Selectivity of stationary phases with alkyl, phenyl and pentafluorophenyl groups on core shell particle

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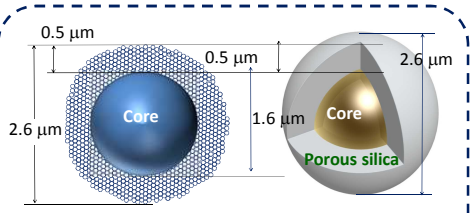
Abstract

Brand columns packed with superficially porous particles have been available for some time. The superficially porous media or so called core-shell media offers significant improvements such as higher efficiency and lower pressure drop for existing HPLC operations without having to replace existing HPLC systems with UHPLC systems.

In this study, a 2.6 µm core-shell silica with a non-porous core approximately 1.6 µm in diameter and a superficially porous layer of 0.5 µm was used as a based material. Core-shell silicas bonded with C18, C28, phenylethyl and pentafluorophenyl (PFP) groups were evaluated for hydrogen bonding capacity, hydrophobicity, steric selectivity and both peak shape and retention of oxine as a metal chelating compound.

A core-shell C28 with long chain ligands was suitable for separation of both high polar compounds using 100% aqueous mobile phase and a fat-soluble compound to compare with a conventional C18, while a core shell PFP could separate 3 kinds of isomers of cresol completely although a C18 could not separate meta-cresol and para-cresol. Different selectivity by different stationary phases was confirmed on core shell silica particles as well as fully porous silica particles.



Particle diameter: 2.6 µm, Core diameter: 1.6 µm, Thickness of porous silica: 0.5 µm, Pore volume: 0.30 mL/g, Specific surface area: 150 m^2/g, Pore diameter: 9 nm, The ratio of porous silica volume: 77%

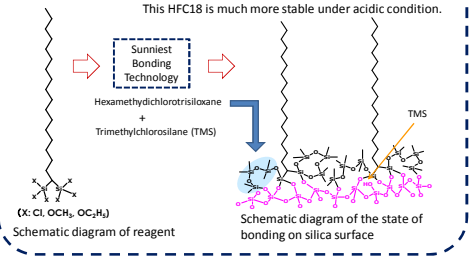
Figure 1. Schematic diagram of a core shell silica particle

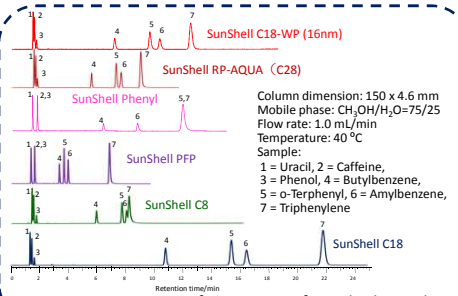
Table 1. Characteristics of SunShell

	Core shell silica			Bonded phase		
	Particle size (µm)	Pore diameter (nm)	Specific surface area (m^2/g)	Carbon content (%)	Bonded phase	Available pH range
SunShell C18	2.6	9	150	7	C18	1.5 - 10
SunShell C8	2.6	9	150	4.5	C8	1.5 - 9
SunShell PFP	2.6	9	150	4.5	Pentafluorophenyl	2 - 8
SunShell Phenyl	2.6	9	150	5	Phenylhexyl	1.5 - 9
SunShell P-AQUA	2.6	16	90	4	C28	2 - 8[a)]
SunShell C18-WP	2.6	16	90	5	C18	1.5 - 10
SunShell HFC18-16	2.6	16	90	2.5	C18	2 - 9
SunShell HFC18-30	2.6	30	40	1.3	C18	2 - 9
SunShell C8-30	2.6	30	40	1.2	C8	2 - 9
SunShell C4-30	2.6	30	40	0.9	C4	2 - 8

a) This value is evaluated under 100% aqueous condition because SunShell RP-AQUA has reproducible retention under 100% aqueous condition.

Figure 2. What is HFC18?



