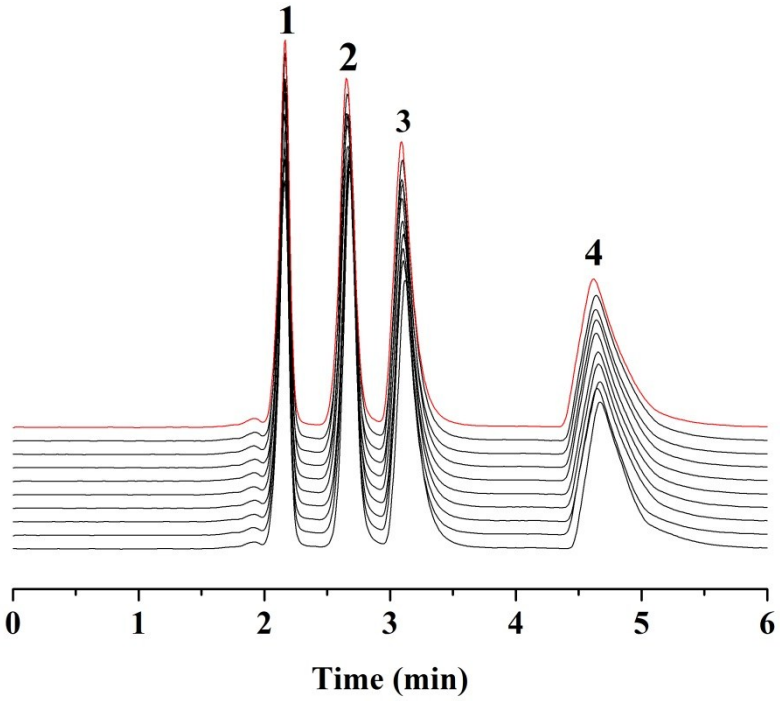


**Fig. S3** The reproducibility test of Sil-OND column with sulfanilamide(1), sulfapyridine(2), sulfamethazine(3), sulfamerazine(4); Mobile phase: 89% acetonitrile: 11% 20 mmol ammonium acetate solution, flow rate = 1.0 ml $min^{-1}$, $T$ = 30 °C.