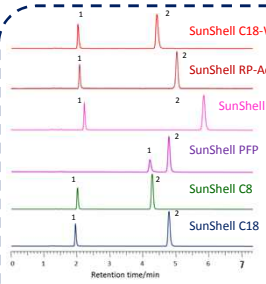


Column dimension: 150 x 4.6 mm
Mobile phase: CH_3OH/H_2O=75/25
Flow rate: 1.0 mL/min
Temperature: 40 °C
Sample:
1 = Uracil, 2 = Caffeine,
3 = Phenol, 4 = Butylbenzene,
5 = o-Terphenyl, 6 = Amylbenzene,
7 = Triphenylene

Figure 3. Comparison of separation of standard samples

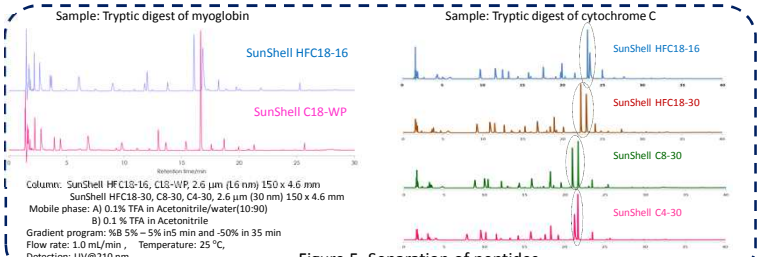
Table 2. Comparison of selectivity

	Hydrogen bonding (Caffeine/Phenol)	Hydrophobicity (Amylbenzene/ Butylbenzene)	Steric selectivity (Triphenylene/ o-Terphenyl)
C18-WP	0.40	1.55	1.35
RP-AQUA	0.52	1.52	1.30
PhEenyl	1.00	1.58	1.01
PFP	1.00	1.31	2.38
C8	0.32	1.46	1.08
C18	0.39	1.60	1.46



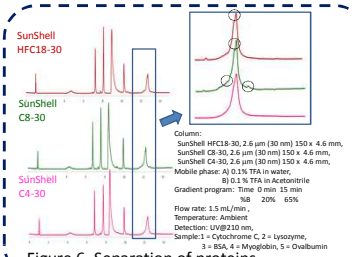
Column dimension: 150 x 4.6 mm
Mobile phase: CH_3CN/20mM H_3PO_4=10/90
Flow rate: 1.0 mL/min
Temperature: 40 °C
Detection: UV@250nm
Sample: 1 = 8-Quinolinol (Oxine)
2 = Caffeine

Figure 4. Comparison of oxine and caffeine



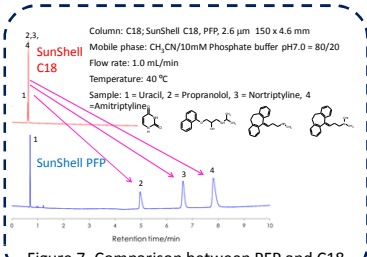
Column: SunShell HFC18-16, C18-WP, 2.6 µm (16 nm) 150 x 4.6 mm
SunShell HFC18-30, C8-30, C4-30, 2.6 µm (30 nm) 150 x 4.6 mm
Mobile phase: A) 0.1% TFA in Acetonitrile/water(10:90)
B) 0.1 % TFA in Acetonitrile
Gradient program: %B 5% – 5% in5 min and -50% in 35 min
Flow rate: 1.0 mL/min , Temperature: 25 °C,
Detection: UV@210 nm.

Figure 5. Separation of peptides



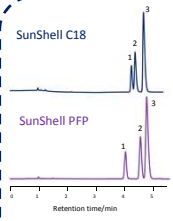
Column:
SunShell HFC18-30, 2.6 µm (30 nm) 150 x 4.6 mm,
SunShell C8-30, 2.6 µm (30 nm) 150 x 4.6 mm,
SunShell C4-30, 2.6 µm (30 nm) 150 x 4.6 mm,
Mobile phase: A) 0.1% TFA in water,
B) 0.1 % TFA in Acetonitrile
Gradient program: Time 0 min 15 min
%B 20% 65%
Flow rate: 1.5 mL/min ,
Temperature: Ambient
Detection: UV@210 nm,
Sample:1 = Cytochrome C, 2 = Lysozyme,
3 = BSA, 4 = Myoglobin, 5 = Ovalbumin

Figure 6. Separation of proteins



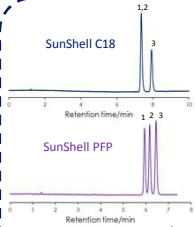
Column: C18; SunShell C18, PFP, 2.6 µm 150 x 4.6 mm
Mobile phase: CH_3CN/10mM Phosphate buffer pH7.0 = 80/20
Flow rate: 1.0 mL/min
Temperature: 40 °C
Sample: 1 = Uracil, 2 = Propranolol, 3 = Nortriptyline, 4 =Amitriptyline

Figure 7. Comparison between PFP and C18



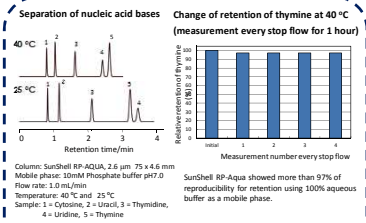
Column:
SunShell C18, PFP 2.6 µm 150 x 2.1 mm
Mobile phase:
CH_3OH/water=75:25 for SunShell C18
CH_3OH/water=60:40 for SunShell PFP
Flow rate: 0.3 mL/min
Temperature: 25 °C
Detection: UV@250nm
Sample: 1 = o-Xylene
2 = m-Xylene
3 = p-Xylene

Figure 8. Comparison between PFP and C18 (3)
Separation of isomer of xylene



Column:
SunShell C18, 2.6 µm 150 x 4.6 mm
SunShell PFP, 2.7 µm 150 x 4.6 mm
Mobile phase: CH_3OH/H_2O=40/60
Flow rate: 1.0 mL/min
Temperature: 25 °C
Sample: 1 = p-Cresol
2 = m-Cresol
3 = o-Cresol

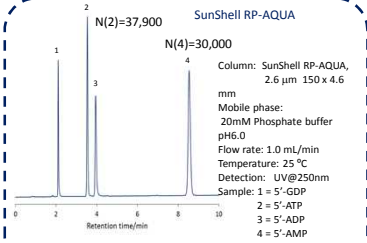
Figure 9. Comparison between PFP and C18 (4)
Separation of isomer of cresol



Column: SunShell RP-AQUA, 2.6 µm 75 x 4.6 mm
Mobile phase: 10mM Phosphate buffer pH7.0
Flow rate: 1.0 mL/min
Temperature: 40 °C and 25 °C
Sample: 1 = Cytosine, 2 = Uracil, 3 = Thymidine, 4 = Uridine, 5 = Thymine

SunShell RP-Aqua showed more than 97% of reproducibility for retention using 100% aqueous buffer as a mobile phase.

Figure 10. Reproducibility of retention under 100% aqueous condition



Column: SunShell RP-AQUA, 2.6 µm 150 x 4.6 mm
Mobile phase:
20mM Phosphate buffer pH6.0
Flow rate: 1.0 mL/min
Temperature: 25 °C
Detection: UV@250nm
Sample: 1 = 5'-GDP
2 = 5'-ATP
3 = 5'-ADP
4 = 5'-AMP

Figure 11. Separation of nucleotides

Conclusion

*Hydrogen bonding, hydrophobicity and steric selectivity of alkyl groups, phenyl group and pentafluorophenyl group were evaluated.

*Pentafluorophenyl showed highest hydrogen bonding and highest steric selectivity and much different from C18 group. Pentaflorophenyl group showed much longer retention time for a polar compounds and could separate isomers better than C18 group.

*C28 group showed reproducible retention under 100% aqueous condition.